Crop Protection 148 (2021) 105737

Contents lists available at ScienceDirect

Crop Protection

journal homepage: www.elsevier.com/locate/cropro

Bioactivity of the essential oil from sweet orange leaves against the coconut mite *Aceria guerreronis* (Acari: Eriophyidae) and selectivity to a generalist predator

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ARTICLE INFO

Keywords:
Essential oil
Sustainable pest management
Biopesticides
Biological control

ABSTRACT

Several species of predatory mites, naturally occurring in coconut plantations, play an important role in regulating populations of phytophagous mites that are key pests of this crop. Plant essential oils (EOs) are bioactive mixtures of compounds that hold potential for controlling phytophagous arthropods with minimal impacts on non-target organisms and the environment. This study aimed to characterize the chemical composition and assess the bioactivity of the EO extracted from the leaves of the 'Pera' sweet orange grafted on 'Rangpur lime' to a key pest of coconut plantations worldwide, namely the coconut mite *Aceria guerreronis* (Acari: Eriophyidae), and its compatibility with the naturally-occurring generalist predatory mite *Typhlodromus ornatus* (Acari: Phytoseiidae). Briefly, sabinene was the major component, followed by δ-3-carene and (*E*)-β-ocimene. The EO was toxic to *A. guerreronis* (LC_{50} = 4.28 mg/mL; LC_{80} = 10.39 mg/mL) but not to *T. ornatus*. The LC_{80} of the EO did not repel *A. guerreronis* and was toxic to the pest mite only in the first hours (<9 h). Moreover, this concentration did not affect the growth rate of the predator, which was positive over the 10 days of exposure to the oil. Therefore, the EO of 'Pera' sweet orange holds potential for the management of coconut mite and is compatible with the generalist predatory mite *T. ornatus*.

1. Introduction

Essential oils (EOs) are lipophilic chemical compounds synthesized as a result of plant secondary metabolism. Research over the last decades have demonstrated that EOs display bioactivity against macro- and microorganisms. Depending on their composition and dose, they can cause mortality, or have sublethal effects such as repellence, reduced fertility and fecundity, or affect the behaviour of phytophagous arthropods (Plata-Rueda et al., 2018). EOs are promising for managing agricultural pests, as they have low toxicity, selectivity to non-target organisms and low persistence in the environment (Chae et al., 2014). Although dosages of EOs that are lethal to phytophagous pests are not generally toxic to predatory mites, sublethal effect on development, longevity and fecundity on beneficial arthropods may occur (Amer and Momen, 2002; Tsolakis and Ragusa, 2008), and should be considered when assessing compounds with pesticide potential.

Among EOs, those produced by citrus species hold potential for applications in pest management programs (Campolo et al., 2017). In Brazil, sweet orange [*Citrus sinensis* L. (Osbeck)] cv. 'Pera' is one of the most grown cultivars, particularly in the northeastern region of the country, and plants are usually grafted on 'Rangpur' lime (*Citrus limonia* Osbeck) (Carvalho et al., 2020).

The coconut mite, *Aceria guerreronis* Keifer (Acari: Eriophyidae), is a key pest of coconut plantations in the Americas, Africa and Asia (Navia et al., 2013). This mite builds up on the fruit perianth under the bracts. Mite feeding results in the development of white lesions that turn necrotic as injury progresses, leading to fruit fall, reduced fruit size and water content, and yield losses as high as 60%. Moreover, fruits intended for the fresh market have their value reduced due to visual necrosis (Navia et al., 2013). The coconut mite is mainly managed by preventive applications of chemical pesticides (Lima et al., 2015) which can lead to an array of well-known negative effects such as increased pest

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https://doi.org/10.1016/j.cropro.2021.105737
Received 1 February 2021; Received in revised form 7 June 2021; Accepted 10 June 2021
Available online 14 June 2021

resistance, ecosystem damage and decreased natural biological control (Geiger et al., 2011; Cordeiro et al., 2013).

The generalist predatory mite *Typhlodromus ornatus* Denmark & Muma (Acari: Phytoseiidae) commonly occurs in coconut plantations of northeastern Brazil (Navia et al., 2005; Reis et al., 2008) and feeds on *A. guerreronis* (Freitas et al., 2018). Here, we aimed to characterize the chemical composition of the EO extracted from leaves of sweet orange cv. 'Pera' grafted on 'Rangpur' lime and to further assess its bioactivity against *A. guerreronis* and compatibility with the non-target generalist predatory mite *T. ornatus*.

2. Material and methods

2.1. Living material

Leaves from sweet orange cv 'Pera' trees grafted on 'Rangpur' lime were collected in 2019 in a commercial plantation located in the municipality of Rio Real, Bahia state, Brazil (11°29′ S; 37°56′4″ W) and kept frozen until oil extractions.

Unsexed adults of the coconut mite used in the bioassays were collected from young fruits of green dwarf coconut palms in the city of Aracaju, Sergipe state, Brazil (10°56′46″ S; 37°03′12″ W). Although the exact ages of *A. guerreronis* could not be determined, we collected mites from colonies in early stage of oviposition (Oliveira et al., 2017). A colony of *T. ornatus* was started with individuals from a stock colony maintained at the laboratory. The predatory mites were reared on a 9-cm-diameter PVC disc placed on a water-soaked foam in a 15-cm-diameter plastic pot, hereafter referred to as arena. Cottonwool was placed around the disc and kept moistened to prevent mites from escaping. Two four-cm^2-plastic pieces were placed on the arena as oviposition sites. Mites were provided daily with castor bean (*Ricinus communis* L.) pollen as food, and kept under controlled conditions (27.0 ± 3.0 °C, RH 70 ± 10%, in the dark).

2.2. Extraction and analysis of the essential oil

Ca. 725 g of leaves were taken from three plants and further pooled before the EO was hydrodistilled using a modified Clevenger apparatus for 2 h. The sample was analysed by gas-chromatography/mass spectrometry (GC-MS/FID) (QP2010 Ultra, Shimadzu Corporation, Kyoto, Japan) under standard chromatographic conditions as described in Farias et al. (2020). Quantification of individual compounds was achieved by estimating the normalized peak area (%) and identification of individual compounds by injecting a mixture of hydrocarbons (C_7–C_{30}), calculation of the retention times according to van den Dool and Kratz (1963) and comparison of the acquired mass spectra with reference libraries (NIST107, NIST21, WILEY8) and the literature (Adams, 2007).

2.3. Contact toxicity to Aceria guerreronis

A preliminary test was conducted to establish the highest concentration that did not cause mortality and the lowest concentration that caused 100% mortality. Acetone (Sigma-Aldrich) was used as solvent and as control. The arenas (experimental units) were prepared on a 9-cm-Petri dish and consisted of 1.3-cm diameter discs of the perianth of young fruits, immersed in potato dextrose agar (PDA) medium. The fruit tissue was exposed by the aid of a mold (Teodoro et al., 2017). Aliquots of the oil (9.3 mL) were sprayed on the arenas in a Potter Tower (Burkard, United Kingdom) at a pressure of 5 psi. Based on the preliminary test, the following concentrations were selected: 1, 2, 5, 8 and 12 mg/mL. After spraying, the arenas were covered with a black cloth to simulate dark conditions under the bracts and kept under controlled conditions (27 ± 3 °C; 70 ± 10% RH). Sprayed discs were air-dried for 30 min before ten adults of *A. guerreronis* were transferred to each disc. The experiment followed a completely randomized design with 10 replicates (discs) per concentration, totalizing 100 mites per concentration. Mortality was assessed 24 h after spraying by gently touching mites with a fine paintbrush (Oliveira et al., 2017).

2.4. Repellence to Aceria guerreronis

Preparation of the arenas and spraying of the EO or acetone (as control) followed the same procedures described for the toxicity bioassay for *A. guerreronis*. However, only half of the arena was sprayed with the EO or acetone. For this, half of the arena was covered with a double layer of tape placed on a paper sheet before spraying (Teodoro et al., 2017). After spraying the LC_{80} of the EO, a small drop of non-toxic glue (Art-Maxi, São Paulo, Brazil) of about 0.2 cm in diameter was applied on the middle of the arena. Adult mites were individually placed on the dried glue and allowed to move freely on the arena for 24 h. The position (i.e., on untreated or on treated half of the arena) of each individual was recorded 1, 14 and 24 h afterwards. A total of 60 replicates (mites and arenas) for either the EO or control were observed.

2.5. Residual toxicity

The experiment was conducted following the same methodology as described for the toxicity bioassay. Arenas were sprayed with either acetone (control) or the LC_{80} of the oil to the coconut mite. Ten mites were transferred to each arena 0.5, 3, 6 and 9 h after spraying, the mortality was recorded 24 h after introducing the mites. The experiment followed a completely randomized design with 13 replicates, totalling 130 mites per evaluated time.

2.6. Mortality of Typhlodromus ornatus

Aliquots (9.3 mL) of the LC_{50} and LC_{80} of the EO estimated for *A. guerreronis* and acetone (control) were sprayed on the arenas. Each arena consisted of a 2.5-cm-diameter PVC disc sitting on a polyurethane foam and surrounded by filter paper placed on a 9-cm Petri dish. The foam and the filter paper were kept moistened to prevent mites from escaping. After spraying, ten 7-to-10-day-old *T. ornatus* adult females were transferred to the arena. Castor bean pollen placed on a 0.36 cm^2 transparent plastic was provided as the food source. The experiment followed a completely randomized design with 15 replicates (150 mites). The arenas were kept under controlled conditions as described for the toxicity bioassay. The number of dead mites in the treated and control arenas was recorded after 24 h.

2.7. Population growth of Typhlodromus ornatus

The LC_{80} of the EO, as estimated for *A. guerreronis,* was used to assess the instantaneous growth rate (r_i) of the predatory mite *T. ornatus*. The experiment followed the same methodology described for the mortality of *T. ornatus*. Additionally, a 0.35 cm^2 plastic piece was placed in the arena as an oviposition site. After spraying, four 7-to-10-day-old *T. ornatus* adult females and one male were transferred to each arena. The experiment followed a completely randomized design with 9 replicates per treatment. The total numbers of mites (eggs, immatures, and adults) in each arena were counted 10 days after the establishment of the experiment.

2.8. Statistical analyses

All statistical analyses were performed using SAS statistical package version 9.1 (SAS Institute, 2008). Lethal concentrations of the EO to *A. guerreronis* were estimated with Probit analysis using the SAS procedure PROC PROBIT. Repellence of the oil to *A. guerreronis* was assessed by a Chi-square frequency test using the SAS procedure PROCFREQ. The susceptibility of *T. ornatus* to the LC_{50} and LC_{80} estimated with *A. guerreronis* and the residual toxicity to *A. guerreronis* were submitted to regression analysis. Parsimonious regression models were

selected based on standard errors and R^2 values. The instantaneous growth rate (r_i) was calculated by the following equation: [ln (Nf/Ni)]/T, where Nf is the final number of living mites including eggs and immatures, Ni is the initial number of mites and T is the period of the bioassay (10 days) (Walthall and Stark, 1997). The r_i of the predatory mites exposed to the oil and control were compared by a t-Student test.

3. Results

Thirty-three compounds, mainly monoterpenoids, were identified in the EO of 'Pera' sweet orange (Supplementary table). The major compound was sabinene (33.17%), followed by δ-3-carene (11.24%), (*E*)-β-ocimene (9.44%), linalool (7.96%) and limonene (5.11%). The EO LC_{25}, LC_{50} and LC_{80} of the EO to *A. guerreronis* were estimated to be 2.10 mg/mL, 4.28 mg/mL and 10.39 mg/mL, respectively (n = 500, slope = 2.18, $\chi^2 = 1.643$, $P = 0.122$). These LC did not kill the predatory mite *T. ornatus* (Fig. 1). The LC_{80} of the oil did not repel *A. guerreronis* 1 h, 14 h and 24 h after spraying, as mites did not show preference towards sprayed or unsprayed disc halves (Fig. 2). The mortality of *A. guerreronis* exposed to the LC_{80} of the oil decreased over time ($F_3 = 201.14$, $P = 0.0001$) (Fig. 3). The instantaneous growth rate (r_i) of the predatory mite was not affected by the LC_{80} of the oil, as estimated for *A. guerreronis* (1.13 ± 0.10 with the LC_{80}; 1.12 ± 0.14 with the control (means ± SD); t = 0.18, df = 8, $P = 0.856$).

4. Discussion

The EO extracted from leaves of the sweet orange cv. 'Pera' grafted on 'Rangpur' lime presented the monoterpene sabinene (33.17%) as the major compound in contrast with the EO of fruit peel that contain limonene as main component (Farias et al., 2020). We also have shown that the EO extracted from leaves of 'Pera' displays bioactivity against a major coconut pest, *A. guerreronis*. This EO has previously been shown to present activity against the cassava green mite *Mononychellus tanajoa* Bondar (Acari: Tetranychidae) (Farias et al., 2020). Although the mechanisms have not been elucidated, the toxic effects reported here occur possibly due to either fumigant or contact acute toxicity of sabinene, alone or in combination with other compounds (Isman, 2000). Little is known about the lethal and sub-lethal effects of this major compound on arthropods (Wang et al., 2015). Other compounds present in this EO are known to exhibit bioactivity against arthropod pests. For example, terpinen-4-ol is toxic against *Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae) and *Lasioderma serricorne* Fabricius

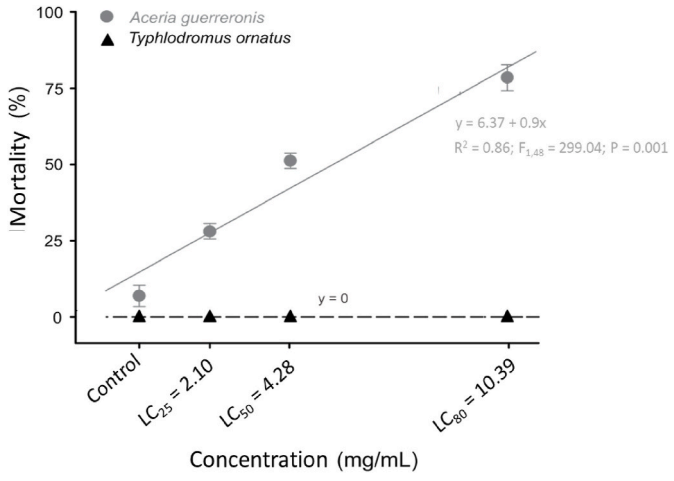

Fig. 1. Mortality of *A. guerreronis* and the predatory mite *T. ornatus* exposed to LC_{25} (2.10 mg/mL), LC_{50} (4.28 mg/mL) and LC_{80} (10.39 mg/mL) of the essential from leaves of 'Pera' sweet orange, as estimated for *A. guerreronis*.

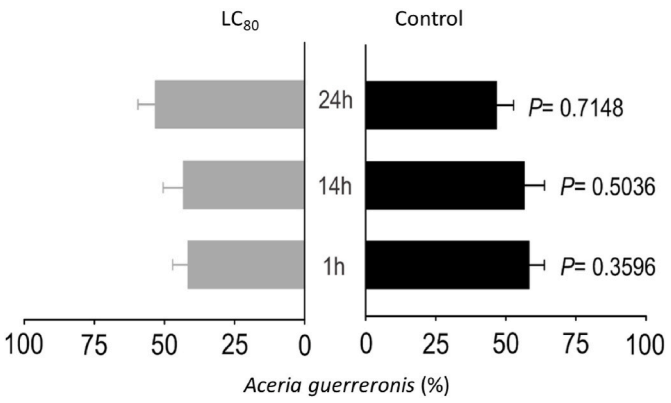

Fig. 2. Percentage of *A. guerreronis* choosing between disc halves that were unsprayed (black bars) or sprayed (grey bars) with the LC_{80} (10.39 mg/mL) of the essential from leaves of 'Pera' sweet orange, as estimated for the pest.

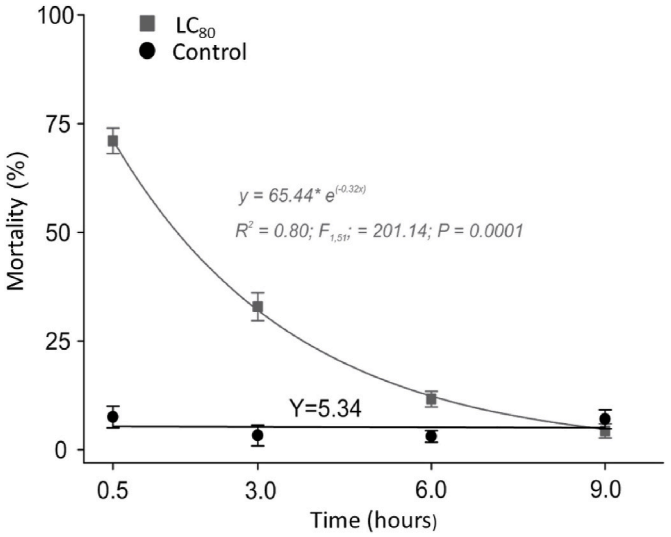

Fig. 3. Residual toxicity of LC_{80} (10.39 mg/mL) of the essential oil from leaves of 'Pera' sweet orange to *A. guerreronis*, as estimated for the pest.

(Coleoptera: Anobiidae) (Wang et al., 2015). Also, limonene displays toxic and repellent activity against an array of arthropods (Ibrahim et al., 2001; Ribeiro et al., 2019).

The LC_{50} and LC_{80} of the oil, estimated for *A. guerreronis,* did not affect the generalist predatory mite *T. ornatus*. Moreover, the LC_{80} of the EO did not affect population growth of the predator either. Plant-derived oils have proven to be highly selective to *T. ornatus* (Oliveira et al., 2017; Freitas et al., 2018). Selectivity may occur due to anatomic and physiological differences between species including the size of the predator in relation to the prey; cuticular differences such as the tegument that covers the mite body may differently prevent the penetration of chemical compounds (Tsolakis and Ragusa, 2008; Lima et al., 2013); or the presence of detoxifying enzymes (Sato et al., 2006). This ability of this predatory mite to withstand concentrations that are lethal to the prey suggest that the mite could be exposed directly or indirectly to the oil in field conditions without significant negative effects.

Mono- and sesquiterpenoids are compounds with low vapour pressure and high volatility, and consequently EOs are low persistence. In our study, the EO remained bioactive for the first few hours after spraying, virtually disappearing 9 h thereafter. Although low persistence is an appreciated characteristic in chemical pest management, fast environmental degradation and volatility of EOs may constrain the control of pests such as *A. guerreronis*, and therefore, improved

formulations such as microemulsion systems that extend the lifetime of volatile compounds and boost the bioactivity of the oil should be developed. Further studies are also needed to assess the role of individual main components of the oil such as sabinene on this pest.

CRediT authorship contribution statement

Dalton R.B. Brito: Investigation, Writing – original draft, preparation. **Delia M. Pinto-Zevallos:** Conceptualization, Investigation, Writing – review & editing. **José G. de Sena Filho:** Investigation, Writing – review & editing. **Caroline R. Coelho:** Formal analysis. **Paulo C.L. Nogueira:** Methodology. **Helio W.L. de Carvalho:** Funding acquisition. **Adenir V. Teodoro:** Conceptualization, Investigation, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors would like to thank the National Council of Technological and Scientific Development (CNPq) process (113817/2018-5) for financial support.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.cropro.2021.105737.

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