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ORIGINAL ARTICLE

Fumigant insecticidal activity of plant essential oils against pest blister beetle *Epicauta atomaria* (Germar) (Coleoptera: Meloidae)

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Abstract

Epicauta atomaria is a phytophagous insect pest of agricultural crops controlled through the application of synthetic insecticides that cause serious human health and environmental damage. Bioinsecticides formulated from essential plant oils are suitable as an alternative to synthetic insecticides in the control of pest insects. For this reason, the objective of this work is to determine the fumigant insecticidal activity of a screening of plant essential oils against *E. atomaria* and determine the chemical composition of the essential oils with greater toxicity. The fumigant insecticidal activity was evaluated at different concentrations; the chemical composition of the essential oils with greater toxicity was determined by gas chromatography mass spectrometry. *Mentha spicata* and *Salvia rosmarinus* essential oils showed strong fumigant activity against *E. atomaria* with LC_{50} values of 21.7 and 23.3 µL/L air, respectively, followed by *Laurus nobilis* and *Pascalia glauca* essential oils showing strong fumigant activity with LC_{50} value of 32.8 µL/L air in both cases. The major components identified in the *M. spicata* essential oil were pulegone (55.08%) and isopulegone (12.57%) and those in the *S. rosmarinus* essential oil were camphor (19.42%), 1.8-cineole (18.72%), α-pinene (15.87%) and camphene (11.88%). In conclusion, *M. spicata*, *S. rosmarinus, L. nobilis* and *P. glauca* essential oils could be considered as components in bioinsecticide formulations for future integrated pest management (IPM) programmes.

Keywords *Mentha spicata* · *Salvia rosmarinus* · Bioinsecticide · Hydrodistillation · Toxicity · Integrated pest management

Introduction

The blister beetle *Epicauta atomaria* (Germar) is a polyphagous phytophagous insect, pest of agricultural crops such as soybeans, quinoa, peanuts, potatoes, eggplant, tomatoes, peppers, chard and beets (Boito et al. 2009; Campos-Soldini and Roig-Juñent 2015). Blister beetles such as *E. atomaria* are currently controlled by the application of synthetic insecticides mainly organochlorines, pyrethroids, organophosphates and carbamates (Ghoneim 2013). However, it is known that their application poses serious health and environmental harm (de Vlaming et al. 2004; Sulak et al. 2005; Jabran et al. 2015) which is why it is necessary to develop new products for the control of this pest insect.

In this context, the development and implementation of bioinsecticides formulated from essential oils extracted from plants, for the control of pest insects, provide an effective alternative to synthetic insecticides, mainly due to their low toxicity in non-target organisms, specificity against pest insects, biodegradable nature and production from renewable resources (Isman 2000; Liu et al. 2006; Koul et al. 2008). The toxicity generated in plant essential oils against certain organisms is attributed to the presence of allelochemical compounds, mostly terpenes, ketones, aldehydes, alcohols, esters and ethers (Mudrončeková et al. 2019). Plants synthesize these compounds to protect themselves from other organisms such as insects, fungi, bacteria, viruses and other plant species (Bakkali et al. 2008; Koul et al. 2008; Mahdavikia and Saharkhiz 2015; Sadgrove and Jones 2015; Hazrati et al. 2017).

Despite the proven toxicity that plant essential oils have against numerous pest insects, to date, only the toxicity of *Lavandula dentata* essential oil against *E. atomaria* has been

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evaluated (Wagner et al. 2021). Surprisingly, the insecticidal activity of any other essential oil against this pest insect has not been assessed. For this reason, the objective of this work is to determine the fumigant insecticidal activity of a screening of plant essential oils against *E. atomaria* and to characterize the chemical composition of the essential oils with higher toxicity.

Material and methods

Plant materials

Aerial parts of plants were collected during December 2020 and January 2021 from fields and organic gardens nearby the city of Diamante, Argentina (32° 04′ 00″ S, 60° 39′ 00″ W; 14 m.a.s.l.). The aerial parts of the plants were taken to the laboratory for processing after being harvested and identified by a plant taxonomist from the Departamento de Botánica, Universidad Nacional de Entre Ríos (UNER). Voucher specimens were deposited at the Colección Botánica del Laboratorio de Ecología de la Vegetación, Centro de Investigación Científica y de Transferencia Tecnológica a la Producción (CICYTTP) (Table 1). Plant samples were dried in a room in total darkness at 23 ± 3 °C and 55 ± 6% relative humidity for further processing.

Essential oil extraction and GC–MS analysis

The dried aerial parts (300 g) of each plant were ground with an electric grinder to have small fragments subsequently subjected to hydrodistillation process with distilled water (500 mL) using a Clevenger-type apparatus for three hours. The essential oils obtained were dehydrated with sodium sulphate anhydrous and later stored in amber bottles in a refrigerator at 4 °C until chemical analysis and fumigant insecticidal activity assay. Essential oils were analysed by GC–MS using the PerkinElmer Clarus 580–SQ8 chromatography apparatus equipped with a DB–5 capillary column (30 m × 0.25 mm i.d. and 0.25 µm coating thickness). The oils were then diluted in *n*-hexane (ratio of 1:50). One µL sample was manually injected using the split mode (split ratio 1:50). The oven temperature was 60 °C for 5 min and increased to 240 °C at a rate of 5 °C/min, having a final holding time of 10 min. Injector and detector temperatures were 250 and 280 °C, respectively. Helium was used as the carrier gas at a flow rate of 1 ml/min and an electron impact at 70 eV. Mass spectra range was 50–350 m/z. To determine the retention indices (RIs) of each essential oil compound, a mixture of *n*-alkanes (C_8–C_{20}) (Sigma-Aldrich, Argentina) was injected into the GC–MS system, under the same conditions as those under which the essential oils were injected. The compounds were identified comparing their retention indices (RIs) and mass spectra with the literature data (Adams 2007) and computer libraries (NIST 2008). The relative proportion (%) of the essential oil components was calculated from the GC–MS peak areas.

Insects

Epicauta atomaria adults were collected manually from their host plants *Amaranthus hybridus*, *Amphilophium carolinae* and *Salpichroa origanifolia*, found in fields bordering the city of Diamante, Argentina (32° 04′ 00″ S, 60° 39′ 00″ W; 14 m.a.s.l.), during January and February 2021. The insects were brought to the insectary of the Entomology Laboratory, CICYTTP, Diamante, Argentina, and were placed in glass containers (50 × 30 × 50 cm) containing fresh leaf host plants. The insects were kept at 27 ± 2° C, 65 ± 5% relative humidity and with a 16:8 h light–dark cycle photoperiod.

Table 1 Plant species used in the essential oil extraction process

Plant species	Family	Plant parts	Voucher serial
Baccharis salicifolia (Ruiz & Pav.) Pers	Asteraceae	Stem and leaf	BASA/2020
Gaillardia megapotamica (Spreng.) Baker	Asteraceae	Stem and leaf	GAME/2020
Pascalia glauca Ortega	Asteraceae	Stem and leaf Flower	PAGL/2020
Solidago chilensis Meyen	Asteraceae	Leaf Flower	SOCH/2020
Xanthium stromarius L	Asteraceae	Leaf	XAST/2020
Pelargonium × *citrosum* Van leenii	Geraniaceae	Stem and leaf	PECI/2020
Laurus nobilis L	Lauraceae	Leaf	LANO/2020
Mentha spicata L	Lamiaceae	Stem and leaf	MESP/2020
Ocimum basilicum L	Lamiaceae	Stem and leaf	OCBA/2020
Salvia rosmarinus (L.) Schleid., 1852	Lamiaceae	Stem and leaf	SARO/2020

Fumigant insecticidal activity assay

The fumigant toxicity of plant essential oils against *E. atomaria* was evaluated with a similar fumigant toxicity assay to that used by Huang et al. (1997) but with modification. Five unsexed, mixed-age adult insects were placed in each 127 mL glass vial, sealed with rubber stoppers, containing a filter paper disc (1 cm diameter) at its base, to deposit the essential oils. Different amounts of pure essential oils were deposited at concentrations corresponding to 0 (negative control), 19.7, 27.6, 35.4, 47.2 and 78.7 µL/L air. Chlorpyrifos was used as positive control. Filter paper discs were covered with a fine mesh to avoid contact effect. The rubber plug was hermetically sealed with parafilm. All treatments were replicated three times. Mortality was determined after 6 h. Insects were considered dead if they showed no movement when touched with entomological forceps.

Statistical analysis

LC_{50} and LC_{90} values (lethal concentration producing 50 and 90% mortality after 6 h) were determined by Probit analysis (Finney 1971) using POLO–Plus Software (LeOra Software 2002–2014). The differences of LC_{50} and LC_{90} values were taken as significant when 95% confidence limits did not overlap.

The mortality percentage of the highest concentration used (78.7 µL/L air) was determined and analysed using Kruskal–Wallis test followed by a Conover test for post hoc comparisons (Conover 1999) at the 0.05 level of significance using InfoStat version 2018 statistical software.

Results

Fumigant insecticidal activity of plant essential oils against *E. atomaria* adults

The fumigant insecticidal activity of plant essential oils against *E. atomaria* is shown in Table 2. *Mentha spicata* and *Salvia rosmarinus* essential oils presented strong fumigant activity (LC_{50} = 21.7 and 23.3 µL/L air, respectively), *Laurus nobilis* and *Pascalia glauca* (leaves) essential oils also had a strong fumigant activity both with a LC_{50} value of 32.8 µL/L air; all these oils presented a toxicity similar to the chlorpyrifos (LC_{50} = 25.0 µL/L air). *Xanthium strumarium* and *Gaillardia megapotamica* essential oils had good fumigant activity but with LC_{50} values of 45.3 and 51.7 µL/L air, 1.8 and 2.1 times less toxic than chlorpyrifos, respectively. Moreover, *P. glauca* (flowers) and *Ocimum basilicum* essential oils showed a high mortality percentage at the highest concentration evaluated (100 and 60.0% at

Table 2 Fumigant insecticidal activity of plants essential oils after 6 h against *Epicauta atomaria* adults

Plant species	Plant parts	Mortality (%) median ± interquartile range; concentration at 78.7 µL/L air[a]	LC_{50} (µL/L)	95% confidence interval (µL/L)	LC_{90} (µL/L)	95% confidence interval (µL/L)	Slope ± SE	$(X^2)^b$
Baccharis salicifolia	Stem and leaf	0.0 ± 20.0 c	ND					
Gaillardia megapotamica	Stem and leaf	80.0 ± 40.0 abc	51.7	40.5–82.3	142.7	87.3–675.6	2.9 ± 0.8	1.14
Pascalia glauca	Leaf	100.0 ± 0.0 a	32.8	27.4–38.8	60.1	48.1–98.0	4.9 ± 1.1	2.05
	Flower	100.0 ± 20.0 ab	ND					
Solidago chilensis	Leaf	40.0 ± 40.0 bc	ND					
	Flower	20.0 ± 40.0 c	ND					
Xanthium strumarium	Leaf	100.0 ± 20.0 ab	45.3	34.3–73.8	81.4	57.0–351.5	5.0 ± 1.0	3.13
Pelargonium × citrosum	Stem and leaf	20.0 ± 0.0 c	ND					
Laurus nobilis	Leaf	100.0 ± 20.0 ab	32.8	23.1–43.3	95.7	63.0–396.0	2.8 ± 0.8	2.33
Mentha spicata	Stem and leaf	100.0 ± 0.0 a	21.7	14.7–25.7	38.5	32.0–62.2	5.1 ± 1.5	1.45
Ocimum basilicum	Stem and leaf	60.0 ± 20.0 abc	ND					
Salvia rosmarinus	Stem and leaf	100.0 ± 0.0 a	23.3	18.7–26.7	36.0	30.9–50.8	6.8 ± 1.7	0.46
Chlorpyrifos		100.0 ± 20.0 ab	25.0	14.5–31.9	67.56	48.7–191.6	3.0 ± 0.9	0.87

[a]Values (median ± interquartile range) with different letters represent significant differences between treatment groups (Kruskal–Wallis test followed by Conover post hoc comparisons, significant at $p < 0.05$ level). Kruskal–Wallis test: H = 30.29; df = 12; p = 0.0008

[b]Chi-square values, significant at $p < 0.05$ level

ND not determined

78.7 µL/L air, respectively); nevertheless, the LC_{50} of these oils could not be determined because they did not show a concentration-dependent linear behaviour. Finality, *Solidago chilensis* (leaves and flowers), *Baccharis salicifolia* and *Pelargonium × citrosum* essential oils showed a low mortality percentage that did not exceed 40% at the highest concentration evaluated.

Chemical composition of the essential oils

The yield and chemical composition of the essential oils of *M. spicata* and *S. rosmarinus* were determined due to their high fumigant toxicity. The leaves and stems of *M. spicata* and *S. rosmarinus* produced an essential oil yield of 1.3% and 0.2% (v/w) by hydrodistillation, respectively. By GC–MS analysis, 8 compounds were identified in the *M. spicata* essential oil and 12 compounds in the *S. rosmarinus* essential oil (Table 3). The major components identified in the essential oil of *M. spicata* were pulegone (55.08%) and isopulegone (12.57%); those identified in the *S. rosmarinus* essential oil were camphor (19.42%), 1,8-cineole (18.72%), α-pinene (15.87%) and camphene (11.88%).

Table 3 Chemical composition of essential oils extracted from leaves and stems of *Mentha spicata* and *Salvia rosmarinus*

Components	RI [a]	Chemical composition (%)		Identification method [b]
		M. spicata	*S. rosmarinus*	
α-Pinene	926	1.03	15.87	*I*, MS
Camphene	934		11.88	*I*, MS
β-Pinene	954	1.73		*I*, MS
α-Terpinene	966		1.36	*I*, MS
Limonene	972	3.03	5.10	*I*, MS
1.8-Cineole	974	5.11	18.72	*I*, MS, Co-GC
γ-Terpinene	987		0.73	*I*, MS
β-Terpineol	992	1.43		*I*, MS
β-Linalool	1007		1.80	*I*, MS
Camphor	1028		19.42	*I*, MS
Borneol	1040		6.71	*I*, MS
Isopulegone	1041	12.57		*I*, MS
Terpinen-4-ol	1044		1.16	*I*, MS
α-terpineol	1050		1.39	*I*, MS
Pulegone	1067	55.08		*I*, MS
Verbenone	1103	9.03		*I*, MS
Bornyl acetate	1200		1.46	*I*, MS
Total identified		**89.01**	**85.60**	

[a]RI: Linear retention indices calculated according to van Den Doll and Kratz (1963) formula in reference to *n*-alkanes (C8–C20) injected on DB–5 capillary column

[b]Identification method: I = comparison of the RI values with those cited in the ADAMS and NIST 08 libraries; MS = comparison of MS matching with the NIST 17 library; Co-GC = comparison with analytical standard

Discussion

The chemical composition of the essential oils of *M. spicata* and *S. rosmarinus* has been extensively studied. A recent review by Mahendran et al. (2021) shows that essential oils extracted from *M. spicata* have pulegone, menthone, carvone, piperitone, limonene and menthol as major components. On the other hand, a review by Borges et al. (2019) shows that oils extracted from *S. rosmarinus* contain α-pinene, camphene, 1,8-cineole, camphor, borneol and limonene as its main components. In our study, the chemical composition of *M. spicata* essential oil, with pulegone as its central compound, is similar to that reported by Gonçalves et al. (2009) and Tayarani-Najaran et al. (2013), whereas the chemical composition of *S. rosmarinus* essential oil, with camphor and 1,8-cineole as its major compounds, was similar to that determined by Jordán et al. (2013) and Laborda et al. (2013). The chemical composition of the essential oils of plants such as *M. spicata* and *S. rosmarinus* can vary considerably due to factors inherent to the type of soil, climatic conditions, development stage and genotype of plants and oil extraction methods (Aprotosoaie et al. 2017).

Of the total essential oils evaluated in this work, *M. spicata*, *S. rosmarinus, L. nobilis* and *P. glauca* essential oils were those that presented the highest fumigant activity against *E. atomaria*, similar toxicity than that of the synthetic insecticide chlorpyrifos. Pulegone, the major compound identified in *M. spicata* essential oil, could cause the high toxicity observed against *E. atomaria*. Indeed, *Mentha pulegium* essential oil (55.58% pulegone) showed a strong fumigant toxicity against *Lasioderma serricorne* (Fabricius) and *Tribolium castaneum* (Herbst) (LC_{50} = 8.5 and 11.6 µL/L air; 24 h exposure, respectively) (Salem et al. 2017), while the pulegone pure compound, at a concentration of 50 mg/L air, caused 100% mortality in insects such as *Sitophilus oryzae* (L.), *T. castaneum*, *Oryzaephilus surinamensis* (L.), *Musca domestica* (L.) and *Blattella germanica* (L.), in fumigant activity tests during 14 h of exposure (Lee et al. 2003). By contrast, another study showed that *M. pulegium* essential oil (70.4% pulegone) had low fumigant toxicity against *Rhyzopertha dominica* (Fabricius) (38.2% mortality; 96 h exposure) at a high concentration (2000 µL/L air) (Brahmi et al. 2016). Other essential oils extracted from *M. spicata* with a molecular composition different from that found in this work demonstrate high fumigant toxicity against the insect *Callosobruchus chinensis* (L.) (mortality = 72.67%; concentration: 100 µL/L air; 6 h exposure) (Kedia et al. 2014) and against the phytophagous mite *Tetranychus urticae* (C.L.Koch) (LC_{50} = 1.3 µL/L air; 24 h exposure) (Pavela et al. 2016). Similarly, the strong fumigant activity

shown by *S. rosmarinus* essential oil against *E. atomaria* could be attributed to its major components. Indeed, *S. rosmarinus* essential oils having a similar composition showed strong fumigant toxicity against insects of stored grains in general, such as *T. confusum* (mortality = 100%; concentration: 320 µL/L air; 72 h exposure; essential oil composition: 21.45% 1,8-cineole, 19.70% camphor); *Callosobruchus maculatus* (Fabricius) (LC_{50} = 15.7 µL/L air; 24 h exposure; essential oil composition: 22.64% α-pinene, 21.84% camphor, 21.53% 1,8-cineole); and *S. zeamais* (LC_{50} = 121.8 mg/L air; 24 h exposure; essential oil composition: not determined) (Sener et al. 2009; Krzyżowski et al. 2020; Yang et al. 2020). A further study determined that the concentration of 0.20% (v/v) of an essential oil emulsion of *S. rosmarinus* (26.7% 1,8-cineole, 18.6% α-pinene, 17.5% camphor and 11.8% camphene) caused 100% mortality against the phytophagous mite *T. urticae* in slide-dip assays in only 4 h exposure (Laborda et al. 2013).

Similarly, the strong fumigant toxic activity demonstrated by *L. nobilis* and *P. glauca* essential oils against *E. atomaria* agrees with what was found in other studies. For example, essential oils extracted from *L. nobilis* have a powerful fumigant activity against other phytophagous insects such as the aphid *Aphis gossypii* (Glover) (LC_{50} = 15.7 ppm; 24 h exposure; essential oil composition: 25.50% 1,8-cineole, 13.95% α-terpinyl acetate) and the moth *Ephestia kuehniella* (Zeller) (LC_{50} = 20.8 µL/L air; 24 h exposure; essential oil composition: 34.62% 1,8-cineole, 12.57% linalool) (Ebrahimi et al. 2013; Jemâa et al. 2013). Additionally, *L. nobilis* essential oil also showed strong fumigant activity against stored grain insects such as *R. dominica* (LC_{50} = 67.9 µL/L air; 24 h exposure; essential oil composition: 38.86% 1,8-cineole, 10.47% isovaleraldehyde) and *Acanthoscelides obtectus* (Say) (LC_{50} = 10.0 (male insects) and 5.7 (female insects) µL/L air; 24 h exposure; essential oil composition: not determined) (Papachristos and Stamopoulos 2002; Jemâa et al. 2012). However, several studies revealed that the fumigant toxicity of *L. nobilis* essential oil decreases against *T. castaneum* (LC_{50} = 172.3 µL/L air; 24 h exposure; essential oil composition: 38.86% 1,8-cineole, 10.47% isovaleraldehyde; LC_{50} = 208.7 µL/L air; essential oil composition: 21.15% 1,8-cineole, 14.47% α-terpinenyl acetate, 12.27% linalool; LC_{50} = 243.78 µL/L air; 24 h exposure; essential oil composition: not determined) (Jemâa et al. 2012; Senfi et al. 2014; Haouel-Hamdi et al. 2020). On the other hand, the similar and high toxicity observed in the essential oils of *P. glauca* leaves and flowers could be attributed to a similar chemical composition of both oils. Unfortunately, in this work, the chemical composition of these essential oils was not determined, it is possible that both oils are rich in limonene, sabinene and α-pinene, major compounds found in the essential oil extracted from the *P. glauca* aerial parts in the flowering–fruiting stage (Bailac et al. 2005). To date, the insecticidal activity of *P. glauca* essential oil (38.0% limonene; 23.4% β-pinene; 23.2% α-pinene) has only been evaluated against the honey bee *Apis mellifera* (L.) (LC_{50} = 12.0 µL/Petri dish). Yet, due to the type of test carried out, the authors explain that the high toxicity observed may be attributed to the combination of fumigant, contact and ingestion effects (Ruffinengo et al. 2005). In addition, the same authors also observed a high fumigant toxicity against mite *Varroa destructor* (Anderson and Trueman) (LC_{50} = 3.5 µL/Petri dish).

With respect to the essential oils extracted from *X. strumarium* and *G. megapotamica,* both had an acceptable toxic fumigant effect against *E. atomaria*. Surprisingly, the toxic effect of these essential oils has not been reported against any insect. Diaz Napal et al. (2015), however, demonstrated that *G. megapotamica* ethanolic extracts have strong antiforaging activity against the leaf-cutting ant *Acromyrmex lundii* (Guérin-Méneville) (inhibitory concentration 50 (IC_{50}) = 61.96 µg/cm^2 rose leaf), while Gökçe et al. (2011) reported that ethanol extracts of *X. strumarium* have strong insecticidal activity by ingestion against grape berry moth *Paralobesia viteana* (Clemens) larvae (mortality > 90%, concentration: 10% w/w (extract/diet)). It is known that the essential oils of *G. megapotamica* aerial parts are rich in α-pinene (7.7–13.5%), β-pinene (7.9–24.2%), limonene (7.5–16.7%), 1,8-cineole (12.2–12.5) and β-caryophyllene (6.5–11.7); while the essential oils from *X. strumarium* leaves are rich in *cis*-β-guaiene (34.2–79.6%), limonene (20.3–24.7%) and borneol (10.6–11.6%) (Duschatzky et al. 2003; Esmaeili et al. 2006; Adams et al. 2008; Scherer et al. 2010; Sharifi-Rad et al. 2015) compounds that could be responsible for the toxicity caused against *E. atomaria*. Finally, *S. chilensis*, *B. salicifolia* and *P.* × *citrosum* essential oils did not show relevant toxicity against *E. atomaria.*

In this work, the mode of action of essential oils with strong toxicity has not been evaluated; however, it is known that terpenoid compounds such as pulegone, camphor and 1,8-cineole affect the nervous system of insects by inhibiting the activity of the enzyme acetylcholinesterase (AChE), causing paralysis and death (Abdelgaleil et al. 2009; López and Pascual-Villalobos 2010; Rizvi et al. 2018; Shahriari et al. 2018).

In conclusion, the protection of agricultural crops against different pest insects through the application of formulations containing essential oils extracted from plants, instead of synthetic pesticides, is one of the most promising areas in integrated pest management (IPM) programmes. Our results demonstrated that essential oils extracted from *M. spicata, S. rosmarinus, L. nobilis* and *P. glauca* have great potential as future components in bioinsecticide formulations due to the high fumigant toxicity presented against blister beetles *E. atomaria*. However, additional studies are

needed to determine potential costs, field applicability and human biosecurity.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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