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Citrus essential oils control the cassava green mite, *Mononychellus tanajoa*, and induce higher predatory responses by the lacewing *Ceraeochrysa caligata*

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ABSTRACT

Plant essential oils and their active compounds are recognized as sustainable tools for the management of arthropod pests. Here, the chemical composition of essential oils obtained from different *Citrus* scions was characterized (i.e., sweet oranges 'Kona' and 'Pera CNPMF-D6'; acid lime 'Persian lime 58' and mandarin 'Piemonte'), and it was evaluated whether these oils could be used to control *Mononychellus tanajoa* (Acari: Tetranychidae), a key pest of cassava crop. In addition, it was assessed whether exposure to acid lime oil alters the predatory ability of the generalist predator *Ceraeochrysa caligata* (Neuroptera: Chrysopidae). Predatory bioassays were conducted at two prey densities (i.e., 25 and 50 mites in an arena of 7.1 cm^2) immediately after exposure to essential oil and over three consecutive days. Sabinene and linalool were main components in oranges and mandarin oils, while limonene made up more than half of acid lime oil. Based on their LC_{50} values, all the essential oils and the limonene isomers equally controlled *M. tanajoa*. However, mites exposed to the LC_{25} or LC_{50} of acid lime essential oil exhibited reduced survival rates compared to control. In comparison, exposure to the LC_{80} of acid lime essential oil caused low mortality (i.e., 20.4 ± 5%) of *C. caligata* larvae. Moreover, *C. caligata* larvae that survived exposure to LC_{50} and faced prey scarcity exhibited higher predatory ability immediately after exposure. Overall, this study demonstrates that the essential oils of these *Citrus* scions could be successfully integrated into management programs for *M. tanajoa*.

1. Introduction

Plant essential oils are complex mixtures of small, volatile, and lipophilic chain compounds (Benelli et al., 2018). Essential oils are bioactive to a myriad of arthropod pests, with their performance being contingent on the susceptibility of target species (Khani and Asghari, 2012). In addition to causing mortality, these essential oils may affect the life span, fertility, fecundity, and behavior of pests (Ribeiro et al., 2015; Ferreira et al., 2017; Jesser et al., 2017; Benelli et al., 2018; Lourenço et al., 2018; Plata-Rueda et al., 2018). Furthermore, such oils are generally nontoxic to predators; however, sublethal concentrations/doses sometimes have adverse effects (Castilhos et al., 2018; Toledo et al., 2019).

Citrus essential oils exhibit insecticidal and acaricidal activities (Camara et al., 2015; El-Akhal et al., 2015; Zarrad et al., 2015; Dutra et al., 2016; Campolo et al., 2017; Fouad and Camara, 2017; Papanastasiou et al., 2017). Although several *Citrus* scion cultivars (e.g., 'Kona' sweet orange, 'Piemonte' mandarin, and 'Persian lime 58' acid lime) have been introduced for diversification purposes (Carvalho et al., 2016; Martins et al., 2016), the sweet orange 'Pera' [*Citrus sinensis* (L.) Osbeck] is the main scion cultivar in orchards across Brazil (Passos et al., 2013; Carvalho et al., 2018). The essential oils of these *Citrus* scion cultivars could function as source of terpenes for use in integrated pest management, especially in Neotropical agricultural systems.

Among the arthropod specialists that have co-evolved with their crop, the cassava green mite *Mononychellus tanajoa* (B.) (Acari: Tetranychidae) is a key pest that causes large losses in cassava root production (Pinto-Zevallos et al., 2016). Probably native to Northeast

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Brazil (Bellotti et al., 1999), this mite is now globally distributed, causing yield losses as high as 80% in Africa (Yaninek and Herren, 1988) and 60% in China (Chen et al., 2010).

The main strategies used to control *M. tanajoa* include plant resistance and biological control, using natural enemies and entomopathogenic fungi (Zannou et al., 2007; Agboton et al., 2013; Onzo et al., 2013; Parsa et al., 2015). However, few cultivars combine resistance to pests with desired agronomical characteristics (Mahungu et al., 1994; Dixon et al., 2002; Mutisya et al., 2013). In particular, the use of entomopathogens is restricted to conditions with high humidity (Delalibera et al., 2006). Predatory mites (mainly those belonging to the family Phytoseiidae) and generalist predatory insects (e.g., lacewings, ladybirds) are important natural enemies of this pest (Zannou et al., 2007; Rêgo et al., 2013; Sattayawong et al., 2016; Boopathi et al., 2017). Lacewings (*Ceraeochrysa* spp.) (Neuroptera: Chrysopidae) are native to the Americas, with most species occurring in the forests and agricultural areas of Neotropical region, increasing their potential to be used for controlling a wide array of agricultural pests (Tauber et al., 2000; Albuquerque et al., 2001; Farias et al., 2018; Viteri Jumbo et al., 2019). However, along with other reasons, the high reproductive capacity of *M. tanajoa* during the dry season limits the actions of natural enemies, including the *Ceraeochrysa* lacewing (Onzo et al., 2003, 2013; Rêgo et al., 2013). Thus, this study aims at evaluating the chemical composition and toxicity of essential oils extracted from four *Citrus* scion cultivars and their potential impacts on the predatory ability of the generalist predator *Ceraeochrysa caligata* B. (Neuroptera: Chrysopidae) against the cassava green mite.

2. Materials and methods

2.1. Sources and rearing M. tanajoa and C. caligata

Stock colonies of *M. tanajoa* were stablished with individuals collected from a cassava plantation in Boquim (11°08′38″ S, 37°38′37″ W), located in the northeastern state of Sergipe, Brazil. The mites were reared on potted cassava plants (30 days-old), of 'Caravela' cultivar, maintained under controlled conditions (temperature 28 ± 2 °C, relative humidity 60 ± 10%, and 12 h photoperiod) in Aracaju (10°57′04″ S, 37°02′58″ W), Sergipe state, Brazil. The substrate used was sand, black soil, and coconut powder (3:1:1). After plant depletion, mite colonies were transferred to new 30 days-old cassava plants.

The stock culture of *C. caligata* was started with individuals collected from Itaporanga D'Ajuda (11°06′40″S, 37°11′15″W) Sergipe State, Brazil. Eggs of *C. caligata* were kept in separate Petri dishes of 5 cm diameter under controlled conditions (temperature 27 ± 2 °C, relative humidity 70 ± 10%, and 12 h photoperiod) until the larvae emerged. Pieces of water-soaked cotton wool were distributed in the Petri dishes to prevent the larvae dehydrating. Larvae were starved for 12 h before being used in the bioassays.

2.2. Essential oils

2.2.1. Extraction

We used *Citrus* scions located at the experimental orchard of Embrapa (11° 22′ 37″ S, 37° 40′ 26″ W, Sergipe State, Brazil) to collect the leaves used to extract the essential oils. The leaves were collected in September 2016 and were obtained from the following *Citrus* scion cultivars: *1)* mandarin 'Piemonte' ['Clementina' mandarin (*C. clementina* hort. ex Tanaka) x 'Murcott' (hybrid of unknown origin, possibly resulting from the crossing between mandarin and sweet orange, according to Hodgson, 1967)]; *2)* clone of Tahiti acid lime 'Persian lime 58'; *3)* sweet orange 'Kona' (*C. sinensis*); *4)* sweet orange 'Pera CNPMF-D6' (*C. sinensis*). All scion cultivars (9 years old) were grafted on 'Rangpur' lime (*C. limonia* O.). Essential oils were extracted from 600 g of fresh leaves by hydrodistillation using a modified Clevenger apparatus for 3 h and 2 L of water volume (Andrade et al., 2016; Sena Filho et al., 2017).

2.2.2. Chemical composition

The chemical constituents of the essential oils were analyzed in GC–MS/FID (Gas chromatography coupled to mass spectrometry/flame ionization detector) (Shimadzu model QP2010 ultra), equipped with an autosampler (AOC-20i, Shimadzu) used to separate compounds with an RtxR®-5MS Restek fused silica capillary column (5%-diphenyl–95%-dimethyl polysiloxane) of 30 m x0.25 mm i.d., 0.25 µm film thickness, at a constant helium (99.999% purity) flow rate of 1.2 mL/min. The temperature was programmed to maintain the oven at 60 °C for 4 min, followed by increase of 3 °C/min until 220 °C. The injection volume was 1.5 µL of the sample solubilized in dichloromethane (CH_2Cl_2), with a split ratio of 1:10. The injector and detector temperatures were 250 °C and 280 °C, respectively. The MS were obtained in the electron ionization mode at 70 eV with a range of 0.5 s and fragments of 40–550 Da.

Estimates of each were estimated by FID peak-area normalization (%). Compound concentrations were calculated from the GC peak areas relative to the total peak area of all peaks, and were arranged in order of GC boiling. The retention index was obtained by co-injecting the oil sample with a C9–C30 linear hydrocarbon mixture. Compounds were identified based on the retention indices, and by comparing the mass spectra stored in the mass spectra database (NIST107 and NIST21; WILEY), together with the mass spectra in the literature (Adams, 2007).

2.3. Toxicity of essential oils and enantiomers of limonene to M. tanajoa

Concentrations of the four citrus essential oils were sprayed onto leaf arenas (2 cm diameter) containing 20 adult females of *M. tanajoa* at the onset of their reproductive period (8 days old). The arenas formed the experimental unit, and contained cassava leaves placed in Petri dishes (10 cm diameter, 2 cm deep), with a mixture of 5% pure bacteriological agar, 0.3% of fungicide methylparaben (Nipagim™), and distilled water (Teodoro et al., 2017). Oil solutions and the acetone control were sprayed through a Potter tower (Burkard, Rickmansworth, UK), with a volume of 9.3 mL, pressure of 0.34 bar (34 Kpa), and deposition of 1.7 mg/cm^{-2}, in accordance with the International Organization for Biological Control (Hassan et al., 1994). The concentrations were selected after pilot bioassays, ranging from the highest nonlethal concentration (lower limit) to the lowest concentration, causing 100% mortality (upper limit), using acetone as solvent and control. Five concentrations were used for the oils from 'Pera CNPMF-D6′ (1.7, 6.1, 10.4, 14.8, 17.4 mg/mL) and 'Kona' (1.7, 6.0, 10.3, 14.6, 17.2 mg/mL) sweet oranges, and 'Piemonte' mandarin (1.6, 5.7, 9.7, 13.8, 16.2 mg/mL). Six concentrations were used for the 'Persian lime 58' (1.7, 4.2, 5.8, 8.3, 10.0, 14.2 mg/mL) in a completely randomized design, with six replicates (arenas) for each concentration, totaling 120 females of *M. tanajoa* per concentration. After spraying, the arenas were surrounded with distilled water-soaked cotton wool, covered by plastic film and were kept in BOD (temperature 27 ± 2 °C, relative humidity of 70 ± 10%, and 12 h photoperiod). Mortality was assessed after 24 h exposure, and the mites were considered dead when they did not move when touched with a fine brush (Stark et al., 1997).

Based on the toxicity of the oils, lethal concentrations of the *(S)*-limonene and *(R)*-limonene enantiomers (which are major components of 'Persian lime 58') were estimated following the same methodology. Five concentrations of (*R*)-limonene (1.7, 6.1, 8.7, 13.1, 17.5 mg/mL) and six concentrations of *(S)*-limonene (1.7, 4.2, 5.8, 8.4, 12.5, 16.7 mg/mL) were used in the bioassays. Both enantiomers were acquired from Sigma-Aldrich (99% purity).

2.4. Sublethal effects of 'Persian lime 58' oil on survival of M. tanajoa

The survival of *M. tanajoa* was evaluated using females treated with the LC_{25} and LC_{50} of 'Persian lime 58' essential oil. Control arenas were sprayed with acetone. The experiment consisted of a completely

Table 1
Chemical composition of the essential oils of *Citrus* scion cultivars.

Peak	RT[a] (min)	Compounds	RI[b] exp	RI[c] lit	Peak area (FID)/Cultivar (%)			
					Kona	Pera	Persian	Piemonte
1	5.650	butyl acetate	840	807	0.12	0.12	0.22	0.16
2	7.425	3-methyl-1-butanol acetate	883	869	0.12	0.10	0.21	0.16
3	9.295	α-Thujene	929	924	0.35	0.41	–	0.27
4	9.600	α-Pinene	936	932	1.72	2.02	0.34	1.27
5	11.350	Sabinene	979	969	39.49	44.72	0.98	30.76
6	11.465	β-Pinene	981	974	2.24	2.66	tr	2.24
7	11.785	6-Methyl-5-hepten-2-one	990	981	0.61	0.13	0.90	–
8	11.990	Myrcene	994	988	4.23	4.31	0.80	2.76
9	12.620	α-Phellandrene	1008	1002	–	–	–	0.32
10	12.945	δ-3-Carene	1015	1008	10.73	10.78	–	4.28
11	13.180	α-Terpinene	1020	1014	–	–	–	0.60
12	13.430	p-Cymene	1025	1020	0.16	0.16	–	–
13	13.555	o-Cymene	1028	1022	2.19	2.49	0.18	0.23
14	13.775	Limonene	1032	1024	4.33	3.57	52.63	2.15
15	13.970	1,8-Cineole	1032	1026	–	–	3.38	–
16	14.155	*(Z)*-β-Ocimene	1040	1032	0.15	0.13	–	0.21
17	14.725	*(E)*-β-Ocimene	1052	1044	1.35	1.10	–	8.07
18	15.215	γ-Terpinene	1062	1054	–	–	–	1.06
19	15.620	*cis*-sabinene hydrate	1071	1065	0.79	0.57	–	0.60
20	16.685	Terpinolene	1093	1086	0.24	0.21	–	0.98
21	17.255	Linalool	1105	1095	12.59	10.78	1.33	38.14
22	18.325	*cis*-p-Menth-2-en-1-ol	1126	1118	0.18	0.19	–	0.14
23	18.907	*cis*-Limonene-oxide	1138	1132	–	–	2.22	–
24	19.120	*trans*-Limonene-oxide	1143	1137	Tr[e]	0.14	1.17	–
25	19.870	Citronellal	1157	1148	0.62	0.74	0.37	0.47
26	21.190	Terpinen-4-ol	1184	1174	2.65	3.10	–	2.27
27	21.545	p-Cymen-8-ol	1191	1179	0.30	0.38	–	–
28	21.820	α-Terpineol	1197	1186	1.42	0.93	0.82	0.85
29	23.585	Citronellol	1233	1223	1.26	0.91	3.07	–
30	24.240	Neral	1247	1235	3.11	1.80	7.48	–
31	24.890	Geraniol	1258	1249	0.92	0.51	2.60	–
32	25.695	Geranial	1277	1264	4.05	2.95	9.51	–
33	28.125	NI[d]	1328	–	–	–	0.20	–
34	28.285	NI	1332	–	–	–	0.37	–
35	28.485	Neric acid	1336	1330	–	–	0.49	–
36	28.790	NI	1343	–	–	–	0.51	–
37	28.905	NI	1345	–	–	–	0.57	–
38	29.010	Limonene-1,2-diol	1347	1343	–	–	0.41	–
39	30.005	Neryl acetate	1369	1359	0.20	–	1.96	–
40	30.325	Geranic acid	1376	1355	–	–	0.66	–
41	30.605	NI	1382	–	–	–	0.55	–
42	30.950	Geranyl acetate	1389	1379	0.20	0.14	1.23	–
43	31.445	β-Elemene	1400	1389	0.66	1.06	–	1.29
44	32.730	(*E*)-Caryophyllene	1429	1417	0.14	0.22	–	0.18
45	39.805	Caryophyllene oxide	1596	1582	–	–	0.47	–
46	44.205	β-Sinensal	1708	1699	0.77	0.47	–	–
Total					97.89	97.80	95.63	99.46

[a] retention time.
[b] retention indices on RTX-5MS column calculated according to Van Den Dool and Kratz (Van Den Doolan and Kratz, 1963).
[c] retention indices according to literature (Adams, 2007).
[d] non-identified.
[e] trace amounts of compound were detected.

randomized design with 10 replicates (arenas) and 20 females per arena, totaling 200 mites for each treatment. The arenas were replaced every three days, to maintain constant conditions and mitigate deterioration. Mortality of *M. tanajoa* was recorded every 3 h on the first day, at 6 h intervals on the second day, and at 12 h intervals afterwards until all mites died.

2.5. Selectivity towards C. caligata

2.5.1. Mortality

First instar larvae of *C. caligata* were sprayed with the LC_{50} and LC_{80} of 'Persian lime 58' oil estimated for *M. tanajoa*. The larvae (12 h old) were placed in separate plastic cylindrical arenas (2 cm in diameter, 1 cm high, 9.9 cm^3 in volume), glued to a Petri dish (15 cm diameter), and surrounded by a water barrier to prevent the insects from escaping. The cluster of 10 arenas was considered to represent one replicate, with three replicates being used for each treatment. The sprayed arenas were air-dried before *Anagastra kuehniella* Z. (Lepidoptera: Pyralidae) eggs were added as food source for the lacewing larvae. Mortality was evaluated after 24 h of *C. caligata* exposure to treatments.

2.5.2. Consumption of M. tanajoa

First instar larvae of *C. caligata* were treated with the LC_{50} of 'Persian lime 58' oil, as estimated for *M. tanajoa*. Control larvae were sprayed with acetone. Sprayed and unsprayed *C. caligata* larvae were transferred to arenas (as described in section 2.5.1, except for being 3 cm in diameter, and containing either 25 or 50 adults of *M. tanajoa*). The experiment consisted of a completely randomized design with 10 replicates (arenas) for each treatment. The consumption of mites by the larvae of *C. caligata* was evaluated at 2, 4, 6, 8, and 24 h intervals after

application for three days. The mites that were consumed were counted for each timeframe. Afterwards, the number of individuals was restored to the initial densities.

2.6. Data analyses

Mortality data were subjected to Probit analyses to obtain lethal concentrations (LC) using the PROC PROBIT procedure (SAS, 2013). The LC_{50} of oranges, mandarin oils, and enantiomeric compounds were divided by the LC_{50} 'Persian lime 58' oil to obtain the toxicity ratio. Acaricidal activity was compared using the toxicity ratio. The 95% of its confidence limits were considered significantly different ($P < 0.05$) if they did not include the value 1 (Robertson et al., 2007). The survival of *M. tanajoa* was estimated by the Kaplan-Meier model. The curves were compared by the Holm–Sidak's test in Sigma Plot v. 11.0 (Systat, 2008). Mortality rates of *C. caligata* exposed to sublethal concentrations of 'Persian lime 58' oil were subjected to analysis of variance using the PROC ANOVA (SAS, 2013). Consumption data of *M. tanajoa* by *C. caligata* were submitted to repeated measures ANOVA, using PROC ANOVA. The number of prey consumed by exposed and unexposed predators for each time (h) in each day were compared by t-Student tests using the PROC TTEST (SAS, 2013). Daily consumption by the predator as a function of time (h) for each density and oil was subjected to regression analysis by the PROC REG of the SAS program (SAS, 2013).

3. Results

3.1. Composition of essential oils

The chromatographic analysis detected 46 compounds in the essential oils of those four *Citrus* cultivars (Table 2). The main components of sweet oranges essential oils were the monoterpenes sabinene (39.49%), linalool (12.59%), and δ-3-carene (10.73%) for 'Kona'. The main components of 'Pera CNPMF-D6' were sabinene (44.72%), linalool (10.78%) and δ-3-carene (10.78%). The main components of 'Piemonte' mandarin essential oil were linalool (38.14%), sabinene (30.76%), and *(E)*-β-ocimene (8.07%). The main components of 'Persian lime 58' acid lime essential oil were limonene (52.63%), geranial (9.51%), and neral (7.48%). Minor quantities (< 5%) of some monoterpenes were also detected in the essential oils, including myrcene, 1,8-cineole, and terpinen-4-ol (Table 1).

3.2. Susceptibility of M. tanajoa to Citrus essential oils and limonene isomers

The essential oils of the four *Citrus* scions and the limonene isomers (i.e., *(S)*-limonene and *(R)*-limonene) were equally toxic to *M. tanajoa*, with LC_{50} ranging from 5.3 to 14.0 mg/mL (Table 2). Although essential oils exhibited no differences in their toxicity to *M. tanajoa*, we selected the 'Persian lime 58' for the subsequent toxicological studies (e.g., survival abilities, selectivity, and impact on the predatory responses of *C. caligata*), because it presented the lowest LC_{50} value (Table 2). The results also showed that the *M. tanajoa* that survived exposure to the LC_{25} (3.1 mg/mL) and LC_{50} (5.8 mg/mL) of 'Persian lime 58' essential oils exhibited significantly lower survival abilities (Holm–Sidak test: $\chi^2 = 173.68$, *d.f.* = 2, $P < 0.001$) than *M. tanajoa* that were not exposed (Fig. 1). While the non-exposed mites exhibited a LT_{50} of 9.6 (8.8–10.4) days, sublethally exposed *M. tanajoa* exhibited a maximum LT_{50} of 3.0 (for LC_{50} exposure) and 6.0 (for LC_{25} exposure) days (Fig. 1).

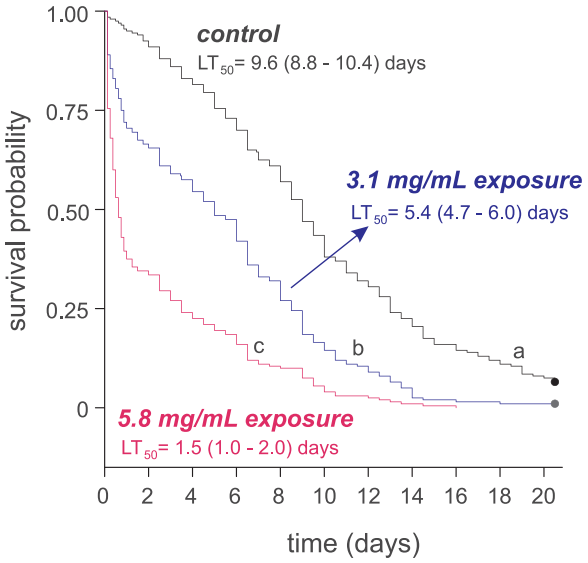

Fig. 1. Survival curves of *Mononychellus tanajoa* females exposed to LC_{25} (3.1 mg/mL) and LC_{50} (5.8 mg/mL) of 'Persian lime 58' essential oil. Survival curves grouped with the same letters were not significantly different using Holm–Sidak's test ($P > 0.05$).

3.3. Toxicity of 'Persian lime 58' essential oil to the predator C. caligata

Concentrations of 5.8 (LC_{50}) and 12.5 (LC_{80}) mg/mL of 'Persian lime 58' essential oil showed high selectivity levels to *C. caligata* larvae, as these exposures resulted, respectively, in mortality levels of just 10.0 (± 5.8%) and 20.4 (± 5.5%).

Table 2
Lethal concentrations (LC) (mg/mL) of the essential oils of four citrus scion cultivars and limonene enantiomers to *Mononychellus tanajoa*. χ^2: chi-square; *P*-value; n: number of mites; CI: confidence interval.

Essential oils	LC_{25} (95% CI)	LC_{50} (95% CI)	LC_{80} (95% CI)	χ^2	*P*	n	TR[a]
Persian lime 58	3.12 (2.65-3.55)	5.79 (5.26-6.35)	12.53 (11.06-14.68)	4.76	0.31	720	–
Piemonte	4.16 (3.47-4.79)	7.74 (6.93-9.66)	16.82 (14.71-19.93)	3.99	0.26	600	1.34 (1.18-1.52)
Kona	4.62 (3.87-5.30)	8.55 (7.67-9.48)	18.44 (16.09-21.92)	4.98	0.17	600	1.48 (1.30-1.68)
Pera CNPMF-D6	4.96 (4.12-5.72)	9.54 (8.54-10.65)	21.59 (18.47-26.52)	4.26	0.23	600	1.65 (1.43-1.89)
(S)-limoneno	4.55 (3.88-5.18)	9.71 (8.65-11.07)	24.97 (20.33-33.01)	7.56	0.11	720	1.68 (1.41-1.99)
(R)-limoneno	5.73 (4.79-6.58)	12.04 (10.63-13.95)	30.47 (24.30-42.07)	6.21	0.10	600	2.08 (1.72-2.51)

[a] toxicity ratio = higher LC50/lower LC50.

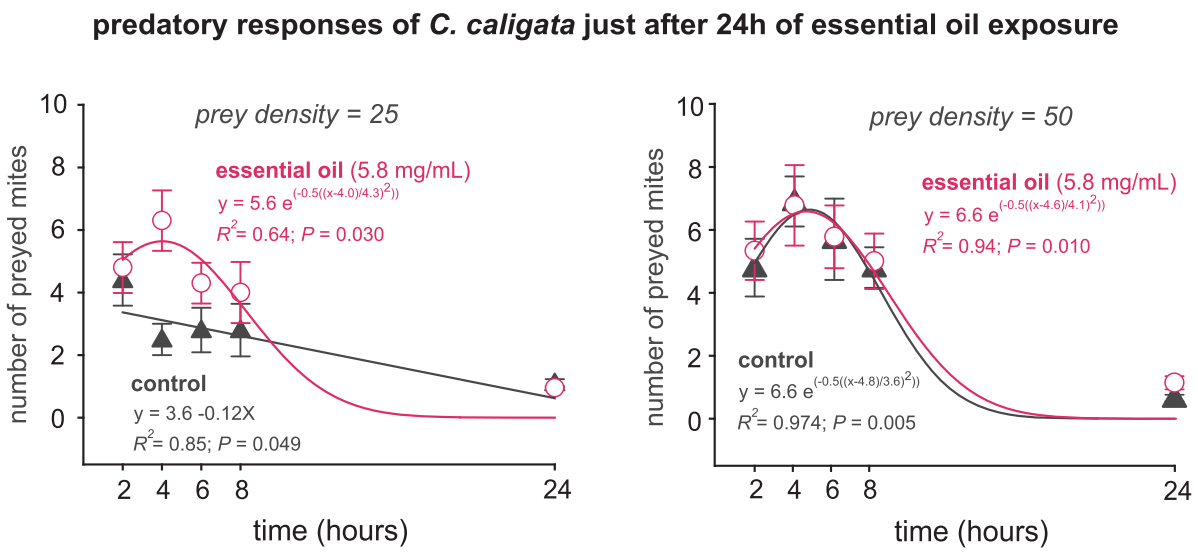

Fig. 2. Number of *Mononychellus tanajoa* adults eaten by *Ceraeochrysa caligata* larvae after 24 h exposure to 'Persian lime 58' essential oil. The ability of predators was assessed at densities of 25 and 50 *M. tanajoa* mites/arena (i.e., 7.1 cm^2). Mite density was reestablished after each evaluation. Symbols show the average number of mites predated on by each *C. caligata* larva (n = 10). Data are the mean ± SE.

3.4. Consumption of *M. tanajoa* by *C. caligata* larvae

It was further evaluated whether sublethal exposure to 'Persian lime 58' essential oil reduced the predatory ability of *C. caligata* larvae. Predators were exposed to a 'Persian lime 58' essential oil concentration of 5.8 mg/mL, which corresponded to the LC_{50} estimated in the concentration-mortality bioassays with *M. tanajoa* (Fig. 2).

The analysis of variance with repeated measures over time for the number of *M. tanajoa* adults eaten by the *C. caligata* larvae showed significant effects of *M. tanajoa* density, evaluation day (i.e., immediately after essential exposure and over the subsequent two days), and their interaction (Table 3). This analysis also showed the significant effects of time and the interactions between time and essential oil exposure, time, and *M. tanajoa* density and time and evaluation day (Table 3). When the larvae of *C. caligata* were exposed to an environment with higher densities of *M. tanajoa* (i.e., 50 mites in a 7.1 cm^2 arena), previous exposure to 'Persian lime 58' essential oil did not impact their predatory responses (Fig. 2). Interestingly, when essential oil sublethally-exposed predators faced an environment with prey scarcity (i.e., 25 mites in a 7.1 cm^2 arena) immediately after exposure (i.e., at the first evaluation day), their predatory ability was significantly higher than that of predators not exposed to the essential oil (Fig. 2). However, such enhanced predatory responses were not detected for the total number of *M. tanajoa* preyed upon by exposure to sublethal concentrations of essential oils on the subsequent days at any prey density.

Table 3
Repeated measures ANOVAs for the consumption of adults *Mononychellus tanajoa* by *Ceraeochrysa caligata* larvae that were unexposed or sublethally exposed to the 5.8 mg/mL of 'Persian lime 58' essential oil, which correspond to the LC_{50} obtained for *M. tanajoa* mites.

Variation source	df[a]	F-test	P-value
Between subjects			
Essential oil (EO)	1	0.02	0.89
Density (D)	1	59.85	< *** 0.001
Evaluation days (ED)	2	165.68	< *** 0.001
EO vs D	1	0.22	0.64
EO vs ED	2	0.88	0.42
D vs ED	2	8.26	< *** 0.001
D vs EO vs ED	2	0.38	0.68
Error	105	–	–

Within subject effects	df_{den}[b]	df_{num}[c]	Wilks' lambda	F	P
Time in hours (T)	103	3	0.573	25.55	< *** 0.001
T vs EO	103	3	0.902	3.72	*** 0.01
T vs D	103	3	0.957	1.52	0.21
T vs ED	206	6	0.761	5.02	< *** 0.001
T vs EO vs D	103	3	0.975	0.89	0.44
T vs EO vs ED	206	6	0.896	1.94	0.08
T vs D vs ED	206	6	0.990	0.16	0.98
T vs EO vs D vs ED	206	6	0.965	0.62	0.72

[a] degrees of freedom.
[b] within effects.
[c] between effects.

4. Discussion

Citrus essential oils represent potential tools for integration in the management programs of insect and mite pests (Camara et al., 2015; Dutra et al., 2016; Campolo et al., 2017; Papanastasiou et al., 2017). This study determines the chemical composition of essential oils from four *Citrus* scions and shows their potential to control the cassava green mite *M. tanajoa*. Furthermore, this study also demonstrated that exposure to sublethal concentrations of acid lime essential oil did not affect but, under certain circumstances, increase the predatory behavior of *C. caligata* larvae. Overall, factors (such as plant parts, harvesting season, extraction, and analytical methods) exerted an influence on the chemical composition of essential oils (Ellouze et al., 2012; Wu et al., 2013; Andrade et al., 2016; Moghaddam and Mehdizadeh, 2017). The chromatographic results revealed a similar pattern, with sabinene and linalool representing the main components of sweet oranges and mandarin essential oils, while limonene made up more than half of acid lime essential oil (Table 1).

Sabinene is the main component of the essential oils of *C. sinensis* orange leaves (Kasali et al., 2011; Germanà et al., 2013; Družić et al., 2016; Eldahshan and Halim, 2016). This compound certainly contributed to the toxicity of the essential oils of sweet oranges and mandarin to *M. tanajoa*, as previously demonstrated for other arthropod pests, including *Culex quinquefasciatus* S. (Diptera: Culicidae) and *Sitophilus zeamais* M. (Coleoptera: Curculionidae) (Wang et al., 2011; Pavela et al., 2018). The other major component of these *Citrus* scions was linalool, which is highly toxic to *Tyrophagus putrescentiae* S. (Acari: Acaridae), (Sánchez-Ramos and Castañera, 2000), thrips (*Thrips palmi* K.) (Thysanoptera: Thripidae) (Kim et al., 2015), lepidopteran pests (Pavela, 2014), and fruit flies (*Ceratitis capitata* W., *Bactrocera dorsalis*

H., *Bactrocera cucurbitae* C.) (Diptera: Tephritidae) (Chang et al., 2009). However, other compounds that were present in minor concentrations (e.g., δ-3-carene, myrcene, limonene, *(E)*-β-ocimene, and terpinen-4-ol) alone or in some combinations may potentiate the essential oil bioactivity against arthropod pests. Indeed, synergy among the major and minor components underpins the insecticidal/acaricidal activity of several essential oils (Pavela, 2014; Camara et al., 2015; Tak and Isman, 2017). For instance, terpinen-4-ol inhibits the activity of *Musca domestica* L. (Diptera: Muscidae) ATPases (Abdelgaleil et al., 2004). In particular, certain compounds exhibit residual repellency to *Tetranychus urticae* K. (Acari: Tetranychidae), including limonene and δ-3-carene (Camara et al., 2015).

Limonene is the major component of acid lime essential oil, and exerts its insecticidal and acaricidal activities by inhibiting acetylcholinesterase (Abdelgaleil et al., 2009; Kim et al., 2013; Zarrad et al., 2015). The current study demonstrated that the two limonene isomers (i.e., *(S)*-limonene and *(R)*-limonene) were equally toxic against *M. tanajoa*. Similar results were obtained for *Aedes albopictus* S. (Diptera: Culicidae) and *S. zeamais* (Giatropoulos et al., 2012; Fouad and Camara, 2017). In contrast, *(S)*-limonene was more toxic than *(R)*-limonene on *M. domestica* and *Dendroctonus ponderosa* H. (Coleoptera: Curculionidae) (Palacios et al., 2009; Chiu et al., 2017). Contradictory results were obtained for the repellency of various limonene isomers. While some investigations demonstrated that *(S)*-limonene repelled insect pests strongly (Giatropoulos et al., 2012; Fouad and Camara, 2017), other studies showed that *(R)*-limonene had higher repellency (Malacrinò et al., 2016). This variability in the activity of limonene isomers might result from the specific conditions under which the bioassays were conducted, or from the physiology of the arthropod species tested, which might have genetic characteristics of tolerance to one of these molecules (Malacrinò et al., 2016).

Interestingly, despite being able to surviving exposure (after 24 h exposure) to sublethal concentrations of acid lime essential oil, the survival of adult cassava green mites declined (Fig. 1). Reduced survival causes a further reduction in the oviposition and feeding by pests (Yaninek et al., 1989), which could diminish the population growth of such organisms. Furthermore, acid lime essential oil exhibited selectivity against the larvae of the lacewing predator *C. caligata*. This finding supports the results obtained for other essential oils against lacewing (Castilhos et al., 2018). However, the potential impact of these botanical compounds on the predatory ability of such generalist predators has been overlooked by previous studies, increasing the pioneering status of the present investigation. This study demonstrates that exposure to sublethal concentrations of acid lime essential oil minimally impacted the ability of *C. caligata* to prey on *M. tanajoa* adults; however, under conditions with increased stress (e.g., immediately after 24 h exposure and when prey density was low) the predatory response was higher (Fig. 2). This phenomenon might be an essential oil–induced hormetic-like response (Haddi et al., 2015; Papanastasiou et al., 2017; Silva et al., 2017). Although the physiological basis for regulating such stimulatory responses (i.e., hormesis) in insects is not well understood, such effects might result from disruptions of endocrine, antioxidant, and/or detoxification systems (Rand et al., 2015; Guedes et al., 2017).

5. Conclusions

This study demonstrates that the four different *Citrus* scion cultivars tested produced essential oils of equal toxicity against a relevant agricultural pest (i.e., the cassava green mite *M. tanajoa*), without causing major threats to a key generalist predator, the lacewing *C. caligata*. These findings reinforce the potential of *Citrus* essential oils as relevant and safer tools to control arthropod pests. Future studies should focus on identifying the molecular loci on which the major components (e.g., sabinene, linalool or limonene) of these *Citrus* essential oils act, which would help establishing novel management programs for the control of the cassava green mite, *M. tanajoa* in Brazil.

Ethical approval

All applicable international, national, and institutional guidelines for the care and use of animals were considered in the present investigation.

CRediT authorship contribution statement

Adriano Pimentel Farias: Conceptualization, Methodology, Investigation, Formal analysis, Writing - original draft. **Maria Clezia dos Santos:** Methodology, Investigation, Formal analysis. **Luis Oswaldo Viteri Jumbo:** Conceptualization, Methodology, Investigation, Formal analysis. **Eugênio E. Oliveira:** Conceptualization, Formal analysis, Writing - review & editing. **Paulo César de Lima Nogueira:** Formal analysis, Resources. **José Guedes de Sena Filho:** Formal analysis, Resources, Writing - review & editing. **Adenir Vieira Teodoro:** Conceptualization, Funding acquisition, Resources, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no conflict of interest.

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