

Chemical Composition and Insecticidal Activity of Essential Oils from *Vanillosmopsis pohlii* Baker against *Bemisia argentifolii*

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Essential oils from the heartwood and leaves of specimens of *Vanillosmopsis pohlii* collected in two different localities were analyzed by GC-MS. The major constituent of both heartwood essential oils was the sesquiterpene α -bisabolol. Essential oil composition from leaves was quite different for two specimens and showed β -pinene and *E*-caryophyllene as principal constituents. The essential oil of heartwood and the pure sesquiterpene α -bisabolol were tested against *Bemisia argentifolii*, the white fly fruit plague, and pronounced insecticidal effects were observed.

KEYWORDS: *Vanillosmopsis pohlii*; *Bemisia argentifolii*; α -bisabolol; *E*-caryophyllene; β -pinene; insecticidal activity

INTRODUCTION

Vanillosmopsis is a small genus of the Asteraceae family, represented by seven species native to Brazil (1). Previous reports on essential oils from species of *Vanillosmopsis* have indicated high contents of α -bisabolol (2), a sesquiterpene of commercial value for its antiphlogistic properties and wide use as an antiinflammatory in the comestic industry and obtained industrially from the essential oil of *Chamomilla recutita* (Asteraceae) (3). This fact spurred great commercial and agronomic interest in species of the *Vanillosmopsis* genus. The difference in price between (–)- α -bisabolol from *Vanillosmopsis* oil and that from chamomile has often led to replacement of (–)- α -bisabolol from chamomile with the cheaper *Vanillosmopsis* compound.

In continuation of our investigation to find biologically active essential oils from Brazilian flora, we report herein the chemical variation of essential oils from leaves and heartwood from two populations of *Vanillosmopsis pohlii* Baker, a small shrub that grows in abundance in northeastern Brazil, where it is popularly known as “candeeiro” and used as an antiinflammatory by the

local population. Previous phytochemical studies from the aerial parts of *V. pohlii* revealed triterpenes and goyazensanolide-type sesquiterpenes (4). However, the volatile compositions of these species have never been reported.

The insecticidal activities of essential oil from the heartwood of *V. pohlii* and of the major component α -bisabolol were evaluated against *Bemisia argentifolii* (white fly), a common pest in Brazil, responsible for the devastation of crops of cotton and edible fruits of commercial value such as melon and watermelon.

MATERIALS AND METHODS

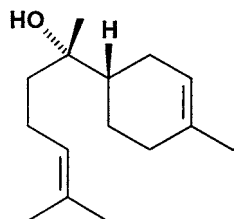
Plant Material. Leaves and heartwood of wild-growing *V. pohlii* plants were collected in February 2000 at the flowering stage, from localities of Seabra and Lençóis Condados, at altitudes of 460 and 850 m, respectively, in Chapada da Diamantina (Diamantina’s Plateau), Bahia state, northeastern Brazil. Voucher specimens (30090 and 29167, respectively) have been identified by Dr. Afrânio G. Fernandes and deposited at the Herbário Prisco Bezerra (EAC), Departamento de Biologia, Universidade Federal do Ceará, Brazil.

Analytical Conditions. GC-MS analysis was carried out on a Hewlett-Packard computerized system, model 5971A, working in electron impact mode at 70 eV, employing a DB-5 fused silica capillary

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Table 1. Collection Site, Plant Part, and Oil Yields of *V. pohlii*

| collection site | plant part | oil yield (v/dry wt) |
|-----------------|------------|----------------------|
| Seabra | leaves | 0.2 |
| | heartwood | 0.2 |
| Lençóis | leaves | 0.03 |
| | heartwood | 0.04 |

**Figure 1.** Structure of α -bisabolol.

column (30 m \times 0.25 mm i.d.; film thickness = 0.25 μ m). Helium was used as the carrier gas at a flow rate of 1 mL/min, and the column temperature was programmed from 35 to 180 $^{\circ}$ C at a 4 $^{\circ}$ C/min rate and from 180 to 280 $^{\circ}$ C at a 10 $^{\circ}$ C/min rate. Injector and detector were maintained at 250 and 200 $^{\circ}$ C, respectively. Individual components were identified by comparing their mass spectra with those of the spectrometer database using the Wiley L-built library and two computer library MS searches based on their Kovats retention indices as a preselection routine (5, 6) and by comparison of the fragmentation pattern with published spectral data (7, 8).

Isolation of the Volatile Constituents. Leaves and heartwood of two specimens of *V. pohlii* were separately submitted to hydrodistillation for 3 h in a Clevenger-type apparatus. The essential oils obtained were dried over anhydrous sodium sulfate. Data of extractions are shown in **Table 1**.

Isolation of α -Bisabolol. Essential oil from the stem of *V. pohlii* collected at Seabra county was submitted to chromatography on a silica gel column and eluted with a mixture of hexane/ CHCl_3 as a binary mixture with increasing polarity to give the pure α -bisabolol (**Figure 1**). The structural characterization was accomplished by mass spectral analysis and NMR experiments, uni- and bidimensional, a Bruker DRX 500 spectrometer, and posterior comparison with published data.

Bioassay Tests. Cultivation of Plants. Melon seeds were sowed in conical plastic flowerpots (10 cm height, 10 and 13 cm diameters, bottom and top, respectively) over a 7 cm layer of a substrate composed of 33% vermiculite, 33% manure, and 33% soil from Pacajus county, on the Experimental Station of EMBRAPA (Empresa Brasileira de Pesquisa Agropecuária, a governmental institution devoted to agricultural research in Paraipaba county, state of Ceará, Brazil). Each vessel received three seeds of the melon variety Hale's Best Jumbo and was isolated from the external environment by a piece of tulle held by a wire frame inside the vessel. They were kept under these conditions for 15 days when at least four to five fully developed leaves had emerged.

Obtention and Massal Rearing of the *B. argentifolii* Population. The insect parents were obtained from an experimental crop of melons maintained by the Experimental Station of EMBRAPA. The colony was kept captive in special "cages" with melon plants at the Laboratory of Entomology of EMBRAPA Agroindustria Tropical, in Fortaleza.

Vessels with seedlings were introduced into plastic cages (35 \times 48 \times 32 cm). The upper part and lateral wall were covered with tulle. Several vessels with plants of different ages were kept in the same cage in order to have the insect colony in different developing stages. Senescent plants were discarded and replaced by young ones.

Insecticidal Activity Assays. Seven-day-old leaves without insects were harvested. Testing substances were dissolved in DMSO/ H_2O 5% and applied on the abaxial face of each leaf, by a portable sprayer, and individually adjusted by its petiole to small vials with distilled water. Each vial containing one leaf was then stored in individual transparent acrylic cups (9.5 cm height, 5.0 and 7.0 cm diameters, bottom and top, respectively), partially covered with a transparent polyethylene film. Ten adult insects were introduced in each cup, the polyethylene cover

Table 2. Percentage Composition of Essential Oils from Leaves and Heartwood of *V. pohlii* of Two Localities

| compound ^b | KI ^c | county/part of plant/yield (%) ^a | | | |
|-------------------------|-----------------|---|-----------|--------|-----------|
| | | Lençóis | | Seabra | |
| | | leaves | heartwood | leaves | heartwood |
| 3-hexen-1-ol | 857 | 0.7 | | | |
| α -pinene | 939 | | | 6.0 | |
| β -pinene | 980 | 3.9 | | 44.1 | |
| β -myrcene | 991 | | | 1.1 | |
| limonene | 1031 | | | 3.9 | |
| α -copaene | 1376 | 2.9 | | 1.4 | |
| β -elemene | 1391 | | 0.7 | | |
| <i>E</i> -caryophyllene | 1418 | 58.1 | 4.6 | 25.3 | 2.2 |
| α -humulene | 1454 | 6.2 | 0.6 | 2.7 | |
| β -cubebene | 1512 | 8.5 | 0.5 | 6.2 | |
| γ -cadinene | 1513 | 0.9 | | | |
| δ -cadinene | 1524 | 3.6 | | 1.7 | |
| spathulenol | 1576 | 1.1 | 2.7 | | |
| bisabolol oxide | 1655 | 1.4 | 3.4 | | 2.4 |
| α -bisabolol | 1683 | | 79.0 | | 89.9 |
| total | | 89.0 | 92.1 | 92.4 | 94.5 |

^a % = percentage composition. ^b Order of elution on DB-5 column. ^c Kovats retention index according to C_8 – C_{26} *n*-alkanes on DB-5 capillary column.

Table 3. Effect of the Essential Oil from Leaves of *V. pohlii* of Lençóis against *B. argentifolii*

| treatment | concn (g/L) | av (%) of dead insects after 72 h | av (%) of layed eggs after 72 h |
|---|-------------|-----------------------------------|---------------------------------|
| essential oil | 2.00 | 100.0 | 1.2 |
| | 1.00 | 95.0 | 3.2 |
| | 0.50 | 69.0 | 24.0 |
| | 0.25 | 54.0 | 46.7 |
| Orthene 750BR | 0.50 | 54.0 | 48.7 |
| Witness (DMSO/ H_2O 5%) | | 28.0 | 86.6 |

was closed, and the vials were stored in a BOD chamber at 24 $^{\circ}$ C for a photoperiod of 14 h. Results (**Tables 2** and **3**) were recorded 72 h later by counting the layed eggs and the number of survivals. The experiments had entirely random delineation, and each leaf was considered to be an experimental parcel. Each treatment had eight repetitions.

RESULTS AND DISCUSSION

According to experimental data, the essential oil from *V. pohlii* collected at an altitude of 460 m showed greater yields when compared to the specimen collected at an altitude of 850 m. Results of the GC-MS analysis of the essential oil from leaves of *V. pohlii* from Lençóis are presented in **Table 2**. The chemical composition was essentially characterized by a large percentage of sesquiterpene hydrocarbons (86.1%), 7.4% of which were oxygenated. The main sesquiterpenes were *E*-caryophyllene (58.1%), β -cubebene (8.5%), and α -humulene (6.2%). The oxygenated sesquiterpenes were represented by spathulenol (1.1%) and bisabolol oxide (1.4%). Monoterpenes were found only in minor percentages (4.6%).

A great difference was observed in the essential oil of leaves collected in Seabra county (**Table 2**). Monoterpenes were the predominant class (55.0%), with large amounts of β -pinene (44.1%). Among the sesquiterpenes *E*-caryophyllene was the major compound (25.0%), but in minor contents compared with those observed in the Lençóis essential oil.

The essential oils of heartwood had very similar compositions for the two populations and showed only sesquiterpenes in their compositions (**Table 2**). From the essential oil of Lençóis, a

Table 4. Effect of the Essential Oil from Leaves of *V. pohlii* of Seabra against *B. argentifolii*

| treatment | concn (g/L) | av (%) of dead insects after 72 h | av (%) of layed eggs after 72 h |
|------------------------------------|-------------|-----------------------------------|---------------------------------|
| essential oil | 2.00 | 93.7 | 0.0 |
| | 1.00 | 95.0 | 0.0 |
| | 0.50 | 100.0 | 0.4 |
| | 0.25 | 80.0 | 5.8 |
| | 0.50 | 55.0 | 28.8 |
| Orthene 750BR | | | |
| Witness (DMSO/H ₂ O 5%) | | 22.5 | 80.6 |

high content of α -bisabolol (79.0%) was observed, whereas bisabolol oxide (3.4%), spathulenol (2.7%), and *E*-caryophyllene (4.6%) were found in low amounts. The essential oil of heartwood from Seabra was represented by just three compounds. The sesquiterpene α -bisabolol was the principal component (89.9%), and *E*-caryophyllene (2.2%) and bisabolol oxide (2.4%) were found in very low concentrations.

Identification of α -bisabolol as the major component of the essential oil from the heartwood of two populations of *V. pohlii* from northeastern Brazil is in accordance with previous investigations reported for others species of the *Vanillosmopsis* genus, such as *V. arborea* (2), which showed 80–90% yield for this compound. The high occurrence of α -bisabolol also in *V. pohlii* confirms the use of *Vanillosmopsis* species as an alternative natural source of α -bisabolol.

Essential oils from heartwood rich in α -bisabolol were tested against *B. argentifolii* insects, and the insecticide Orthene 750BR was used as a positive control for all analyses.

The experiments showed high mortality at 1 g/L dosages for both oils after 3 days. The essential oil from Seabra induced 100% mortality at 0.5 g/L concentration, whereas the essential oil from Lençóis showed 69% mortality for the same dosage. This suggested that the greater insecticidal activity found for the essential oil from Seabra could be attributed to the larger content of α -bisabolol in its composition (Tables 3 and 4).

In an effort to evaluate the individual contribution of the principal component of heartwood oils, the pure sesquiterpene α -bisabolol was tested under identical conditions to compare its activity with that of the investigated oils. Table 5 shows the experiments accomplished at 0.1, 0.25, 0.5, 1.0, and 2.0 g/L dosages and indicates that the insecticidal activity of α -bisabolol was quite similar to that of the essential oil from Seabra except at the 0.25 g/L concentration.

According to a literature survey, α -bisabolol is valued for its antiphlogistic properties and is regarded as one of the pharmacologically active components of chamomile (9). The results obtained led us to believe that *B. argentifolii* insects are susceptible to the composition of the essential oils from the heartwood of *V. pohlii*, and the high insecticidal activity

Table 5. Effects of α -Bisabolol against *B. argentifolii*

| treatment | concn (g/L) | av (%) of dead insects after 72 h | av (%) of layed eggs after 72h |
|------------------------------------|-------------|-----------------------------------|--------------------------------|
| essential oil | 2.00 | 100.0 | 0.0 |
| | 1.00 | 97.5 | 0.0 |
| | 0.50 | 90.0 | 7.6 |
| | 0.25 | 65.0 | 31.1 |
| | 0.10 | 52.5 | 36.8 |
| Orthene 750BR | 0.50 | 47.5 | 28.5 |
| Witness (DMSO/H ₂ O 5%) | | 17.5 | 88.1 |

observed can be attributed to α -bisabolol as the main component. However, the synergistic action of minor components in the essential oil composition should be responsible for the nonlinear correlation of the results.

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