Contents lists available at ScienceDirect

Asian Pacific Journal of Tropical Biomedicine

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

Original article http://dx.doi.org/10.1016/j.apjtb.2017.08.006

Acaricidal activity of *Derris floribunda* essential oil and its main constituent

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ARTICLE INFO

Article history:
Received 17 Jul 2017
Received in revised form 28 Jul 2017
Accepted 14 Aug 2017
Available online 19 Aug 2017

Keywords:
Acaricidal activity
Essential oil
Nerolidol
Organic agriculture
Sesquiterpenes
Timb

ABSTRACT

Objective: To evaluate the acaricidal activity of the essential oil obtained from roots of *Derris floribunda* (*D. floribunda*) (Miq.) Benth, and its main constituent nerolidol against the Mexican mite *Tetranychus mexicanus* (*T. mexicanus*) (McGregor).

Methods: The essential oil from the roots of *D. floribunda* collected in the Amazon region (Brazil) was obtained by hydrodistillation. Its chemical composition was determined by GC–MS analysis. The acaricidal activities of this essential oil and nerolidol, were evaluated by recording the number of dead females (mortality) and eggs (fertility).

Results: The essential oil showed sesquiterpenes as major volatile components. Nerolidol, the main component, represented 68.5% of the total composition of the essential oil. *D. floribunda* essential oil and nerolidol showed acaricidal activity, with LC_{50} of 9.61 μg/mL air and 9.2 μg/mL air, respectively, over a 72 h period. In addition, both the essential oil and nerolidol significantly reduced the fecundity of *T. mexicanus*.

Conclusions: Due to the economic importance of *T. mexicanus* and the lack of new pesticides, our data are very promising in the search for efficient and safer acaricidal products. Furthermore, this is the first report about the chemical composition and bioactivity of the essential oil of the Amazon plant species *D. floribunda*.

1. Introduction

The use of timbó as an ictiotoxic agent has long been known and used by the natives of the Amazon region for catching fish. Timbó is a popular term in several taxa, which grows mainly in the Amazon region, with the most important plant genera belonging to the Fabaceae family *Derris* and *Lonchocarpus* [1]. Alecio *et al*. [2] reported that the rotenoids found in the roots, such as rotenone, deguelin, tephrosin and toxicarol stand out as extremely toxic bioactive substances, and indicate the biological importance of these plants. Rotenone is the most potent of these rotenoids, and it is used to control a wide range of arthropod pests [3].

It is interesting to note that fish poisoned with 'timbó' can be consumed without any risk of food poisoning. This feature, together with the fact that these plants have pesticide activity, has led to their use in agriculture and livestock [4,5]. Biological activities have recently been reported against the cattle tick *Rhipicephalus* (*Boophilus*) *microplus* (Canestrini) [6], and against one of the most important agricultural pests, *Cerotoma arcuata* Olivier, a polyphagus pest of dry beans, soybean and cowpea [7,8].

In this context, special attention should be given to the control of mites due to the high impact on agriculture of damage caused by infestation of different crops, which has led to the exorbitant use of acaricides worldwide, involving expenses of about 900

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Peer review under responsibility of Hainan Medical University. The journal implements double-blind peer review practiced by specially invited international editorial board members.

million euros [9]. *Tetranychus mexicanus* (*T. mexicanus*) (McGregor, red mite) is a pest that affects some economically important crop species. There have been reports of damage in coconut leaves [10], species of *Citrus* [11], *Annona* [12] and *Vitis* [13] in various regions of the Americas. Due to the high fertility and short life cycle of mites of red mite, frequent acaricide application is required. However, this has led to the development of resistance to acaricides in mites [14]. In order to avoid the emergence of resistant populations by selection pressure, and prolong the effective life of the active ingredients, the Insecticide Resistance Action Committee recommends acaricide rotation of different groups [15].

Natural products, especially essential oils, have been evaluated and found to be promising as alternatives to synthetic acaricides, mainly due to their rapid degradation in the environment, lack of bioaccumulation, and low toxicity [16]. The use of *Derris* species in the composition of insecticides is well known, as evidenced by the existence of old and recent patents [17–21].

Besides rotenoids such as rotenone, 12a-hydroxyrotenone, 6a, 12a-dehydrorotenone and tephrosin [22,23], the following compounds have also been detected in *Derris* species: isocordoin, sitosterol, (3S)-2′-*O*-methylvestiol, lupenone, terocarpin, 3-methoxy-8,9-methylenedioxy-6a and 11a-dehydropterocarpan [22], fatty acids-oleic acid, linoleic acid and palmitic acid [24], stilbene derivatives-3,5-dimethoxy-4-prenylstilbene, 3,5,4′-trimethoxy-4-prenylstilbene, and flavonoids (lonchocarpin, 5,7-dihydroxy-6-prenylflavanone, 4-hydroxylonchocarpin and (3S)-7-hydroxy-2′, and 4′-dimethoxyisoflavan) [22].

This paper reports, for the first time, the chemical composition of the essential oil obtained from roots of *Derris floribunda* (*D. floribunda*) (Miq.) Benth, the acaricidal activity of this essential oil, and its main constituent, nerolidol, against *T. mexicanus*, a mite that damages various cultivated plant species, causing significant economic losses.

2. Material and methods

2.1. Botanical material

Roots of *D. floribunda* were collected in October 2012 from the metropolitan region of Manaus, state of Amazonas (03°16′20″S; 060°11′22.7″W). The plant material was compared with botanical specimens deposited at the Herbarium of INPA (Amazonas state), and a voucher was deposited under number 225683. The roots were washed, dried at 40 °C in an air circulation oven, and chopped.

2.2. Extraction of essential oil

Roots of *D. floribunda* (300 g) were chopped and submitted to hydrodistillation (4 h) using a modified clevenger-type apparatus. After distillation, the oil was collected, dried by anhydrous Na_2SO_4 and placed in a centrifuge. The supernatant was removed and transferred to glass flasks and stored at −4 °C for further use. Yields were calculated based on the weight of the plant material used. *E*-nerolidol analytical standard was obtained from Sigma Aldrich Corporation.

2.3. Essential oil analysis by gas chromatography/mass spectrometry (GC/MS)

The identification of essential oil constituents was performed by comparison of their retention indices and mass spectra with those reported in the literature [25] or presented in the Wiley data system library of the GC–MS equipment. The retention indices were calculated for all volatile constituents using *n*-alkane homologous series. GC–MS analyses were performed using a HP 6890 gas chromatograph interfaced with a HP 5973N Mass Selective Detector (ionization voltage 70 eV), equipped with a DB-5MS capillary column (30 m × 0.25 mm, film thickness 0.25 μm), using helium as carrier gas (1.0 mL/min). The oven temperature was programmed from 60 °C to 290 °C at a rate of 3 °C/min, then isothermal at 290 °C for 10 min, using H_2 as the carrier gas (1.0 mL/min). Injector and detector temperatures were 230 °C and 290 °C, respectively. Injection volume was 1.0 μL (2 mg of the sample in 1 mL of CH_2Cl_2), in splitless mode. Linear velocity ($\bar{u}$) was 14 cm/s. MS interface temperature: 280 °C; mass range: 40–700 u; scan speed: 150 u/s; interval: 0.50 s (2 Hz).

2.4. Stock rearing of mites

The Mexican mites, *T. mexicanus*, were obtained from stock cultures maintained under laboratory conditions with (27.4 ± 0.3) °C, 60.0% ± 2.1% RH. Papaya (*Carica papaya*) leaves infested with *T. mexicanus* were collected at the campus of the Universidade Federal do Amazonas (Amazonas Federal University) (03° 6′ 0.975″S; 59° 58′ 30.229″W). Mites were transferred to the test area using a fine bristled brush, with the aid of a stereoscopic microscope (Nikon SMZ800, zoom range was 1× to 6.3×, zoom ratio was 6.3:1).

Test arenas were prepared as follows: a piece of polyethylene foam was placed in a plastic ray (15 cm × 30 cm) and a filter paper disk was placed over the foam, to prevent direct contact of the leaf with water. The surface of a piece of a papaya leaf was sterilized in 1% hypochloride solution for 30 s and the leaf was placed on the filter paper. Moistened cotton was placed around the entire leaf edge to prevent the escape of mites. The base of the test arena was moistened daily with distilled water to prevent premature drying of the leaf, and mites were transferred to new test arenas when necessary [26].

2.5. Acaricidal activity of D. floribunda essential oil and nerolidol

The acaricidal potential of essential oil and nerolidol were evaluated according to Sertkaya *et al.* [27] with some modifications. Glass Petri dishes (90 mm × 20 mm) were used to perform the assays. Papaya leaf disks of 3.33 cm in diameter were arranged in a half of Petri dish (90 mm × 20 mm) containing polyethylene foam and filter paper. Next, 10-d old adult females were transferred to the experimental unit (abaxial surface of leaves) using a brush, 30 min before the application of the essential oil or nerolidol.

The samples were diluted at seven different concentrations (100, 75, 60, 50, 40, 20 and 10 mg/mL) in dimethylsulfoxide. The inner surface of the inverted lid of the Petri dish, covered with filter paper, was used to apply the sample. An aliquot 10 μL of each concentration was applied using a pipette, placed 20 mm from the center of the dish, gave concentrations of 10, 7.5, 6.0, 5.0, 4.0, 2.0 and 1.0 μg/mL air. The experimental units were sealed with parafilm after application of samples, to prevent loss of essential oils from the plates. The test was performed in six replicates for each concentration. The experiments were performed 24, 48 and 72 h after application of the extracts,

recording the number of dead females (effect on survival) and eggs in each unit. The experimental units were incubated at (25.0 ± 0.3) °C, RH 62.0% ± 5.7% and 12 h of photophase.

2.6. Data analysis

Data were submitted to analysis of variance (one-way ANOVA), followed by the Tukey test using software Statistica version 6.0, StatSoft Inc. Homoscedasticity and normality of residues were verified by Bartlett's test and Kolmogorov–Smirnov test, respectively. The significance level was set at $P = 0.05$. The Probit Analysis Method was used to determine the 50% lethal concentrations (LC_{50}). Statistica version 6.0 was used for data representation.

3. Results

3.1. GC–MS analysis of the essential oil of D. floribunda

The essential oil obtained by hydrodistillation of *D. floribunda* roots was orange in color, with a yield of 0.2% (w/v based on the weight of the roots). The essential oil was composed of seventeen sesquiterpenes, of which over 90% are oxygenated (Table 1). Nerolidol was the main component, representing 68.5% of the total composition of the essential oil.

Table 1
Chemical composition of the essential oil from roots of *D. floribunda*.

Compounds	$RI^{lit.}$	$RI^{cal.}$	Oil composition (%)
β-bisabolene	1505	1509	0.9
δ-cadinene	1522	1520	1.0
Italicene ether	1536	1533	0.8
E-nerolidol	1561	1554	68.5
Spathulenol	1577	1581	1.6
Earyophyllene oxide	1582	1582	0.9
α-cedrol	1600	1595	2.3
Guaiol	1600	1602	3.3
Epi-cedrol	1618	1617	4.4
1-epi-cubenol	1627	1628	0.8
δ-cadinol	1630	1631	1.0
Gossonorol	1636	1634	1.1
β-bisabolol	1670	1675	3.3
Epi-α-bisabolol	1683	1682	1.7
α-bisabolol	1685	1685	1.5
Eudesma-4(15),7-dien-1β-ol	1687	1686	1.2
Nootkatol	1714	1712	0.8
Terpenoids class	–	–	
Hydrocarbon sesquiterpenes	–	–	1.9
Oxygenated sesquiterpenes	–	–	93.2
Identified components	–	–	95.1

$RI^{cal.}$: Retention Indices calculated based on a homologous series of normal alkanes; $RI^{lit.}$: Retention Indices from literature.

3.2. Acaricidal assays with D. floribunda essential oil

The mortality rates of *T. mexicanus* after 48 h and 72 h of the initial exposure to the essential oil obtained from roots of *D. floribunda* at concentrations of 6–10 µg/mL were different from control (Table 2). Maximum mortality was 53.3% ± 12.11%, achieved with a concentration of 10 µg/mL air after 72 h. It was significantly different from the mortality achieved with other concentrations tested.

Figure 1 shows the representation of Probit Analysis. The equation regression was $y = 0.0327 + 0.0486\ x$, where y is mortality after 72 h and x is concentration of the essential oil, with coefficient of determination (r^2) of 0.9797. The essential oil showed LC_{50} of 9.61 µg/mL air over a 72 h-period.

The essential oil of *D. floribunda* also affected fecundity of *T. mexicanus* (Table 3). After 48 h and 72 h, the essential oil of *D. floribunda* at 5 µg/mL air significantly reduced fecundity, compared to the control. Effects on mite fecundity soon after exposure – 24 and 48 h – were only detected for exposure to higher concentrations of essential oil (10 µg/mL air).

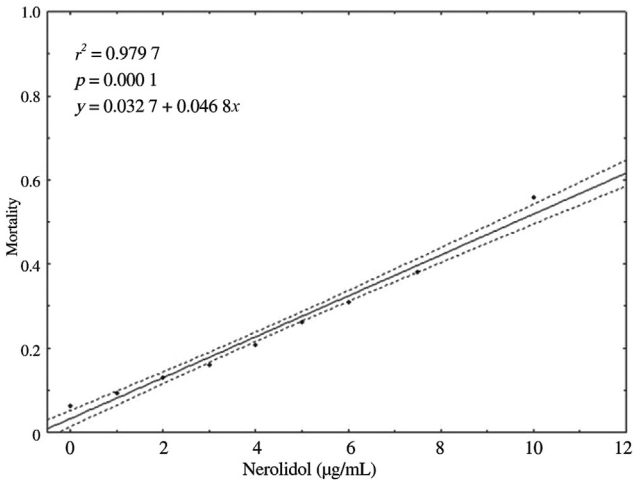

Figure 1. Probit analysis to evaluate the acaricidal activity of the essential oil of *D. floribunda* against *T. mexicanus*.

Table 2
Mortality of *T. mexicanus* after exposition to the essential oil of *D. floribunda* and *E*-nerolidol.

Concentration (µg/mL of air)	Essential oil of *D. floribunda*			*E*-nerolidol		
	24 h	48 h	72 h	24 h	48 h	72 h
0	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	1.70 ± 4.08^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}
1	0.00 ± 0.00^{a}	3.30 ± 5.16^{ab}	6.70 ± 5.16^{ab}	–	–	–
2	3.30 ± 5.16^{a}	6.70 ± 5.16^{abc}	15.00 ± 8.37^{abc}	–	–	–
4	3.30 ± 5.16^{a}	10.00 ± 6.32^{abc}	16.70 ± 10.33^{abc}	0.00 ± 0.00^{a}	3.30 ± 5.16^{a}	5.00 ± 5.48^{ab}
5	5.00 ± 5.48^{a}	11.70 ± 7.53^{abc}	16.70 ± 10.33^{abc}	1.70 ± 4.08^{a}	6.70 ± 5.16^{ab}	8.30 ± 7.53^{ab}
6	5.00 ± 5.48^{a}	13.30 ± 8.16^{bcd}	20.00 ± 6.32^{bc}	5.00 ± 8.37^{a}	10.00 ± 8.94^{ab}	16.70 ± 12.11^{bc}
7.5	6.70 ± 8.16^{a}	16.70 ± 5.16^{cd}	25.00 ± 5.48^{c}	5.00 ± 5.48^{a}	16.70 ± 10.33^{b}	25.00 ± 5.48^{c}
10.0	10.00 ± 8.94^{a}	25.00 ± 10.49^{d}	53.30 ± 12.11^{d}	18.30 ± 7.53^{b}	35.00 ± 8.37^{c}	58.30 ± 7.53^{d}

Mean values ± standard deviation of six replicates. Means followed by the same letter within each column are not significantly different based on the Tukey's test ($P < 0.05$).

Table 3
Fecundity (eggs/replicate) of *T. mexicanus* after exposition to the essential oil of *D. floribunda* and *E*-nerolidol.

Concentration (μg/mL of air)	Essential oil of *D. floribunda*			*E*-nerolidol		
	24 h	48 h	72 h	24 h	48 h	72 h
0	37.00 ± 4.94^{abc}	52.70 ± 7.28^{a}	63.70 ± 7.97^{a}	37.30 ± 5.47^{a}	54.70 ± 5.32^{a}	66.80 ± 7.11^{a}
1	38.00 ± 2.28^{a}	51.50 ± 2.88^{ab}	59.70 ± 3.08^{ab}	–	–	–
2	37.30 ± 2.34^{ac}	50.20 ± 3.82^{abc}	60.00 ± 2.83^{ab}	–	–	–
4	31.70 ± 4.27^{bc}	42.00 ± 5.25^{c}	58.00 ± 3.45^{ab}	37.20 ± 2.14^{a}	45.30 ± 1.21^{b}	53.20 ± 2.64^{b}
5	34.70 ± 2.88^{abc}	48.80 ± 4.75^{abc}	54.80 ± 3.13^{b}	34.50 ± 2.74^{a}	42.00 ± 2.28^{bc}	51.20 ± 3.25^{bc}
6	32.50 ± 2.88^{abc}	44.00 ± 3.58^{bc}	54.50 ± 5.86^{b}	35.80 ± 2.64^{a}	43.20 ± 2.04^{b}	50.70 ± 1.51^{bc}
7.5	30.80 ± 2.64^{b}	41.80 ± 2.14^{c}	53.80 ± 3.37^{b}	31.50 ± 3.56^{ab}	37.30 ± 2.94^{cd}	45.50 ± 1.52^{c}
10.0	22.20 ± 3.92^{d}	30.70 ± 4.76^{d}	39.70 ± 3.87^{c}	25.50 ± 3.51^{b}	32.70 ± 1.86^{d}	37.50 ± 1.64^{d}

Mean values ± standard deviation of six replicates. Means followed by the same letter within each column are not significantly different based on the Tukey's test ($P < 0.05$).

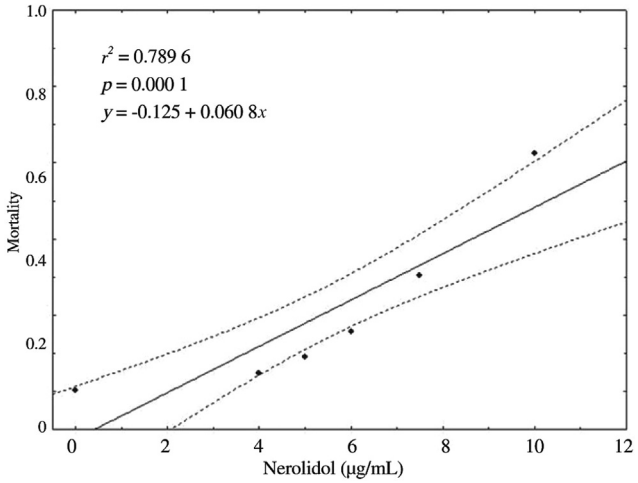

Figure 2. Probit analysis to evaluate the acaricidal activity of nerolidol against *T. mexicanus*.

3.3. Acaricidal assays with E-nerolidol

Nerolidol significantly induced mortality in *T. mexicanus* exposed to 10 μg/mL air after 24 h of the initial exposure. The effect was directly related to nerolidol concentrations after 48 h and 72 h, achieving maximum mortality of 58.3% at 10 μg/mL air after 72 h (Table 2). Nerolidol showed LC_{50} of 9.2 μg/mL air over a 72 h period, calculated by Probit Analysis Method (Figure 2). The equation regression was $y = -0.125 + 0.0608\ x$, where y is mortality after 72 h and x is concentration of the essential oil, with coefficient of determination (r^2) of 0.7896.

Nerolidol also affected the fecundity of *T. mexicanus* (Table 3). Reduced fecundity was observed after 24 h of initial exposure at 10 μg/mL air. At 48 h and 72 h, the decrease in fecundity was from 4.0 μg/mL air.

4. Discussion

There have been few studies on the composition of extracts of *D. floribunda*, and no information is available on the composition of essential oil from its roots. Among the phytochemical studies described in the literature, Braz-Filho *et al*. [22] isolated various phenolic compounds from petroleum extract of *D. floribunda* roots. The essential oil of *Derris indica* (Lam.) Bennet was studied by Rai *et al*. [24], and its composition was found to be rich in fatty acids, which is very different from the essential oil of *D. floribunda* obtained by us in this work. However, there are no references to the plant organ of *Derris indica* investigated by the authors.

We demonstrated that the essential oil of *D. floribunda*, and its main constituent nerolidol, showed similar acaricidal activity. The effects of the essential oil and nerolidol on mites were dose- and exposure-dependent, *i.e.*, the higher the concentration and the longer the exposure time, the greater the mortality. However, we also demonstrated the nerolidol was slightly more active than the essential oil, and 18.4% mortality was detected after 24 h of exposure. Fecundity was also affected by treatments with *D. floribunda* essential oil and nerolidol.

The biological evaluation of a significant number of essential oils obtained from plant species belonging to different botanical families, against a variety of mite species of the Tetranychidae family [27], mainly against *Tetranychus urticae* (*T. urticae*), indicates a promising alternative in the search for new acaricide agents. Some examples are the use of extracts from the families Myrtaceae [28], Verbenaceae [29], Burseraceae [30], Piperaceae [31] and Lamiaceae [32], among others [33]. To our knowledge, there have been no reports on the acaricidal activity of *T. mexicanus*.

In a screening study, Attia *et al*. [33] evaluated the acaricidal activity of the essential oil from 31 plant species, representing 17 families, against *T. urticae* mite. In these assays, the authors demonstrated that the essential oils and some of the constituents are important potential insecticides. Similarly, fresh oleoresin of *Protium bahianum* Daly, which contained monoterpenes and sesquiterpenes, led to 79.6% mortality of the *T. urticae* mite after 72 h of exposure. Aged oleoresin, rich in sesquiterpene β-(*E*)-santalol acetate (83%), caused 59.9% mortality after 72 h, suggesting that sesquiterpenes also had important acaricidal activity. The results were similar to those described by us for *T. mexicanus* using other species of *Tetranychus*.

The literature describes the acaricidal activity of the sesquiterpene nerolidol, which together with β-caryophyllene and α-humulene, is considered important in the acaricidal activity and repellency of *Piper aduncum* essential oil against *T. urticae*. Although it is likely that there is a synergistic effect with other substances present in the oil, the mortality is mainly attributed to β-caryophyllene by fumigation and contact, while nerolidol is considered the most active in terms of repellent action, with 83.2% repellency in 2 h, equivalent to the positive control eugenol [34]. Lage *et al*. [35] studied another mite, *Rhipicephalus microplus* (Canestrini, 1887), and demonstrated that nerolidol has activity on it. These results reinforce the potential and versatility of this class in the search for acaricidal agents.

Concerning the biological activity of the essential oils on mites, the mechanism of action is not fully understood. Physical–chemical characteristics related to lipophilicity and high vapor pressure, combined with structural characteristics such as the presence of oxygen in the molecule and the ability to form hydrogen bond [31,36], could contribute to the biological activity of this class of substances.

Although some studies are related to the acaricidal activity of essential oils, there is no report on the biological effects against *T. mexicanus*. This work therefore represents a significant contribution to this field. Nerolidol may represent an alternative source of synthetic acaricides. Additionally, this paper is the first to present the chemical composition and bioactivity of the essential oil of *D. floribunda*, an important plant species from the Amazon region.

Conflict of interest statement

The authors declare that they have no conflict of interest.

Acknowledgments

J.M.S.M thanks fellowship from a CNPq fellowship. This work was financially supported by Fundação de Amparo à Pesquisa do Estado do Amazonas (FAPEAM – PAPAC program EDITAL N. 015/2014).

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