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CROP PROTECTION

Ovicidal effect of the essential oils from 18 Brazilian *Piper* species: controlling *Anticarsia gemmatalis* (Lepidoptera, Erebidae) at the initial stage of development

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ABSTRACT. The toxicities of essential oils (EOs) from 18 species of Brazilian Piperaceae were assessed on eggs of the velvetbean caterpillar, *Anticarsia gemmatalis*. Oils were extracted using steam distillation, and dilutions were made for bioassays at concentrations of 0.25, 0.5, 1.0, 2.0, and 4.0%. All EOs reduced larval hatching. The lowest lethal concentrations were obtained from *Piper fuligineum* (SP), *Piper mollicomum* "chemotype 1" (SP), *Piper mosenii* (PR), *Piper aduncum* (PA) and *Piper marginatum* (PA). Ovicidal activity is related to the potential toxicity of several compounds, especially dilapiolle, myristicin, asaricine, spathulenol and piperitone. According to our results, EOs from 16 Brazilian *Piper* species have potential for use as biorational botanical insecticides.

Keywords: *Piper abutiloides*, *Piper fuligineum*, *Piper marginatum*, bioinseticides.

Efeito ovicida de óleos essenciais de 18 espécies brasileiras de *Piper*: controlando *Anticarsia gemmatalis* (Lepidoptera, Erebidae) no estágio inicial de desenvolvimento

RESUMO. A toxicidade dos óleos essenciais (OEs) de 18 espécies de Piperaceae brasileiras foi avaliada sobre ovos da lagarta da soja, *Anticarsia gemmatalis*. Os óleos foram extraídos por destilação de arraste de vapor d´água e as diluições foram feitas para os bioensaios em concentrações de 0,25; 0,5; 1,0; 2,0 e 4,0%. Todos os OEs reduzidam a eclosão larval. As menores concentrações letais foram observadas em *Piper fuligineum* (SP), *Piper mollicomum* "quimiotipo 1" (SP), *Piper mosenii* (PR), *Piper aduncum* (PA) e *Piper marginatum* (PA). A atividade ovicida observada está relacionado com a toxicidade potencial de alguns compostos, especialmente o dilapiol, a miristicina, a asaricina, o espatulenol e a piperitona. De acordo com nossos resultados, os OEs de 16 espécies de *Piper* brasileiras têm potencial para uso como inseticidas vegetais bioracionais.

Palavras-chave: *Piper abutiloides*, *Piper fuligineum*, *Piper marginatum*, bioinseticidas.

Introduction

The velvetbean caterpillar, *Anticarsia gemmatalis* Hübner (Lepidoptera: Eribidae), is the primary soybean defoliator in Brazil (Panizzi, Oliveira, & Silva, 2004). This species also damages other crops of economic importance (Rahman, Bridges, Chapin, & Thomas, 2007). It is mainly controlled using synthetic insecticides, but other control methods include the use of transgenic crop plants and the soil-dwelling bacterium, *Bacillus thuringiensis* Berliner (McPherson & Macrae, 2009; Castro et al., 2013).

However, new approaches are needed to reduce risks to the environment and natural enemies and to avoid or delay the onset of insecticide resistance (Loureiro, Moino-Junior, Arnosti, & Souza, 2002; Petroski & Stanley, 2009; Rampelotti-Ferreira et al., 2010). Additionally, they may be safer and more environmentally acceptable. Thus, strategies for insect management should include alternatives to conventional insecticides. The use of plant-based insecticides is an alternative for the control of lepidopteran pests primarily by having low toxicity and short persistence in the environment (Costa, Silva, & Fiuza, 2004). In this context, plants of the family Piperaceae may be a promising alternative for the control of insect pests because they contain active principles with high insecticidal potential (Fazolin, Estrela, Catani, & Alécio, 2005; Fazolin, Estrela, Catani, Alécio, & Lima, 2007; Estrela, Fazolin, Catani, Alécio, & Lima, 2006; Barbosa et al., 2012).

The analysis and identification of chemical compounds of plant origin that are active against insects are important as they allow the discovery of new groups of plants with insecticidal potential, and they provide new perspectives for the synthesis and development of new bioactive compounds (Scott, Jensen, Philogène, & Arnason, 2008). Despite the widespread occurrence of the Piperaceae species in Brazil, research on its bioactivity against agricultural insect pests is incipient. In addition, pest management is performed during the phase in which the insects are causing damage to the crop, and in this case, *A. gemmatalis* is in the larval stage, where the larvae feed mainly on soybean leaves (Lourenção, Reco, Braga, Valle, & Pinheiro, 2010; Moscardi et al., 2012; Franco et al., 2014).

However, few studies have been conducted on the ovicidal effect of Piperaceae on any insect pest group (Laurent et al., 1997; Fazolin et al., 2005; Scott et al., 2008; Carneiro, Pereira, & Galbiati, 2011). Due to the importance of identifying alternative, environmentally sound methods for agricultural pest control, we determined the ovicidal action of essential oils (EOs) from leaves of Piperaceae species of various Brazilian regions against *Anticarsia gemmatalis* eggs.

Material and methods

The eggs used in the bioassays were obtained from a colony of *A. gemmatalis* maintained in the Laboratory of Integrated Control of Insects (LCII), and the oils were extracted by hydrodistillation in the Vegetable Ecophysiology Laboratory, both at the Federal University of Paraná (UFPR), Curitiba, Paraná State, Brazil.

Essential oils extraction - To obtain the EOs, we used dry leaves of *Piper* species collected from various Brazilian regions (Table 1, Figure 1).

Chromatographic analysis

Chromatographic analysis was performed in the Laboratory of Ecophysiology Vegetable and the Laboratory of Natural Products and Chemical Ecology (LAPEQ), both at the UFPR. The EOs were subjected to analysis by gas chromatography coupled to a flame ionization detector (HP- Agilent 7890A GC-FID) and by gas chromatography coupled to mass spectrometry (MS) (60–240°C at 3°C min. rate) using a fused-silica capillary column (30 m x 0.25 mm i.d. x 0.25 μm) coated with DB-5. The injector and detector temperatures were 280°C. Hydrogen was used as a carrier gas at a flow rate of 2.4 mL $min.^{-1}$; injection was in the split mode (1:20), and the injection volume was 1.0 μL. MS spectra were obtained using electron impact at 70 eV, with a scan interval of 0.5 s and mass range from 40 to 550 *m/z*. The initial identification of components of the EOs was carried out by comparison with previously reported values of retention indices, which was obtained by co-injection of oil samples and C11–C24 linear hydrocarbons and calculated according to the equation of Van den Dool and Kratz (1963). Subsequently, the MS acquired for each component was matched with those stored in the Wiley/NBS mass spectral library of the GC-MS system and with other published mass spectral data (Adams, 2007).

Table 1. *Piper* species evaluated and collection sites (geographic coordinates).

Brazilian regions	*Piper* species (State)	Geographic coordinates* - elevation (m)
North region	*Piper aduncum* L. (PA)	7°07'43.56"S 55°23'22.09"W - 231 m
	Piper malacophyllum Prels. (PA)	7°06'56.31"S 55°24'22.19"W - 210 m
	Piper marginatum L. (PA)	7°07'43.56"S 55°23'22.09"W - 231 m
South region	*Piper. caldense* C. DC. "chemotype 2" (PR)	25°29'41.6"S 49°00'50.6"W – 528 m
	Piper gaudichaudianum Kunth. "chemotype 1" (PR)	25°32'59.07"S 49°14'13.09"W - 919 m
	Piper gaudichaudianum "chemotype 2" (PR)	25°29'41.6"S 49°00'50.6"W – 528 m
	Piper mikanianum (Kunth) Steud. (PR)	25°33'21.53"S 49°13'51.34"W - 908 m
	Piper mosenii C. DC. (PR)	25°29'41.6"S 49°00'50.6"W – 528 m
Southeast region	*Piper abutiloides* Kunth. (SP)	24°12'11.27"S 48°33'42.36"W - 858 m
	Piper amalago L. (SP)	22°50'42.03"S 48°25'34.04"W - 763 m
	Piper arboreum Aubl. (SP)	22°51'37.19"S 48°26'14.32"W - 798 m
	Piper caldense "chemotype 1" (SP)	24°01'18.42"S 47°31'46.37"W - 722 m
	Piper crassinervium Kunth. (SP)	22°51'37.19"S 48°26'14.32"W - 798 m
	Piper mollicomum Kunth. "chemotype 1" (SP)	22°53'32.57"S 48°28'57.45"W - 853 m
	Piper mollicomum "chemotype 2" (SP)	22°53'21.40"S 48°28'12.23"W - 822 m
	Piper fuligineum Kunth. (SP)	22°53'32.57"S 48°28'57.45"W - 853 m
	Piper solmsianum C. DC. (SP)	24°01'16.05"S 47°31'48.41"W - 718 m
	Piper lhotzkyanum Kunth. (SP)	22°50'42.03"S 48°25'34.04"W - 763 m
Midwest region	*Piper tuberculatum* Jacq. (MT)	4°37'29.32"S 57°29'09.10"W - 385 m
	Piper hispidum Sw. (GO)	16°29'47.47"S 49°16'50.45"W - 825 m
	Piper umbellatum L. (GO)	16°31'00.40"S 49°16'15.16"W - 741 m

*Datum WGS84.

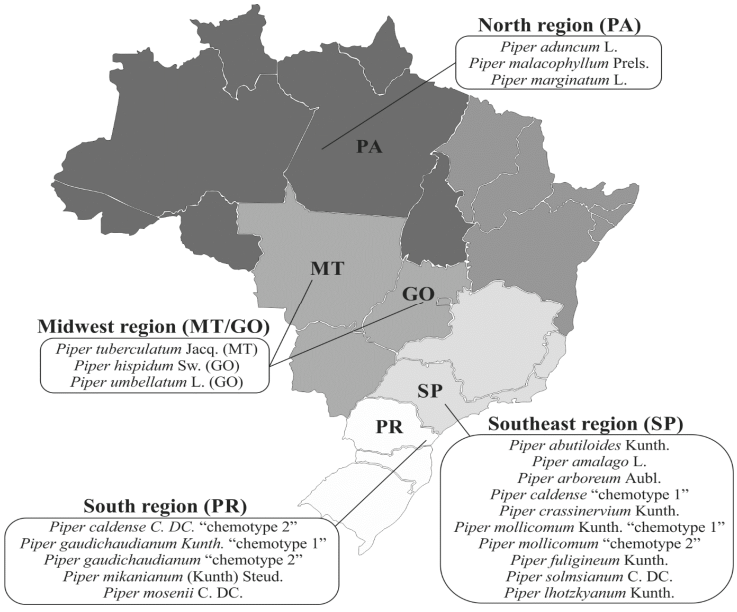

Figure 1. *Piper* species evaluated and collection regions (Brazilian states).

The species were identified, and herbarium specimens were deposited at Universidade do Estado de Mato Grosso, Campus Universitário de Tangará da Serra (UNEMAT/CUTS) in Herbarium Tangará (TANG). Collected leaves were stored in a greenhouse for 96h at 50°C to dry and then ground with a mill to obtain the leaf powder. The milled material was extracted by hydrodistillation. For each oil extraction, 50 g of vegetable powder were placed in a glass flask (2 L) containing 1 L of distilled water. The flask was heated in a heating mantle and boiled for four hours to obtain the EOs.

Ovicidal effects of essential oils on *Anticarsia gemmatalis* eggs

To evaluate the activity of EOs from Piperaceae leaves on *A. gemmatalis* eggs, the bioassays were performed by spraying eggs with an airbrush up to 24 hours after oviposition. Five oil concentrations (0.25, 0.5, 1.0, 2.0, and 4.0% diluted with acetone P.A.) and two control treatments (distilled in water and acetone P.A.) were evaluated, totalling seven treatments for each *Piper* species. The sprayings were performed with a calibrated airbrush at a pressure of 20 psi, which enabled the deposition of 1.5 mg cm^{-2} of each solution/concentration. Ten replicates containing ten eggs glued on blue paper were sprayed in each concentration, and the eggs were left to dry at room temperature. Each replicate was placed in glass tubes (10 cm x 1 cm), and all treatments were maintained in climatic chambers (Eletrolab Model EL 202) at 25 ± 1.0°C (SD), 70 ± 10% RH (SD), and a photoperiod of 12h L:D. Larval hatching was evaluated on the third day after oviposition, which is the normal time for emergence of *A. gemmatalis* caterpillars at 25°C (Magrini, Botelho, & Silveira Neto, 1999).

Statistical analysis

For the statistical analysis, we used the results obtained three days after treatment in each EO. After this time, the hatching of *A. gemmatalis* caterpillars was recorded. The results were compared using an analysis of variance (ANOVA), and means were classified with the Scott Knott test ($p < 0.05$). Lethal concentrations causing 50, 75, and 90% mortality of the eggs (LC_{50}, LC_{75}, and LC_{90}) were calculated by Probit analysis (Finney, 1971) using Statistica software (version 7).

Results

All evaluated EOs reduced the larval hatching of *A. gemmatalis* compared with control treatments (water and acetone), except for *P. solmsianum* (SP) and *P. hispidum* (GO), which did not show significant differences in the average number of hatched larvae. The results showed that there were significant differences among *Piper* species on the ecclosion of *A. gemmatalis* eggs (Table 2). To consider a product as ovicidal, it should inhibit hatching by at least 75% (Picollo & Zerba, 1997). The *Piper* species showing ovicidal effects (mortality of eggs greater than 75%) at each concentration were *P. fuligineum* (SP) from the lowest concentration (0.25%); *P. aduncum* (PA), *P. mollicomum* 'chemotype 1' (SP) and *P. mosenii* (PR) from 0.5%; *P. caldense* 'chemotype 1' (SP) and *P. marginatum* (PA) at concentrations of 1.0%; *P. arboreum* (SP), *P. gaudichaudianum* (PR), *P. lhotzkyanum* (SP), *P. mikanianum* (PR), *P. mollicomum* 'chemotype 2' (SP), *P.*

caldense 'chemotype 2' (PR) and *P. tuberculatum* (MT) at 2.0%; and only *P. abutiloides* (SP) and *P. amalago* (SP) in the highest concentration (4.0%) (Table 2). Data concerning the lethal concentrations that inhibit hatching of 50, 75, and 90% of *A. gemmatalis* eggs (LC_{50}, LC_{75}, and LC_{90}) are reported in Table 3. The LC_{50s} calculated after 72 hours ranged from 0.4% (*P. fuligineum* oil) to 1091.4% (*P. hispidum* oil). The lowest LC_{50s} were observed for *P. fuligineum* (SP), *P. mollicomum* 'chemotype 1' (SP), *P. mosenii* (PR), *P. aduncum* (PA) and *P. marginatum* (PA), in this order. Similar patterns were observed for the LC_{75} and LC_{90} with *P. caldense* 'chemotype 1' and *P. mollicomum* 'chemotype 2' being included among the species with smaller lethal concentrations (Table 3). The major chemical compounds found in each species of Piperaceae evaluated are shown in Table 4.

Table 2. Mean (± SD) number of caterpillars hatching (n= 10) after spraying of essential oils from dried leaves of different species of Piperaceae on *Anticarsia gemmatalis* eggs three days after treatment in each essential oil concentration.

Piper species (Brazilian state)	Treatments/Oil concentrations*							p	CV (%)
	Water	Acetone	0.25%	0.5%	1.0%	2.0%	4.0%		
P. abutiloides (SP)	9.6 ± 0.5 a	9.1 ± 0.9 a	8.0 ± 0.7 Bb	8.0 ± 0.9 Bb	8.0 ± 0.9 Bb	5.3 ± 3.2 Cc	0.0 ± 0.0 Dd	<0.0001	20.41
P. aduncum (PA)	9.6 ± 0.5 a	9.1 ± 0.9 a	3.3 ± 1.7 Ec	2.2 ± 1.4 Dd	5.8 ± 1.8 Cb	1.4 ± 1.1 De	0.0 ± 0.0 Df	<0.0001	16.04
P. amalago (SP)	9.6 ± 0.5 a	9.1 ± 0.9 a	9.0 ± 1.1 Aa	8.4 ± 1.1 Bb	8.5 ± 1.2 Bb	7.8 ± 1.4 Bb	0.0 ± 0.0 Dc	<0.0001	13.23
P. arboreum (SP)	9.6 ± 0.5 a	9.1 ± 0.9 a	8.9 ± 0.7 Aa	8.2 ± 1.0 Bb	8.0 ± 1.3 Bb	2.2 ± 2.2 Dc	0.0 ± 0.0 Dd	<0.0001	13.80
P. caldense 'chemotype 1' (SP)	9.6 ± 0.5 a	9.8 ± 0.4 a	9.5 ± 0.7 Aa	9.5 ± 3.0 Aa	0.0 ± 0.0 Fb	0.0 ± 0.0 Eb	0.0 ± 0.0 Db	<0.0001	8.89
P. caldense 'chemotype 2' (PR)	9.6 ± 0.5 a	9.1 ± 0.9 a	5.6 ± 1.6 Db	5.6 ± 1.7 Cb	4.3 ± 1.3 Dc	1.7 ± 1.1 Dd	0.0 ± 0.0 De	<0.0001	21.74
P. crassinervium (SP)	9.6 ± 0.5 a	9.1 ± 0.9 a	7.3 ± 1.1 Bb	6.5 ± 1.2 Cc	6.3 ± 0.9 Cc	0.2 ± 0.6 Ed	0.0 ± 0.0 Dd	<0.0001	15.27
P. fuligineum (SP)	9.6 ± 0.5 a	9.1 ± 0.9 a	2.3 ± 1.4 Fb	2.3 ± 1.6 Db	2.8 ± 1.8 Eb	0.0 ± 0.0 Ec	0.0 ± 0.0 Dc	<0.0001	30.05
P. gaudichaudianum 'chem. 1' (PR)	9.6 ± 0.5 a	9.1 ± 0.9 a	8.6 ± 1.2 Bb	8.4 ± 1.0 Bb	8.0 ± 1.2 Bb	0.1 ± 0.3 Ec	0.0 ± 0.0 Dc	<0.0001	13.80
P. gaudichaudianum 'chem. 2' (PR)	9.6 ± 0.5 a	9.1 ± 0.9 a	8.3 ± 0.7 Bb	8.0 ± 1.3 Bb	8.3 ± 1.2 Bb	7.8 ± 1.2 Bb	7.8 ± 2.0 Bb	0.0082	14.42
P. hispidum (GO)	9.6 ± 0.5 a	9.8 ± 0.4 a	9.7 ± 0.7 Aa	9.7 ± 0.5 Aa	9.7 ± 0.5 Aa	9.2 ± 0.9 Aa	9.8 ± 0.4 Aa	0.2907	6.06
P. lhotzkyanum (SP)	9.6 ± 0.5 a	9.1 ± 0.9 a	9.3 ± 0.7 Aa	9.5 ± 0.8 Aa	9.2 ± 0.8 Aa	0.0 ± 0.0 Eb	0.0 ± 0.0 Db	<0.0001	9.54
P. malacophyllum (PA)	9.6 ± 0.5 a	9.1 ± 0.9 a	5.4 ± 1.8 Dc	7.6 ± 2.0 Bb	7.7 ± 1.6 Bb	7.2 ± 1.8 Bb	4.4 ± 1.9 Cc	<0.0001	23.01
P. marginatum (PA)	9.6 ± 0.5 a	9.1 ± 0.9 a	6.4 ± 1.6 Cb	6.0 ± 1.9 Cb	2.1 ± 1.5 Ec	0.1 ± 0.3 Ed	0.0 ± 0.0 Dd	<0.0001	26.20
P. mikanianum (PR)	9.6 ± 0.5 a	9.1 ± 0.9 a	8.9 ± 1.4 Aa	7.7 ± 0.9 Bb	8.1 ± 1.3 Bb	0.8 ± 1.2 Ec	0.0 ± 0.0 Dc	<0.0001	16.06
P. mollicomum "chemotype 1" (SP)	9.6 ± 0.5 a	9.1 ± 0.9 a	4.0 ± 1.1 Eb	0.7 ± 1.0 Ed	2.5 ± 1.8 Ee	0.0 ± 0.0 Ed	0.0 ± 0.0 Dd	<0.0001	25.91
P. mollicomum "chemotype 2" (SP)	9.6 ± 0.5 a	9.1 ± 0.9 a	9.2 ± 0.8 Aa	8.2 ± 2.3 Bb	3.4 ± 2.0 Dc	0.0 ± 0.0 Ed	0.0 ± 0.0 Dd	<0.0001	18.80
P. mosenii (PR)	9.6 ± 0.5 a	9.1 ± 0.9 a	4.7 ± 1.8 Db	2.1 ± 1.1 Dd	3.6 ± 1.2 Dc	0.1 ± 0.3 Ee	0.1 ± 0.3 De	<0.0001	23.80
P. solmsianum (SP)	9.6 ± 0.5 a	9.8 ± 0.4 a	9.7 ± 0.5 Aa	9.7 ± 0.6 Aa	9.9 ± 0.3 Aa	9.7 ± 0.7 Aa	9.7 ± 0.5 Aa	0.919	5.39
P. tuberculatum (MT)	9.6 ± 0.5 a	9.1 ± 0.9 a	9.8 ± 0.6 Aa	9.1 ± 2.3 Aa	4.1 ± 2.8 Db	0.0 ± 0.0 Ec	0.0 ± 0.0 Dc	<0.0001	20.58
P. umbellatum (GO)	9.6 ± 0.5 a	9.1 ± 0.9 a	7.8 ± 1.5 Bb	8.0 ± 1.1 Bb	7.7 ± 0.9 Bb	7.9 ± 1.6 Bb	7.5 ± 1.4 Bb	0.0008	14.56
P	-	-	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001		
CV (%)	-	-	16.11	17.33	22.92	40.93	37.10		

*Means followed by the same lowercase letter in the lines (comparing the concentrations for the same species) and capital letters in columns (comparing the same concentration for different species) do not differ by Scott Knott test ($p < 0.05$). Means highlighted in bold show concentrations with ovicidal activity (Picollo & Zerba, 1997).

Table 3. Lethal concentrations of essential oils from leaves of *Piper* species to make unviable 50, 75, and 90% (LC_{50}, LC_{75}, and LC_{90}) of *Anticarsia gemmatalis* eggs.

Piper species (Brazilian state)	Lethal Concentration (%)		
	LC_{50} (*ci*[1])	LC_{75} (*ci*)	LC_{90} (*ci*)
P. abutiloides (SP)	1.9 (1.8 - 2.0)	2.9 (2.8 - 3.1)	3.6 (3.4 - 3.8)
P. aduncum (PA)	0.6 (0.3 - 0.9)	2.0 (1.6 - 2.3)	2.8 (2.4 - 3.3)
P. amalago (SP)	2.2 (2.1 - 2.3)	3.3 (3.1 - 3.4)	3.9 (3.7 - 4.1)
P. arboreum (SP)	1.7 (1.4 - 1.9)	2.6 (2.3 - 2.9)	3.19 (2.8 - 3.6)
P. caldense "chem. 1" (SP)	1.2 (0.6 - 1.8)	1.8 (1.1 - 2.4)	2.1 (1.4 - 2.9)
P. caldense "chem. 2" (PR)	1.1 (0.7 - 1.4)	2.2 (1.8 - 2.6)	2.8 (2.3 - 3.3)
P. crassinervium (SP)	1.3 (0.9 - 1.7)	2.2 (1.7 - 2.6)	2.7 (2.1 - 3.3)
P. fuligineum (SP)	0.4 (0.3 - 1.1)	1.5 (0.9 - 2.0)	2.1 (1.4 - 2.8)
P. gaudichaudianum "chem. 1" (PR)	1.5 (1.2 - 1.9)	2.3 (1.9 - 2.8)	2.8 (2.2 - 3.4)
P. gaudichaudianum "chem. 2" (PR)	12.9 (12.4 - 13.3)	21.6 (20.9 - 22.4)	26.9 (26.0 - 27.8)
P. hispidum (GO)	1091.4 (1095.2 - 1087.7)	1683.2 (1689.0 - 1677.3)	2038.2 (2045.3 - 2031.1)
P. lhotzkyanum (SP)	1.6 (1.3 - 2.0)	2.4 (1.9 - 2.9)	2.9 (2.3 - 3.4)
P. malacophyllum (PA)	3.7 (3.6 - 3.8)	6.8 (6.6 - 6.9)	8.6 (8.3 - 8.8)
P. marginatum (PA)	1.0 (0.4 - 1.5)	1.8 (1.2 - 2.4)	2.3 (1.6 - 3.0)
P. mikanianum (PR)	1.6 (1.2 - 1.9)	2.4 (2.0 - 2.8)	2.9 (2.4 - 3.4)
P. mollicomum "chem. 1" (SP)	0.4 (0.3 - 1.2)	1.4 (0.8 - 2.0)	2.0 (1.3 - 2.7)
P. mollicomum "chem. 2" (SP)	1.3 (0.8 - 1.8)	2.0 (1.5 - 2.6)	2.5 (1.8 - 3.1)
P. mosenii (PR)	0.6 (0.0 - 1.1)	1.7 (1.2 - 2.2)	2.4 (1.8 - 3.0)
P. solmsianum (SP)	765.7 (771.1 - 760.3)	1172.2 (1180.5 -1164.0)	1416.1 (1426.1 - 1406.2)
P. tuberculatum (MT)	1.4 (1.0 - 1.9)	2.1 (1.6 - 2.7)	2.6 (1.9 - 3.2)
P. umbellatum (GO)	12.0 (11.6 - 12.3)	20.6 (20.0 - 21.2)	25.8 (25.1 - 26.6)

[1]*ci*: confidence interval (- 95% and + 95%). Values highlighted in bold show the minors lethal concentrations ($LC_{50} > 1\%$, $LC_{75} > 2\%$, and $LC_{90} > 2.5\%$).

Table 4. Main chemical compounds found in the *Piper* species tested against *Anticarsia gemmatalis* eggs.

RI*	Compounds	*Piper* species**/concentration of each compound (%)																				
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
932	α-pinene	-	-	-	-	6.04	-	-	-	-	9.66	-	-	-	-	-	-	-	-	7.80	-	-
974	β-pinene	-	-	-	-	5.17	-	-	-	-	13.18	-	-	-	-	-	-	-	5.78	-	-	-
1014	α-terpinene	-	-	-	-	-	-	-	-	-	-	-	-	13.85	-	-	-	-	-	-	-	-
1022	o-cimene	-	-	-	-	-	-	-	-	-	-	-	-	6.18	-	-	-	-	-	-	-	-
1024	limonene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5.05	-	-	-
1025	β-phellandrene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1044	(E)-β-ocimene	-	8.16	-	-	-	-	-	-	-	-	-	-	-	6.14	-	4.39	-	-	-	-	-
1054	γ-terpinene	-	-	-	-	-	-	-	-	-	-	-	-	21.46	-	-	-	-	-	-	-	-
1086	terpinolene	-	-	-	-	-	-	-	-	-	-	-	-	8.18	-	-	-	11.34	-	-	-	-
1249	piperitone	-	7.82	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1374	α-copaene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		5.36	-
1417	(E)-caryophyllene	-	-	-	9.5	8.72	-	7.21	-	4.31	17.84	-	8.82	-	-	-	-	-	16.82	9.16	7.87	5.39
1457	allo-aromadendrene	5.53	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1484	germacrene B	-	-	-	20.42	6.79	-	-	-	-	-	6.08	7.46	-	6.11	-	6.38	-	-	-	8.38	-
1489	β-selinene	-	-	-	-	-	-	-	-	-	-	-	-	5.93	-	-		-	-	-	-	16.12
1495	asaricine	-	11.86	-	-	-	-	-	10.80	-	-	-	-	-	-	-	7.21	-	-	-	-	-
1500	bicyclogermacrene	6.33	-	15.78	21.31	-	-	8.89	5.64	5.12	6.39	11.49	21.71	5.39	-	-	20.41	-	-	32.47	-	10.64
1505	(E, E)-α-farnesene	5.47	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1517	myristicin	22.7	12.61	-	-	-	66.75	-	-	-	-	-	-	-	15.75	-	-	-	-	-	-	-
1518	δ-cadinene	-	-	-	-	6.11	-	-	6.69	6.86	-	-	-	-	-	-	-	9.12	-	-	-	-
1537	α-cadinene	-	-	-	-	-	-	-	-	-	-	-	-	-	7.98	-	-	-	-	-	-	-
1555	elemicina	-	-	-	-	-	-	-	-	-	-	-	-	-	-	13.21	-	-	-	-	-	-
1562	(E)-nerolidol	-	-	-	-	-	-	11.74	-	4.02	5.03	-	-	-	-	-	-	-	-	-	-	-
1574	germacrene D-ol	-	-	5.13	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1577	spathulenol	-	-	-	-	-	-	-	4.14	-	-	-	7.22	-	-	-	-	21.08	-	7.54	-	-
1582	caryophyllene oxide	-	-	-	-	-	-	-	-	-	-	-	13.97	-	-	-	-	-	9.82	-	6.35	-
1586	Thujopsan-2-α-ol	-	-	-	-	-	-	-	4.32	-	-	-	-	-	-	-	-	-	-	-	-	-
1608	epoxy II humulene		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1620	dill apiole		6.54	-	-	-	-	-	3.82	-	-	-	-	-	-	-	-	-	-	-	-	-
1624	1-epi-cubenol		-	-	-	-	-	-	-	24.22	-	-	-	-	-	-	-	-	-	-	-	-
1652	α-cadinol		-	5.56	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1654	4,6-dimethyl-5-vinyl-1,2-benzodioxide		-	-	-	-	-	-	-	-	-	-	-	-	-	13.9	-	-	-	-	-	-
1683	cadalene		-	-	-	-	-	-	-	33.73	-	-	-	-	-	-	-	-	-	-	-	-
1700	eudesm-7 (11) -en-4-ol		-	-	-	-	-	-	-	-	-	-	-	-	-	26.4	-	-	-	-	-	-
1759	ciclocolorenone		-	-	-	-	-	-	-	-	-	-	-	-	-	9.58	-	-	-	-	-	-

*RI= retention index.**_Piper_ species: 1- *P. abutiloides* (SP); 2- *P. aduncum* (PA); 3- *P. amalago* (SP); 4- *P. arboreum* (SP); 5- *P. caldense* (PR); 6- *P. caldense* (SP); 7- *P. crassinervium* (SP); 8- *P. fuligineum* (SP); 9- *P. gaudichaudianum* "chemotype 1" (PR); 10- *P. gaudichaudianum* "chemotype 2" (PR); 11- *P. hispidum* (MT); 12- *P. lhotzkyanum* (SP); 13- *P. malacophyllum* (PA); 14- *P. marginatum* (PA); 15- *P. mikanianum* (PR); 16- *P. mollicomum* "chemotype 1" (SP); 17- *P. mollicomum* "chemotype 2" (SP); 18- *P. mosenii* (PR); 19- *P. solmsianum* (SP); 20- *P. tuberculatum* (MT); 21- *P. umbellatum* (SP).

Discussion

The toxicity of some species such as *P. aduncum* against different orders of insects of medical and agricultural importance has already been reported (Estrela et al., 2006; Silva, Ribeiro, Souza, & Correa, 2007; Scott et al., 2008; Misni, Othman, & Sulaiman, 2011; Souto, Harada, Andrade, & Maia, 2012; Piton, Turchen, Butnariu, & Pereira, 2014; Turchen, Piton, Dall'Oglio, Butnariu, & Pereira, 2016a). However, the egg stage is probably the least studied in terms of susceptibility to chemicals, and the few studies that assessed the vulnerability of eggs were often reported by accident, arising from the application of insecticides to other developmental phases of insects (Smith & Salkeld, 1966). Past studies report that mineral oils act primarily as ovicides by depressing the respiratory rate when applied directly to moth eggs (Riedl, Halaj, Kreowski, Hilton, & Westigard, 1995). The length of respiratory expression and the dose of oil in contact with the egg are critical to provoke mortality (Smith & Pearce, 1948). Other studies have reported that the ovicidal nature of the oils is due to the natural tendency of oils to block the oxygen supplied to the developing embryo or due to the toxicity of some inherent chemical constituents of the oil (Rajapakse & Senanayake, 1997).

In general, ovicidal activity is directly proportional to the increase in the concentration tested. Considering previous information, the ovicidal effects of the EOs evaluated in our study can be attributed either to physical or chemical properties. Physically, when the oils come into contact with the surface of the eggs, they can cover the areas of gas exchange between the embryo and the external environment (corium and micropyles), thus interfering in the normal embryo development. Chemically, the compounds present in each oil may exhibit different toxicity rates and can operate concurrently with the physical properties of the oils, thus causing the death of the eggs.

This characteristic was observed for some of the Piperaceae oils tested in our study. We observed that

some compounds in particular (or perhaps synergistically) may be acting on *A. gemmatalis* eggs and causing their death. The EOs that showed bioactivity contained among the most abundant compounds, mainly asaricine (*P. aduncum*, *P. fuligineum* and *P. mollicomum* 'chemotype 1'), myristicin (*P. aduncum*, *P. abutiloides*, *P. Caldense*, and *P. marginatum*), spathulenol (*P. fuligineum*, *P. lhotzkyanum*, *P. mollicomum* SP, and *P. solmsianum*), (E)-caryophyllene and germacrene B (*P. arboreum* and *P. tuberculatum*), dillapiol (*P. aduncum* e *P. fuligineum*), (E)-β-ocimene (*P. aduncum*, *P. Marginatum*, and *P. mollicomum* 'chemotype 1'), limonene (only in *P. mosenii*), (E)-nerolidol (only in *P. crassinervium*), piperitone (*P. aduncum*), 1-epi-cubenol and cadalene (only in *P. gaudichaudianum* 'chemotype 1'), 4,6-dimethyl-5-vinyl-1,2-benzodioxide, eudesm-7 (11)-en-4-ol and ciclocolorenone (only in *P. mikanianum*), α-copaene (only in *P. tuberculatum*), and (E, E)-α-farnesene and allo-aromadendrene (only in *P. abutiloides*) (Table 4).

Some compounds, such as (E)-caryophyllene and bicyclogermacrene, were discarded as possible agents of egg mortality, even when they were in large quantities in some oils, as recorded for *P. caldense* 'chemotype 2', *P. crassinervium*, *P. gaudichaudianum* 'chemotype 2', *P. lhotzkyanum*, *P. solmsianum*, *P. amalago*, *P. Hispidum*, and *P. umbellatum* (Table 4). Apparently, these compounds are common among Piperaceae, as observed in other chemical constitutions of several *Piper* species (Santos, Moreira, Guimarães, & Kaplan, 2001; Mundina et al., 2001; Cruz, Cáceres, Álvarez, Apel, & Henriques, 2011; Santana et al., 2015).

Among the 21 chemotypes of Piperaceae tested, *P. aduncum*, *P. caldense*, *P. fuligineum*, *P. marginatum*, *P. mollicomum* chemotype 1, and *P. mosenii* were the most effective against *A. gemmatalis* eggs. *Piper aduncum* and *P. marginatum* presented compounds already reported as insecticides in previous works, such as dillapiol, asaricine (sarisan), piperitone and myristicin (Bizzo et al., 2001; Morais et al., 2007; Qin, Huang, Li, Chen, & Peng, 2010; Souto et al., 2012; Santana et al., 2015; Ribeiro, Camara, & Ramos, 2016; Krinski & Foerster, 2016). For *P. caldense*, *P. fuligineum*, *P. Mollicomum*, and *P. mosenii,* there are no studies regarding the insecticidal activity, and our work is the first to test the ovicidal activity against a lepidopteran pest of importance in Brazilian agriculture.

It was shown that the chemical compounds present in these Piperaceae can be further evaluated among the isolation of active molecules to develop new botanical formulations, especially when we consider that the majority of these substances has been reported as potential insecticides in many other studies conducted worldwide (Santos et al., 2010; 2011; 2013; Cáceres & Kato, 2014, Brito, Baldin, Silva, Ribeiro, & Vendramim, 2015; Krinski & Foerster, 2016, Sanini et al. 2017, Turchen, Hunhoff, Paulo,Souza, Pereira, 2016b) and other plant families (Isman, 2000; Koul, Walia, & Dhaliwal, 2008; Tripathi, Upadhyay, Bhuiyan, & Bhattacharya, 2009; Coitinho, Oliveira, Gondim-Júnior, & Câmara, 2010; Ntalli & Menkissoglu-Spiroudi, 2011, Zoubiri & Baaliouamer, 2011; Baskar & Ignacimuthu, 2012; Baskar, Muthu, Raj, Kingsley, & Ignacimuthu, 2012; Krishnappa & Elumalai, 2012; Cáceres & Kato, 2014, Krinski & Massaroli, 2014; Krinski, Massaroli, & Machado, 2014; Backiyaraj et al., 2015; Massaroli, Pereira, & Foerster; 2016; Costa, Santana, Oliveira, & Serrão, 2017).

The effects of EOs from *Piper* species on egg hatching of *A. gemmatalis* were evaluated, and their respective major compounds were reported as alternative plant products with effectiveness to control insect pests. The literature reports that there are many phytochemical studies on several species of *Piper*, demonstrating the presence of a variety of secondary metabolites, including alkaloids, amides, propenilfenóis, lignins, neoligninas, terpenes, steroids, kawapirenos, chalcones, dihydrochalcones, flavones, and flavonones (Dyer & Palmer, 2004; Tchoumbougnang et al., 2009).

Thus, the use of these oils can affect different functions in the insects, possibly due to the synergism that occurs in unique, natural and complex mixtures that may decrease the resistance of these organisms (Ntalli & Menkissoglu-Spiroudi, 2011).

Field and semi-field studies must be conducted to assess whether the same pattern of results obtained in laboratory studies is maintained in the field, both for *A. gemmatalis* and for other insects and food crops (Albuquerque, 1993; Krinski & Pelissari, 2012; Krinski, Favetti, & Butnariu, 2012; Favetti, Krinski, & Butnariu, 2013; Krinski, 2013; Krinski, 2015; Krinski & Godoy, 2015; Krinski, Foerster, & Grazia, 2015; Krinski & Foerster, 2017; Martins & Krinski, 2016).

Therefore, more studies are needed to search for new plant species with bioactive principles as well as for the chemical synthesis of effective ingredients and identification of the target sites of the toxic molecule. There is a lack of major commitment by the chemical industry to promote research and development of new biopesticides, based on the results obtained for plants considered bio-insecticides. Such studies have increased in recent years, however, without the concomitant development of new compounds for use in agriculture (Isman & Grieneisen, 2014).

Conclusion

The ovicidal activity observed in our study may indicate the potential toxicity of the main chemical components found in 16 *Piper* species tested and the possible synergistic action among these compounds. Based on the ovicidal effect and lethal concentrations, the species *P. aduncum* (PA), *P. caldense* (SP), *P. fuligineum* (SP), *P. marginatum* (PA), *P. mollicomum* (SP), and *P. mosenii* (PR) were the most promising for the management of *A. gemmatalis* in egg stage.

Thus, we noticed that the Piperaceae species showed to be toxic to *A. gemmatalis* eggs by reducing or inhibiting larval hatching of treated eggs.

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