Antifungal and Insecticidal Activities of Essential Oils of Four *Mentha* Species

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Abstract

**Background:** *Mentha* species are commonly used in traditional medicine for their several pharmacological properties. *Mentha* species are also used as spice and are known for their bactericidal, antiviral and fungicidal properties.

**Objectives:** The main objective of this work was to evaluate the antifungal activity and fumigation toxicity of essential oils of *Mentha* spicata, *M. pulegium*, *M. piperita* and *M. rotundifulas* against fungi and *Bactrocera oleae* insect responsible for olive rot.

**Methods:** Essential oils of the four *Mentha* species were extracted by a Clevenger-type apparatus. Their antifungal activity was tested using radial growth technique, and their insecticidal activity was examined by fumigant test.

**Results:** Oxygenated monoterpenes were the main components of the four *Mentha* essential oils. All the essential oils presented antifungal activity against *Aspergillus flavus*, *A. niger*, *Alternaria* spp. and *Penicillium* spp. At the highest concentration (15 μL/mL air), essential oil of *M. pulegium* caused 100% mortality after 1.5 h of exposure. However, for *M. piperita* and *M. rotundifula* essential oils, 25 μL/mL air was required to have mortality of 100%.

**Conclusions:** The essential oils could act as antifungal agents and fumigants against *B. oleae*.

**Keywords:** Antifungal, Insecticidal Activities, *Mentha* Species

1. Background

In Algeria, olive oil production is a developing industry. Olives are infected with several soilborne fungal pathogens such as *Alternaria*, *Aspergillus* and *Penicillium* (1). *A. niger* causes many diseases called black mold on fruits and vegetables and produces potent mycotoxins called ochratoxins that can be harmful to human beings. On the other hand, *A. flavus*, *Alternaria* spp. and *Penicillium* spp. are the most dominant fungal species during postharvest storage condition (2). It is known that fungal strains that occur most frequently at mild and cold temperatures affect fruits. Furthermore, many olives are attacked mainly by *Bactrocera oleae* insect that is considered to be a serious threat to olive production in the Mediterranean region.

Chemical fungicides are widely used to control phytotoxic fungi; nevertheless, the use of these types of compounds represents a concern associated with the risk of exposure and environmental hazards; therefore, new alternatives are needed (3). The genus *Mentha* belongs to Lamiaceae family and includes 25 species of herbaceous perennials. Mints are distributed predominantly in the temperate regions of the world and have varied growth characteristics, and aromas. Many *Mentha* species are used in traditional folk medicine for its stimulant, carminative, antispasmodic, stomachic and diuretic proprieties (4).

Many mint species are grown for commercial purposes such as their use in food flavors, cosmetics and pharmaceuticals (4, 5). Numerous studies have been carried out on the fungicidal and insecticidal activities of mint species (6-12).

2. Objectives

The main objective of this study was to assess (I) the antifungal activity of four mint essential oils against several phytopathogens responsible for olive diseases, such as *A. flavus*, *A. niger*, *Alternaria* spp. and *Penicillium* spp. and (II) insecticidal activity of these four oils against *B. oleae* insect responsible for olive rot.
3. Methods

3.1. Plant Material
The plant materials of *M. spicata*, *M. pulegium*, *M. piperita* and *M. rotundifolia* were collected from Tlemcen region (Algeria) in July 2014 during full bloom stage.

Each mint specimen was identified by Professor Noury Benabadji of University of Tlemcen (Algeria) and deposited in the Herbarium of the University with voucher specimens (*M. spicata*: MSP-0714; *M. pulegium*: MPU-0715; *M. piperita*: MPI-716 and *M. rotundifolia*: MRO-0716).

3.2. Essential Oils Isolation
The aerial parts were stored at 18°C after harvest, and 550-600 g of each species was subjected to a Clevenger-type apparatus (13) for 5 h. The yields of the oils were 0.5% for *M. spicata*, 0.7% for *M. pulegium*, 0.67% for *M. piperita*, and 0.9% for *M. rotundifolia*. Before chromatographic analysis, the essential oils were dried over sodium sulfate and stored in sterilized amber glass flasks.

3.3. Gas Chromatography

The gas chromatography (GC) apparatus used for the determination of retention indices was a Perkin Elmer Clarus 600 GC equipped with two flame ionization detectors (FIDs) and two fused-silica capillary columns (60 m × 0.22 mm, film thickness 0.25 μm) with different stationary phases: Rtx-1 (polydimethylsiloxane) and Rtx-Wax (polyethylene glycol). Program conditions were temperature of 60 to 230°C at 2°C min⁻¹ and then held isothermal at 230°C (30 min); the carrier gas was hydrogen (0.7 mL min⁻¹). Injector and detector temperatures were held at 280°C. Injected volume was 0.1 μL.

3.4. Gas Chromatography-Mass Spectrometry

The essential oils were investigated using a Perkin Elmer TurboMass quadrupole apparatus, directly coupled with a Perkin Elmer Autosystem XL equipped with two fused-silica capillary columns (60 m × 0.22 mm, film thickness 0.25 μm), Rtx-1 (polydimethylsiloxane) and Rtx-Wax (polyethylene glycol), with the same program as GC described above. Ion source temperature was 150°C and energy ionization was 70 eV; electron ionization mass spectra were acquired with a mass range of 35 - 350 Da and scan mass of 1 s. The injected oil volume was 0.1 μL.

3.5. Component Identification

The different components of essential oils were identified by comparison of GC retention indices (RI), determined from retention time of a series of n alkanes with linear interpolation, with those of authentic compounds (14, 15). For this purpose, computer matching with commercial mass spectral libraries and comparison of the spectra with those of the in-house laboratory library were performed (16).

3.6. Pathogenic Fungi

Aspergillus flavus, A. niger, Alternaria spp. and Penicillium spp., the four fungal isolates causing olive rot, were isolated directly from rotten olive harvested from orchards of Remchi, Ain Temouchent (Algeria). The four fungal species were transferred to sterilized Petri dishes, and 20% of lactic acid was added to the middle to stop the growth of bacteria. The plates were incubated at 25 ± 2°C for eight days away from light. Strains identification was firstly based on morphological characters and secondly on microscopic observations according the following references (17, 18).

3.7. In Vitro Antifungal Activity

The radial growth technique was used for testing the antifungal activity of essential oils (18). The concentrations varying from 0.1 to 300 mL/L used in the in vitro tests were obtained from stock solutions. For this purpose, appropriate volumes of essential oils were dissolved in dimethyl sulfoxide (DMSO) and added to Potato Dextrose Agar (PDA) medium immediately before it was poured into the Petri dishes of 9.0 cm diameter at 40°C - 45°C. The controls were prepared with DMSO mixed with PDA (without essential oils). The mycelial discs were filled with plant pathogenic fungi taken from 7-day-old cultures on PDA plates, and then they were transferred aseptically to the center of Petri dishes and incubated. This process was performed in triplicate.

The treatments were incubated at 27°C in the dark. Colony growth diameter was measured after the fungal growth in the control treatments had completely covered the Petri dishes. The half maximal inhibitory concentration (IC₅₀) and the minimum inhibitory concentration (MIC) were determined at 95% confidence intervals (19) using Probit analysis.

3.8. Fumigation Toxicity of Essential Oils Against Bactrocera oleae

To determine the fumigant toxicity of essential oils, appropriate concentrations were applied separately on filter papers (Whatman No. 1, 2 cm diameter) to achieve the concentrations of 8 to 65 mL/L air without using any solvent, and the filter papers were attached to the under surface of plastic jar lids at 50-ml volumes. The control sets received no oil. The lids were screwed tightly on the jars containing 15 insects each, all of the same age. These were kept at a temperature of 25 - 26°C and in 80% - 85% relative humidity (19). Mortality was checked 24 h after commencement of exposure. The mortality of insects was expressed in % and calculated by using the Abbott correction formula:

\[
\text{Corrected mortality} = \left(\frac{\text{OMT} - \text{OMC}}{100 - \text{CM}}\right) \times 100
\]

OMT, observed mortality in treatment; OMC, observed mortality in control; CM, control mortality.

Percentage mortality = (NDI/NII) × 100
of 0.2 and 1.2 mL/L, respectively. However, essential oil exhibited moderate activity against A. flavus and 0.8 mL/L and MICs of 0.1 and 0.08 mL/L, respectively. However, essential oil of A. flavus against M. spicata spp. and Penicillium spp. showed that essential oils of the four Mentha species have good antifungal activity against A. flavus, A. niger, Alternaria spp. and Penicillium spp. Furthermore, in review of the fumigant toxicity results of essential oils of the four mints, it can be noticed that oils show very interesting activities. Essential oils are complex volatile mixtures. Monoterpenes and sesquiterpenes are usually the main groups of compounds that are responsible for many of their biological activities. On the basis of these results, we suggest that antifungal activity and fumigant toxicity of Mentha essential oils was due to their major components such as linalool, carvone, pulegone, menthone and linalyl acetate with percentages exceeding 28%.

Carvone is abundantly found in cumin, dill and spearmint. It is a natural product with strong antiseptic properties used as a mosquito repellent (20). It has been demonstrated that carvone has strong fungicidal activity against different mycotoxigenic fungi involved in several plant diseases (20). Naigre et al. (21) and Flaminì et al. (22) also found that pulegone, limonene, carvone and menthone showed biocidal activity. We found that M. pulegium essential oil is rich in pulegone and M. spicata is rich in carvone and that they have significant insect antifeedant (M. pulegium) and nematocidal (M. spicata) effects (11).
We demonstrated in this study that the essential oils could act as antifungal agents and fumigants against *Bactrocera oleae*. Thus, due to their antifungal and insecticidal effects, these essential oils could be used as in fungicides and insecticides to prevent the infestation of olive trees. However, further trials are necessary to devise a method for the application of essential oils in fungicides against *Bactrocera oleae*.

Footnotes

Authors’ Contribution: Kenza Mejdoub: Identification of antifungal and insecticidal activities; Fatima Zohra Benomari: Analysis of essential oils and harvesting plants; Mohammed El Amine Dib: Writing the manuscript and identifying the chemical composition of the four mint species; Nassim Djabou: Analysis of essential oils and harvesting plants; Nassira Gnaouar Benyelles: Harvesting and identifying plants; Jean Costa: Director of laboratory of CPN; Alain Muselli: Identification of the chemical composition of the four mint species and correction of the manuscript.

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**Table 2.** Minimum (MIC) and Medium (IC50) Inhibitory Concentration Values Against Radial Growth of Fungal Species Determined After Seven Days of Incubation on PDA + Tween Amended with the Essential Oils

| Treatment (mL/L) | *A. flavus* | *A. niger* | Alternaria Spp. | Penicillium Spp. |
|-----------------|-------------|-------------|----------------|-----------------|
|                 | CMI IC50    | CMI IC50    | CMI IC50       | CMI IC50        |
| *M. spicata*    | 0.2±        | 45±         | 1.2±           | 50±             |
|                 | 0.1±        | 4.2±        | 0.2±           | 1.1±            |
| *M. pulegium*   | 1.5±        | 0.08±       | 1.3±           | 0.08±           |
| *M. rotundifolia* | 1.3±      | 90±         | 12.5±          | 25±             |

| Treatment (%) | A. flavus | A. niger | Alternaria Spp. | Penicillium Spp. |
|---------------|-----------|----------|----------------|-----------------|
|               | CMI IC50  | CMI IC50 | CMI IC50       | CMI IC50        |
| *M. spicata*  | 0.2±      | 45±      | 1.2±           | 50±             |
| *M. pulegium* | 0.1±      | 4.2±     | 0.2±           | 1.1±            |
| *M. rotundifolia* | 1.3±      | 90±      | 12.5±          | 25±             |

4 Jundishapur J Nat Pharm Prod. 2019; 14(1):e64165.

**Table 3.** Larvicidal Efficacy of *Mentha* Species Essential Oils Against *Bactrocera oleae*

| Concentrations (µL/mL air) | % Mortality ± SE | *M. spicata* | *M. pulegium* | *M. piperita* | *M. rotundifolia* |
|----------------------------|------------------|--------------|---------------|---------------|-------------------|
| 8                          | -                | 16.6±±1.2    | 20.2±±1.6     | 0.0±±0.0      |
| 10                         | 0.0±±0.0        | 50.0±±2.1    | 66.6±±3.2     | 46.6±±3.2     |
| 15                         | 40.3±±4.2       | 100.0±±0.0   | 86.5±±4.2     | 76.6±±5.6     |
| 25                         | 53.3±±5.3       | -            | 100.0±±0.0    | 100.0±±0.0    |
| 45                         | 76.6±±3.5       | -            | -             | -              |
| 65                         | 86.6±±6.6       | -            | -             | -              |
| LC50 (µL/mL air)           | 0.22             | 0.27         | -             | -              |
| LC90 (µL/mL air)           | 0.33             | 0.45         | -             | -              |

4 The results are expressed as mean ± standard deviation.
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Table 1. Mentha Species Essential Oils

| Compounds                  | M. spicata | M. pulegium | M. piperita | M. rotundifolia |
|----------------------------|------------|-------------|-------------|-----------------|
| 1. (E)-hex-3-en-1-ol       | 812        | 810         | 1360        | tr              |
| 2. Ethyl-2-methyl butyrate  | 829        | 829         | 1016        | 0.1             |
| 3. (E)-2-hexenal           | 830        | 810         | 1210        | tr              |
| 4. (Z)-hex-3-en-1-ol       | 831        | 832         | 1175        | 0.1             |
| 5. (Z)-2-hexenol           | 851        | 848         | 1400        | tr              |
| 6. 1-hexenol               | 852        | 851         | 1444        | tr              |
| 7. α-thujene               | 922        | 923         | 1021        | 0.4             |
| 8. α-pinene                | 931        | 932         | 1023        | 0.5             |
| 9. Camphene                | 943        | 944         | 1066        | tr              |
| 10. Oct-1-en-3-ol          | 959        | 962         | 1440        | 0.8             |
| 11. Sabineene              | 964        | 966         | 1118        | 0.5             |
| 12. β-pinene               | 970        | 972         | 1008        | 0.7             |
| 13. Myrcene                | 976        | 982         | 1159        | 3.3             |
| 14. 3-octanol              | 982        | 982         | 1350        | 0.8             |
| 15. γ-phellandrene         | 997        | 998         | 1164        | 0.1             |
| 16. α-terpinene            | 1008       | 1010        | 1175        | 0.3             |
| 17. P-cymene               | 1010       | 1012        | 1259        | 1.0             |
| 18. Limonenene             | 1020       | 1021        | 1195        | 21.9            |
| 19. L-cineole              | 1020       | 1021        | 1205        | 0.6             |
| 20. (Z)-3-octimene         | 1024       | 1025        | 1225        | 0.4             |
| 21. (E)-4-octimene         | 1034       | 1036        | 1241        | 0.4             |
| 22. γ-terpinene            | 1047       | 1049        | 1237        | 0.7             |
| 23. Trans-hydrate sabinene | 1051       | 1054        | 1444        | 1.7             |
| 24. Terpinolene            | 1078       | 1080        | 1247        | 0.1             |
| 25. Linalool               | 1078       | 1075        | 1280        | 0.2             |
| 26. cis-sabineene hydrate  | 1083       | 1082        | 1335        | 0.5             |
| 27. 1-oct-3-enyl acetate   | 1093       | 1087        | 1390        | tr              |
| 28. 2-methyl butyl isovalerate | 1098      | 1096        | 1274        | 0.4             |
| 29. cis-P-menth-2-en-1-ol  | 1108       | 1110        | 1600        | tr              |
| 30. 3-octyl acetate        | 1110       | 1110        | 1315        | 0.2             |
| 31. trans-P-menth-2-en-1-ol| 1123       | 1126        | 1612        | tr              |
| 32. Menthone                | 1134       | 1135        | 1456        | 10.8            |
| 33. P-menth-3-en-8-ol       | 1135       | 1135        | 1590        | 28.5            |
| 34. Iso menthone            | 1143       | 1142        | 1490        | 0.7             |
| 35. Bornone                 | 1148       | 1150        | 1690        | 19.0            |
| 36. Neo-menthol             | 1156       | 1157        | 1637        | 0.2             |
| 37. Terpineine-4-ol         | 1161       | 1162        | 1581        | 1.6             |
| 38. Menthol                 | 1164       | 1163        | 1629        | tr              |
| 39. Iso menthol             | 1174       | 1173        | 1660        | tr              |
| 40. Z-dihydro carvone       | 1175       | 1174        | 1601        | 2.6             |
| 41. Dihydro carvone         | 1178       | 1174        | 1723        | tr              |
| 42. α-terpineole            | 1179       | 1177        | 1688        | tr              |
| 43. L-dihydro carvone       | 1180       | 1180        | 1626        | 3.1             |
| 44. α-campholenol           | 1186       | 1188        | 1782        | tr              |
| 45. Nerol                   | 1211       | 1213        | 1799        | 1.1             |
| 46. Pulegone                | 1213       | 1216        | 1640        | 77.3            |
| 47. Carvone                 | 1222       | 1226        | 1739        | 54.1            |
| 48. Piperitone              | 1232       | 1229        | 1727        | 0.3             |
| 49. Geraniol                | 1232       | 1234        | 1844        | 2.4             |
| 50. Linalyl acetate         | 1240       | 1237        | 1557        | tr              |
| 51. Menthol                 | 1242       | 1243        | 1744        | 3.3             |

Jundishapur J Nat Pharm Prod. 2019;14(1):e64165.
|   |   |   |   |   |
|---|---|---|---|---|
| 51. Geranial | 1244 | 1243 | 1731 | 0.2 |
| 52. Neryl formate | 1263 | 1266 | 1647 | 0.1 |
| 53. Neo-menthyl acetate | 1263 | 1268 | 1548 | 0.1 |
| 54. Bornyl acetate | 1269 | 1268 | 1475 | tr |
| 55. Lavandulyl acetate | 1270 | 1273 | 1593 | 0.1 |
| 56. Menthyl acetate | 1282 | 1285 | 1578 | 2.1 |
| 57. Iso-menthyl acetate | 1294 | 1295 | 1594 | 0.1 |
| 58. Dihydro carvyl acetate | 1311 | 1312 | 1661 | 2.2 |
| 59. Piperitenone | 1315 | 1313 | 1900 | tr |
| 60. Piperitenone oxide | 1333 | 1335 | 1945 | 0.3 |
| 61. α-terpenyl acetate | 1336 | 1336 | 1678 | 0.1 |
| 62. Neryl acetate | 1342 | 1345 | 1725 | 1.7 |
| 63. Geranyl acetate | 1361 | 1364 | 1725 | 2.5 |
| 64. α-copaene | 1379 | 1379 | 1475 | 0.1 |
| 65. β-bourbonene | 1385 | 1385 | 1535 | 0.3 |
| 66. Eβ-caryophyllene | 1424 | 1418 | 1583 | 0.6 |
| 67. Eβ-farnesene | 1448 | 1447 | 1660 | 0.1 |
| 68. α-humulene | 1456 | 1456 | 1665 | 0.2 |
| 69. γ-muurolene | 1471 | 1469 | 1679 | 0.1 |
| 70. Germacrene D | 1480 | 1474 | 1692 | 0.1 |
| 71. α-muurolene | 1496 | 1492 | 1709 | 0.1 |
| 72. γ-cadinene | 1507 | 1506 | 1750 | 0.1 |
| 73. Trans-calamenene | 1512 | 1510 | 1810 | 0.1 |
| 74. δ-cadinene | 1516 | 1515 | 1748 | 0.1 |
| 75. Cadina1,4-diene | 1523 | 1520 | 1763 | 0.1 |
| 76. α-calamorene | 1531 | 1528 | 1890 | 0.1 |
| 77. α-cadinene | 1535 | 1530 | 1740 | tr |
| 78. β-calamorene | 1548 | 1546 | 1936 | tr |
| 79. Caryophyllene oxide | 1578 | 1580 | 1980 | 0.3 |
| 80. Globulol | 1580 | 1582 | 2074 | 0.5 |
| Total identification % | 98.1 | 98.5 | 98.8 | 98.9 |

- **Hydrocarbon compounds**: 2.7, 4.8, 6.5
- **Monoterpenes hydrocarbons**: 2.0, 2.8, 4.9
- **Sesquiterpene hydrocarbons**: 0.7, 2.0, 1.6

- **Oxygenated compounds**: 95.8, 94.0, 92.4
- **Oxygenated monoterpenes**: 94.2, 92.5, 91.3
- **Oxygenated sesquiterpenes**: - , 0.8, -
- **Non-terpenic oxygenated compounds**: 1.6, 0.7, 1.1