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# Chemical and Olfactory Analysis of the Volatile Fraction of *Ocimum gratissimum* Concrete from Madagascar

Laure Gilles<sup>[a, b]</sup> and Sylvain Antoniotti<sup>\*[a]</sup>

The chemical composition of the volatile fraction of *Ocimum gratissimum* concrete (romba) from Madagascar has been determined for the first time by GC/MS and GC-FID. A methyl cinnamate chemotype has been determined for this material, along with a set of compounds typical in essential oils and extracts from plants of the *Ocimum* genus. Variability was mostly observed on terpenes and terpenoids components. GC-O-MS was also used for a sensory evaluation of this material

performed by a master perfumer. The chemical composition of this *O. gratissimum* extract was then compared with literature data to assess subtle differences between chemotypes of the same species and other species of the same genus within natural variability. A mapping illustrates the occurrence of the cinnamate chemotype in Eastern Africa, India and now Madagascar, while other origins generally present eugenol, thymol, camphor, or linalool chemotypes.

## Introduction

*Ocimum gratissimum* or clove basil, is a plant of the Lamiaceae family (Table 1, Figure 1) which is commonly found in Africa, Asia and South America. The use of its leaves as an infusion has been reported in folk medicine to treat fever, flu and kidney problems.<sup>[1]</sup>

Many studies have been conducted on *Ocimum gratissimum*, including the total or partial determination of the composition of its essential oil (62 publications), but almost all of these articles focused on the bioactive properties of the plant.<sup>[1–9]</sup> Indeed it showed antibacterial,<sup>[10]</sup> antimicrobial,<sup>[1]</sup> acaricides,<sup>[3]</sup> properties, often associated with the eugenol and the thymol chemotypes.<sup>[11–13]</sup>

The established chemical compositions show that nine different chemotypes have been identified for *Ocimum gratissimum* (Table 2), although eugenol and thymol are the most common (Entries 8 and 9).<sup>[14]</sup> The chemotype is assigned based on the chemical composition of which a predominant chemical entity can be identified, and can vary within the same species according to their ecosystem. It depends essentially on the growing region of the plant and the climate (altitude, humidity, biotope, sun exposure, temperature, rain) since the influence of the ecosystem can cause chemical variations in the secondary metabolites of the plant.

**Table 1.** Classification of *Ocimum gratissimum*.

| Plant Kingdom |                    |
|---------------|--------------------|
| Class         | Equisetopsida      |
| Sub-class     | Magnoliidae        |
| Super order   | Asteranae          |
| Order         | Lamiales           |
| Family        | Lamiaceae          |
| Genus         | <i>Ocimum</i>      |
| Species       | <i>gratissimum</i> |



**Figure 1.** *Ocimum gratissimum* (picture: Rison Thumboor from Thrissur, India).

**Table 2.** Chemotypes for *Ocimum gratissimum*.<sup>[14]</sup>

| Entry | Chemotypes                         |
|-------|------------------------------------|
| 1     | Linalool/estragole                 |
| 2     | Eugenol/1,8-cineole/sesquiterpenes |
| 3     | Methyl cinnamate                   |
| 4     | Methyleugenol/eugenol              |
| 5     | Ethyl cinnamate                    |
| 6     | Citral                             |
| 7     | Geraniol                           |
| 8     | <b>Eugenol</b>                     |
| 9     | <b>Thymol</b>                      |

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Regarding the olfactory evaluation of the essential oil, out of a hundred publications reporting studies of the plant, only two have been interested in describing olfactory properties. The former dealt with the overall olfactory evaluation of the essential oil to confirm the chemotype found by studying the composition by GC/MS and GC-FID.<sup>[15]</sup> The latter carried out GC-O experiments on the essential oils of 4 species of the *Ocimum* genus including *Ocimum gratissimum*.<sup>[16]</sup> This article, written in Mandarin, reported a work carried out for the determination of the composition of the essential oil by GC/MS, and the use of GC-O analysis in order to confirm the olfactory profile of the compounds identified in GC/MS, albeit in the absence of calculations of the Kovats retention indices (RI), essential for a correct characterization in addition to the comparison of mass spectra and coinjection of authentic samples in case of doubt.

In addition, the diversity of the types of extracts on which *Ocimum gratissimum* studies have been performed is quite low. Almost all of them focused on essential oil, except for two articles on the analysis of a solvent extract. The first focused on a freeze-dried ethanolic extract, somehow devoid of volatile molecule.<sup>[17]</sup> In the second, a methanol extraction was carried out, then, after evaporation, the residue was dissolved in distilled water and liquid-liquid extractions with hexane, diethyl ether and ethyl acetate were carried out.<sup>[18]</sup> The fractions obtained were purified on preparative HPLC to perform biological testing. Finally, methyl cinnamate was isolated and found to be responsible for insecticidal and larvicidal activities.

Herein, we wish to describe the work carried out in the frame of global project involving a Master perfumer to identify promising odorant molecules in natural materials besides odor-impact molecules specific of the mixture. In particular, we present our results on a romba (*Ocimum gratissimum*) concrete (solvent extract) with GC-FID, GC/MS and GC-O analysis performed on the concrete and the volatile fraction of the concrete obtained after distillation at low pressure.

## Discussion

### Methodology

For several sample, the superposition of the FID chromatogram with the olfactogram composed of the olfactory impressions of a professional perfumer was generated. Several parameters made it possible to drive us towards the concrete of Romba (*Ocimum gratissimum*) in order to carry out a more in-depth analysis of the data obtained. The factors taken into account were the complexity of the chromatographic profile but also the interest of the perfumer for the scents felt, and on their possible response to a need of the perfumery industry.

### Distillation of romba concrete (*Ocimum gratissimum*)

The romba sample used in this study was a concrete (supplied by Bionexx), consisting in a raw solvent extract made from the

leaves and stems of *Ocimum gratissimum* plants from Madagascar.

In order to focus only on the volatile part, the concrete was distilled in a Kugelrohr-type distillation oven under reduced pressure, down to 1 mbar (Figure 2).

A quantity of 53.4 g of concrete was submitted to the distillation process and 23.3 g of distillate was obtained (44% yield).

The GC/MS chromatogram of the distillate showed the same profile as the chromatogram of the concrete, which confirmed that all volatile compounds have been preserved and that distillation has not altered their relative proportions nor generated new compounds.

### Distillate composition

The chemical composition of the distillate was established following standard procedures by combining two methods:

Comparison of the mass spectrum of each peak of the GC/MS chromatogram with those of the commercial library databases (NIST15, Wiley) and those of the database established by Adams.<sup>[19]</sup>

The comparison of the Kovats retention indices (RI) determined on our mixture with those of this database.

The main components of this distillate were found to be camphor, estragole, methyl (*Z*)-cinnamate, eugenol, methyl (*E*)-cinnamate (major compound) and *trans*- $\alpha$ -bergamotene, indicating a cinnamate chemotype (Figure 3).

A total of 56 volatile compounds constituting 99.23% of the distillate of the romba concrete were identified (Table 3), their relative proportion was determined by GC-FID. Interestingly, metabolites of different biosynthetic origin were found with terpenes and terpenoids (including phenolics and sesquiterpenes), phenylpropanoids, and fatty acid derivatives.

Thanks to the identification of most of the compounds, the correlation between the olfactory notes felt by the perfumer and the associated odorous molecule could be established (Table 4). In addition, the organoleptic properties of these molecules have been confirmed by comparison with informa-

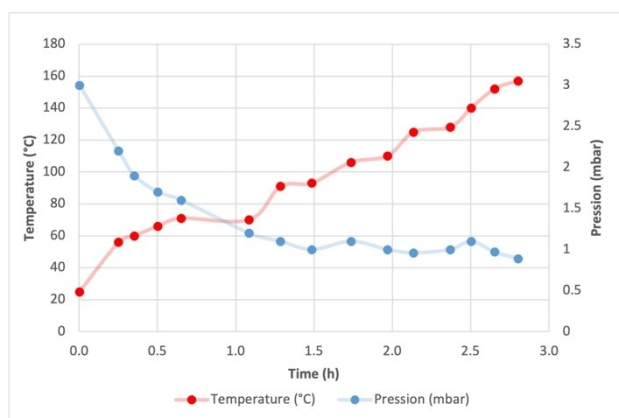


Figure 2. Monitoring of temperature and pressure during the distillation of romba concrete (*Ocimum gratissimum*).

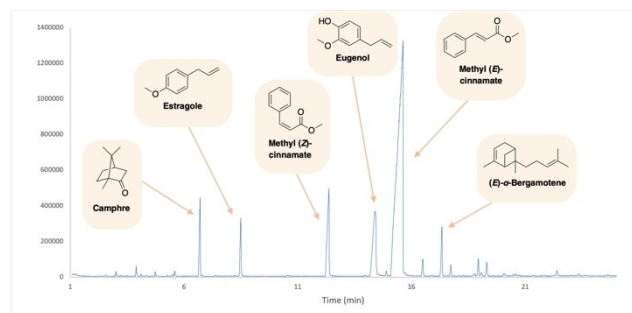


Figure 3. Main components of the distillate of the romba concrete.

tion found in the database of odorous ingredients "The Good Scents Company"<sup>[22]</sup> which lists a very large number of ingredients in the field of aromas and perfumes and their olfactory and / or aromatic description.

In addition, GC-O-MS analyses made it possible to focus in more detail on the areas of the chromatogram identified as being of olfactory interest to the perfumer. Several samples of the distillate at different concentrations (1, 5 and 10% in MTBE) were analyzed in order to assess both the largest and smallest peaks, generate their mass spectrum and confirm the assignments listed in Table 4.

The compositions of romba essential oils described in a selection of scientific articles representing the 14 most recent publications have been grouped in Table 5 in order to compare them with the composition of the volatile fraction of the romba extract that we established during this work.<sup>[1-5,10,12,16,23-28]</sup> It is worth noting that some essential oils were obtained from leaves, other from leaves and stems, or other from leaves and flowers. This adds to the natural variability. Compounds are classified by increasing retention index (identification by EI-MS spectra comparison and RI determination) and their percentage abundance is reported (quantification by GC-FID).

The essential oils studied in these publications are mostly obtained from a eugenol chemotype (10 out of 14), some of which are composed of more than 70% of this compound. Thymol and linalool chemotypes were also found. The extract we have studied has features of a methyl (*E*)-cinnamate chemotype. Apart from a few compounds present in almost each of the compositions analyzed, such as 1,8-cineole, (*E*)- $\beta$ -ocimene, eugenol or caryophyllene, the compositions are relatively varying even within the same chemotype.

Principal Component Analysis (PCA) was performed and confirmed the relatively distributed variance, component 1 accounting for only 28.9% and component 2 for 14.3% (Figure 4). Individual contribution of components 1 and 2 were significantly brought by our sample from Madagascar with 62%, followed by a Brazilian sample with 25% and a Vietnamese sample with less than 3%. Compounds contributing strongly to the variance were found to be allo-ocimene,  $\alpha$ -terpineol, *trans*-bergamotene, (*Z*)- $\beta$ -farnesene,  $\beta$ -copaene,  $\delta$ -2-carene and *p*-cymene, all belonging to the terpenes and terpenoids family.

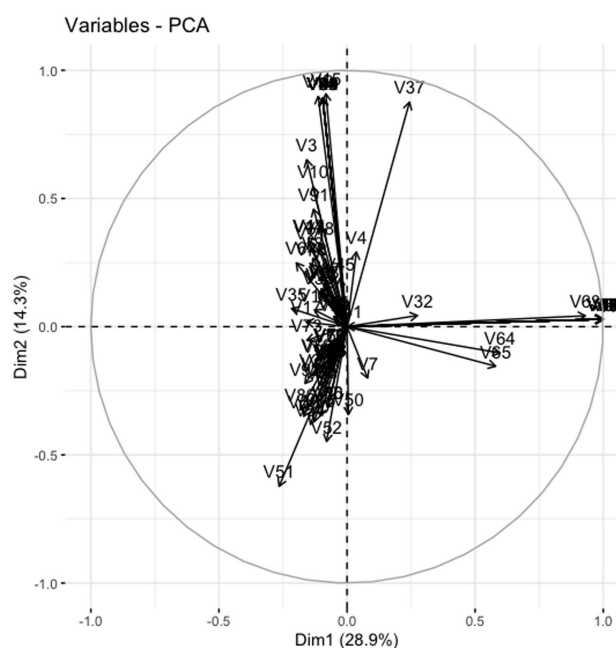


Figure 4. PCA plot of the chemical composition (96 variables) for 14 essential oils.

This diversity of composition can be explained by several factors. Indeed, the composition of an essential oil fluctuates a lot depending on the parts of the plant (leaves, flowers, stem, etc.) used, and can also present a significant natural variability depending on the practices of cultivation and harvesting, the type of soil, the biotope, the climate, the season and the time of the day of harvest.<sup>[29-32]</sup> In addition, the type of extraction method can lead to different compositions from the same raw material.

Our sample shows some similarities with essential oils of *Ocimum americanum* which presents also a cinnamate chemotype. This prompted us to examine our data in more details in comparison with the chemical composition of essential oils from aerial parts of plants of the *Ocimum* genus described in the literature.

#### Possible similarities with *Ocimum americanum* and *Ocimum canum*

Issues of authenticity, conformity and adulteration are not negligible in essential oils and natural extracts.<sup>[33-34]</sup> Having in hand a solvent extract and not a botanical specimen, its authenticity as an *Ocimum gratissimum* vs. *Ocimum americanum* or *Ocimum canum* for example was a fair question. A cinnamate chemotype was indeed reported for *O. americanum* from India on the basis of the chemical profile of the essential oil obtained from the whole plant (over 81% for methyl (*E*)- and (*Z*)-cinnamate).<sup>[15]</sup> However, beside supposedly from the same chemotype, out of 119 compounds, our sample have only 41 in common with the sample from this study. They differ indeed mostly in the terpene and terpenoids composition.

**Table 3.** Chemical composition of romba distillate established by MS spectrum and RI comparison (identity) and GC-FID (quantity).

| Entry | RI exp. | [RI litt. <sup>[19–21]</sup> | Compound                               | % (GC-FID)     | Chemical class            |
|-------|---------|------------------------------|--|----------------|---------------------------|
| 1     | 922     | 921                          | Tricyclene                             | 0.02           | Monoterpene hydrocarbon   |
| 2     | 928     | 932                          | $\alpha$ -Pinene                       | 0.03           | Monoterpene hydrocarbon   |
| 3     | 941     | 946                          | Camphene                               | 0.07           | Monoterpene hydrocarbon   |
| 4     | 950     | 952                          | Benzaldehyde                           | tr.            | Phenylpropanoid           |
| 5     | 965     | 969                          | Sabinene                               | 0.02           | Monoterpene hydrocarbon   |
| 6     | 970     | 974                          | 1-Octen-3-ol                           | 0.2            | Aliphatic alcohol         |
| 7     | 978     | 979                          | 3-Octanone                             | tr.            | Aliphatic ketone          |
| 8     | 984     | 988                          | Myrcene                                | 0.08           | Monoterpene hydrocarbon   |
| 11    | 999     | 1002                         | $\alpha$ -Phellandrene                 | tr.            | Monoterpene hydrocarbon   |
| 12    | 1003    | 1004                         | (3Z)-hexenyl acetate                   | 0.03           | Aliphatic ester           |
| 13    | 1019    | 1022                         | <i>p</i> -Cymene                       | 0.01           | Monoterpene hydrocarbon   |
| 14    | 1023    | 1024                         | Limonene                               | 0.38           | Monoterpene hydrocarbon   |
| 15    | 1024    | 1026                         | 1,8-Cineole                            | 0.09           | Oxygenated monoterpene    |
| 16    | 1028    | 1026                         | Benzyl alcohol                         | tr.            | Phenylpropanoid           |
| 17    | 1032    | 1032                         | (Z)- $\beta$ -Ocimene                  | 0.09           | Monoterpene hydrocarbon   |
| 18    | 1041    | 1044                         | (E)- $\beta$ -Ocimene                  | 0.03           | Monoterpene hydrocarbon   |
| 19    | 1050    | 1054                         | $\gamma$ -Terpinene                    | 0.01           | Monoterpene hydrocarbon   |
| 20    | 1077    | 1083                         | Fenchone                               | tr.            | Oxygenated monoterpene    |
| 21    | 1079    | 1085                         | <i>p</i> -Mentha-2,4-diene             | 0.03           | Monoterpene hydrocarbon   |
| 22    | 1086    | 1088                         | Methyl benzoate                        | tr.            | Phenylpropanoid           |
| 23    | 1090    | 1086                         | Terpinolene                            | tr.            | Monoterpene hydrocarbon   |
| 24    | 1094    | 1095                         | Linalool                               | 0.30           | Oxygenated monoterpene    |
| 25    | 1107    | 1107                         | Phenyl ethyl alcohol                   | 0.01           | Phenylpropanoid           |
| 26    | 1124    | 1128                         | <i>allo</i> -Ocimene                   | tr.            | Monoterpene hydrocarbon   |
| 27    | 1134    | 1141                         | Camphor                                | 4.04           | Oxygenated monoterpene    |
| 28    | 1161    | 1155                         | Isoborneol                             | 0.02           | Oxygenated monoterpene    |
| 29    | 1192    | 1195                         | Estragole                              | 2.73           | Phenylpropanoid           |
| 30    | 1242    | 1247                         | <i>p</i> -Anisaldehyde                 | 0.05           | Phenylpropanoid           |
| 31    | 1247    | 1254                         | 2-Phenylethyl acetate                  | 0.03           | Phenylpropanoid           |
| 32    | 1295    | 1299                         | Methyl (Z)-cinnamate                   | 7.37           | Phenylpropanoid           |
| 33    | 1338    | 1348                         | $\alpha$ -Cubebene                     | 0.03           | Sesquiterpene hydrocarbon |
| 34    | 1350    | 1356                         | Eugenol                                | 13.90          | Phenylpropanoid           |
| 35    | 1362    | 1374                         | $\alpha$ -Copaene                      | 0.32           | Sesquiterpene hydrocarbon |
| 36    | 1378    | 1376                         | Methyl (E)-cinnamate                   | 62.39          | Phenylpropanoid           |
| 37    | 1398    | 1393                         | Vanilline                              | 0.41           | Phenylpropanoid           |
| 38    | 1404    | 1403                         | Methyleugenol                          | 0.14           | Phenylpropanoid           |
| 39    | 1408    | 1408                         | (E)-b-Caryophyllene                    | 1.09           | Sesquiterpene hydrocarbon |
| 40    | 1417    | 1419                         | $\beta$ -Cedrene                       | tr.            | Sesquiterpene hydrocarbon |
| 41    | 1426    | 1432                         | $\alpha$ - <i>trans</i> -Bergamotene   | 2.65           | Sesquiterpene hydrocarbon |
| 42    | 1433    | 1440                         | (Z)- $\beta$ -Farnesene                | 0.08           | Sesquiterpene hydrocarbon |
| 43    | 1437    | 1452                         | $\alpha$ -Humulene                     | 0.59           | Sesquiterpene hydrocarbon |
| 44    | 1451    | 1454                         | (E)- $\beta$ -Farnesene                | 0.02           | Sesquiterpene hydrocarbon |
| 45    | 1464    | 1465                         | <i>cis</i> -Muurola-4(14),5-diene      | 0.16           | Sesquiterpene hydrocarbon |
| 46    | 1472    | 1457                         | Sesquisabinene                         | 0.79           | Sesquiterpene hydrocarbon |
| 47    | 1478    | 1476                         | $\beta$ -Chamigrene                    | 0.65           | Sesquiterpene hydrocarbon |
| 48    | 1487    | 1483                         | $\alpha$ -Amorphene                    | 0.01           | Sesquiterpene hydrocarbon |
| 49    | 1490    | 1493                         | $\alpha$ -Zingiberene                  | tr.            | Sesquiterpene hydrocarbon |
| 50    | 1498    | 1493                         | <i>trans</i> -Muurola-4(14),5diene     | 0.04           | Sesquiterpene hydrocarbon |
| 51    | 1509    | 1511                         | $\delta$ -Amorphene                    | 0.12           | Sesquiterpene hydrocarbon |
| 52    | 1512    | 1521                         | $\beta$ -Sesquiphellandrene            | 0.11           | Sesquiterpene hydrocarbon |
| 53    | 1532    | 1532                         | $\gamma$ -Cuprenene                    | 0.01           | Sesquiterpene hydrocarbon |
| 54    | 1538    | 1548                         | Allyl (E)-cinnamate                    | 0.01           | Phenylpropanoid           |
| 55    | 1574    | 1544                         | Methyl (Z)- <i>p</i> -methoxycinnamate | 0.05           | Phenylpropanoid           |
| 56    | 1653    | 1614                         | Methyl (E)- <i>p</i> -methoxycinnamate | 0.02           | Phenylpropanoid           |
|       |         |                              | Monoterpene hydrocarbon                | 0.77           |                           |
|       |         |                              | Oxygenated monoterpene                 | 4.45           |                           |
|       |         |                              | Sesquiterpene hydrocarbon              | 6.67           |                           |
|       |         |                              | Aromatic                               | 87.11          |                           |
|       |         |                              | Others                                 | 0.23           |                           |
|       |         |                              | <b>Total</b>                           | <b>99.23 %</b> |                           |

*O. americanum* essential oils from Burkina Faso were described to be rich in 1,8-cineole (45.0–60.2%), camphor and  $\alpha$ -terpineol.<sup>[35]</sup> With plant from Nigeria, the chemotype was mostly eugenol and farnesol,<sup>[36]</sup> while from Rwanda, some linalool-rich essential oils were obtained.<sup>[37]</sup> Camphor chemo-

types were described for Indian plant material.<sup>[38]</sup> Again in India, chemotypes estragole (methyl chavicol) and citral,<sup>[39]</sup> and citral alone (up to 76%) after natural selection, were described.<sup>[40]</sup>

The less studied *Ocimum canum* species were described as rich in camphor (60%) and limonene (13%) in essential oils

**Table 4.** Assignment of olfactory notes to molecules during the GC-O session by comparison with GC/MS results. Red: high interest, orange: moderate interest, green: low interest.

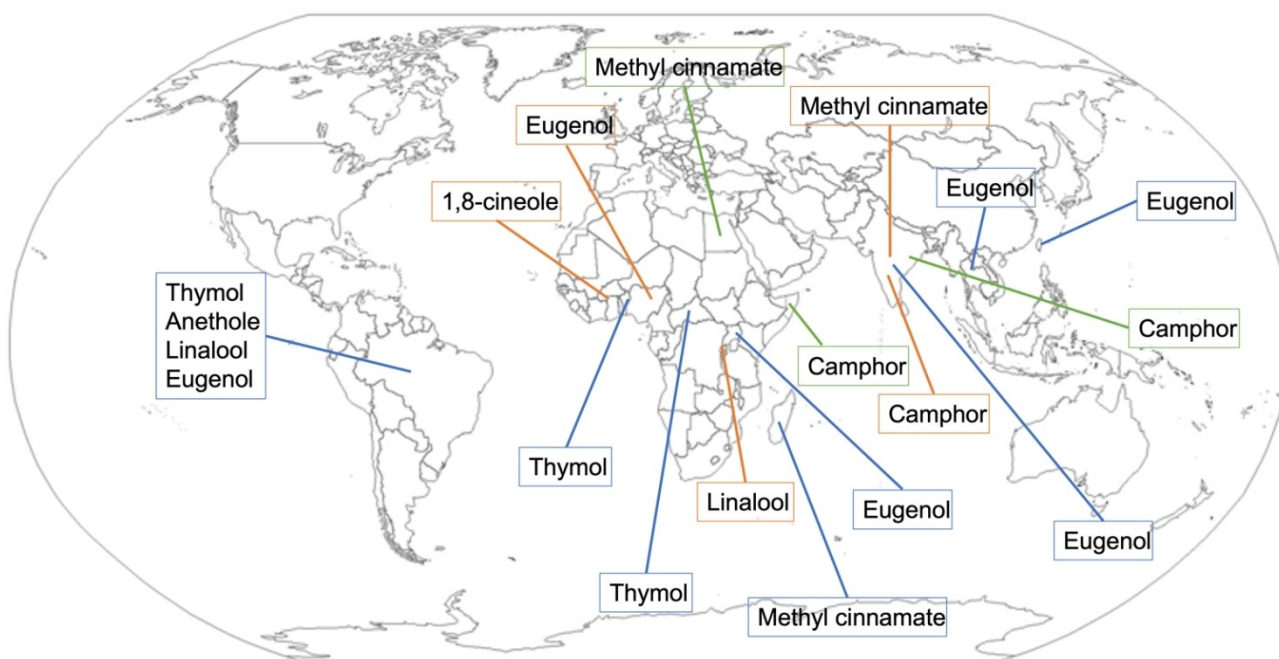
| Entry | RI   | Olfactory notes                                  | Molecule                         |
|-------|------|--|----------------------------------|
| 1     | 961  | Blackcurrant, passion fruit, fruity              | Not detected                     |
| 2     | 979  | Aldehyde, metallic, beautiful, pure, fresh, pear | 1-Octene-3-one                   |
| 3     | 1022 | Woody, copaiba                                   | <i>p</i> -Cymene                 |
| 4     | 1024 | Herbal, myrtle, rosemary, sage                   | <i>L</i> -limonene/1,8-cineole   |
| 5     | 1095 | Green, watery, floral, lotus                     | Linalool                         |
| 6     | 1141 | Aldehyde, fresh, floral rose                     | Camphor                          |
| 7     | 1195 | Anise, fennel                                    | Estragole                        |
| 9     | 1247 | Anise, almost jasmona                            | <i>p</i> -Anisaldehyde           |
| 10    | 1254 | Blackcurrant buds, very beautiful                | 2-Phenylethyl acetate            |
| 11    | 1299 | Woody, very beautiful, copaiba fat               | Methyl ( <i>Z</i> )-cinnamate    |
| 12    | 1348 | Light grass                                      | $\alpha$ -Cubebene               |
| 13    | 1356 | Eugenol  | Eugenol                          |
| 14    | 1376 | White floral fruity, pineapple, jasmine          | Methyl ( <i>E</i> )-cinnamate    |
| 15    | 1393 | Lili, vanillin, vanitrop                         | Vanilline                        |
| 16    | 1452 | Woody, spicy, isoeugenol,                        | $\alpha$ -Humulene               |
| 17    | 1454 | Fruity, tropical, passion fruit                  | ( <i>E</i> )- $\beta$ -Farnesene |
| 18    | 1521 | Woody, blond, cedar of Chile                     | $\beta$ -Sesquiphellandrene      |
| 19    | 1548 | Slightly spicy                                   | Allyl ( <i>E</i> )-cinnamate     |

from Somalia and India<sup>[41]</sup> and in methyl (*Z*)- and (*E*)-cinnamate in *O. canum* from Egypt.<sup>[42]</sup> Interestingly, the authors studied enzymatic pretreatment prior to hydrodistillation, an approach that was described with other plant materials to improve distillation yields or improve the quality post-distillation.<sup>[43–45]</sup> Although of the same chemotype, the chemical profile was different from that of our Malagasy sample.

The possibility of an error of authentication of our *Ocimum gratissimum* sample was thus ruled out based on these comparisons (a mapping of chemotypes is presented in Figure 5).

## Conclusions

As part of this work, the first chemical composition of the volatile fraction of a romba concrete from Madagascar was carried out by GC/MS and GC-FID. In addition, the olfactory evaluation by GC-O was carried out by the internationally renowned master perfumer Christophe Laudamiel, including the first on a sample of Romba of methyl (*E*)-cinnamate chemotype, but also the first on a solvent extract and not an essential oil. This work, supported by GC-O-MS analyses, has thus enabled the identification of important odoriferous compounds in the distillate of this concrete, obtained from leaves and stems of *Ocimum gratissimum* of Madagascar.


**Figure 5.** Mapping of chemotypes described from *Ocimum gratissimum* (blue), *Ocimum americanum* (orange), and *Ocimum canum* (green).

**Table 5.** Comparison of the chemical composition of the romba concrete distillate with literature data.

| rt    | RI   | RI litt. | Compound                             | Madagascar   | Vietnam <sup>23</sup> | Brasil <sup>1</sup> | Unknown <sup>16</sup> | Vietnam <sup>2</sup> | Brasil <sup>3</sup> | India <sup>4</sup> | Brasil <sup>12</sup> | Brasil <sup>24</sup> | India <sup>25</sup> | Brasil <sup>10</sup> | Taiwan <sup>5</sup> | Benin <sup>26</sup> | Brasil <sup>27</sup> | Guangdong <sup>28</sup> |
|-------|------|----------|--------------------------------------|--------------|-----------------------|---------------------|-----------------------|----------------------|---------------------|--------------------|----------------------|----------------------|---------------------|----------------------|---------------------|---------------------|----------------------|-------------------------|
|       |      |          |                                      | (this study) | wt%                   |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 2.3   | 922  | 921      | Tricyclene                           | 0.02         |                       |                     |                       |                      | 2.8                 |                    |                      | 0.47                 | 0.6                 |                      |                     |                     |                      |                         |
|       |      | 924      | Origanene                            |              | 0.43                  |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
|       |      | 939      | $\alpha$ -Pinene                     | 0.03         |                       | 0.08                | 0.04                  |                      | 0.7                 | 0.2                | 0.53                 | 3.47                 | 0.1                 |                      |                     | 5.45                |                      |                         |
| 2.57  | 941  | 945      | Camphene                             | 0.07         |                       |                     |                       |                      | 0.1                 | tr.                | 0.46                 | 0.09                 |                     |                      |                     | 0.12                |                      |                         |
| 2.69  | 950  | 952      | Benzaldehyde                         | tr.          |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
|       |      | 969      | Sabinene                             | 0.02         |                       | 0.17                | 0.45                  |                      | 0.4                 | tr.                | 0.6                  | 0.21                 | 0.5                 |                      |                     | 0.34                |                      |                         |
| 3.01  | 970  | 974      | 1-Octen-3-ol                         | 0.2          |                       |                     |                       |                      |                     |                    |                      |                      | 0.5                 |                      |                     |                     |                      | 1.1                     |
|       |      | 979      | $\beta$ -Pinene                      |              |                       | 0.43                | 0.02                  |                      |                     | 0.1                | 1.25                 | 0.63                 |                     |                      |                     | 4.76                |                      |                         |
| 3.12  | 978  | 979      | 3-Octanone                           | tr.          |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 3.21  | 984  | 988      | $\beta$ -Myrcene                     | 0.08         | 0.17                  | 0.14                | 0.3                   |                      | 1.9                 | tr.                | 0.83                 | 0.89                 |                     |                      |                     | 0.18                |                      |                         |
|       |      | 1001     | $\delta$ -2-Carene                   |              |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     | 3.89                |                      |                         |
| 3.43  | 999  | 1002     | $\alpha$ -Phellandrene               | tr.          |                       |                     |                       |                      | 0.4                 |                    |                      | 3.41                 | 0.4                 |                      |                     |                     |                      |                         |
|       |      | 1014     | $\alpha$ -Terpinene                  |              | 0.20                  |                     |                       |                      | 2.2                 |                    |                      |                      | 0.3                 |                      |                     |                     |                      |                         |
| 3.8   | 1019 | 1022     | <i>p</i> -Cymene                     | 0.01         | 0.10                  |                     |                       |                      | 17                  |                    |                      | 2.24                 | 0.8                 |                      |                     | 13.58               |                      |                         |
| 3.9   | 1023 | 1024     | Limonene                             | 0.38         |                       |                     | 0.06                  |                      |                     |                    |                      | 1.15                 | 16.63               |                      |                     | 1                   |                      |                         |
|       |      | 1025     | $\beta$ -Phellandrene                |              | 0.48                  |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 3.93  | 1024 | 1026     | 1,8-Cineole                          | 0.09         |                       | 15.16               |                       |                      |                     | 0.7                | 21.91                | 3.36                 | 0.1                 | 9.69                 | 15.4                | 0.35                | 3.09                 | 3.7                     |
| 4.03  | 1028 | 1026     | Benzyl alcohol                       | tr.          |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 4.11  | 1032 | 1032     | ( <i>Z</i> )- $\beta$ -Ocimene       | 0.09         |                       |                     | 0.35                  |                      |                     |                    |                      | 2.62                 | 3.5                 | 1.49                 |                     | 0.304               |                      | 7.6                     |
| 4.33  | 1041 | 1044     | ( <i>E</i> )- $\beta$ -Ocimene       | 0.03         | 10.38                 | 0.1                 | 6.79                  | 6.355                | 0.1                 | 0.1                |                      | 0.15                 | 0.2                 |                      |                     |                     |                      |                         |
| 4.54  | 1050 | 1054     | $\gamma$ -Terpinene                  | 0.01         | 0.36                  |                     | 0.32                  |                      | 30.3                | 0.2                |                      | 2.68                 | 0.4                 |                      |                     | 17.52               |                      |                         |
| 4.98  | 1069 | 1065     | <i>cis</i> -Sabinene hydrate         |              | 0.57                  |                     |                       | 0.666                | 0.9                 | tr.                |                      |                      | 2.4                 |                      |                     |                     |                      |                         |
|       |      | 1069     | 4-methylstyrene                      |              |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     | 1.54                |                      |                         |
| 5.19  | 1077 | 1083     | Fenchone                             | tr.          |                       |                     |                       |                      |                     |                    |                      | 0.86                 |                     |                      |                     |                     |                      |                         |
| 5.25  | 1079 | 1085     | <i>p</i> -Mentha-2,4-diene           | 0.03         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 5.39  | 1086 | 1088     | Methyl benzoate                      | tr.          |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 5.5   | 1090 | 1086     | Terpinolene                          | tr.          |                       |                     |                       |                      |                     | 0.3                |                      |                      | 0.1                 |                      |                     |                     |                      |                         |
|       |      | 1098     | <i>trans</i> -Sabinene hydrate       |              |                       |                     |                       |                      |                     |                    |                      |                      | 0.6                 |                      |                     |                     |                      |                         |
| 5.6   | 1094 | 1095     | Linalool                             | 0.3          |                       | 0.34                | 0.29                  |                      |                     | 0.2                | 32.95                | 0.12                 | 0.7                 | 0.42                 |                     |                     |                      | 1                       |
| 5.91  | 1107 | 1107     | Phenyl ethyl alcohol                 | 0.01         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 6.42  | 1124 | 1128     | <i>allo</i> -Ocimene                 | tr.          |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 6.74  | 1134 | 1141     | Camphor                              | 4.04         |                       |                     |                       |                      |                     | 0.3                | 11.97                |                      |                     |                      |                     | 0.11                |                      |                         |
| 7.38  | 1161 | 1165     | Borneol                              | 0.02         |                       |                     |                       |                      | 0.1                 | 0.1                |                      |                      |                     |                      |                     | 0.41                |                      |                         |
|       |      | 1166     | $\delta$ -Terpineol                  |              | 0.64                  | 0.12                |                       |                      |                     |                    |                      | 0.06                 |                     |                      |                     |                     |                      |                         |
|       |      | 1177     | Terpinen-4-ol                        |              |                       | 0.16                | 0.75                  |                      | 0.5                 | 0.7                | 0.79                 | 0.16                 | 1.3                 |                      |                     | 1.75                |                      |                         |
|       |      | 1188     | $\alpha$ -Terpineol                  |              |                       | 0.31                | 0.06                  |                      | 0.1                 | tr.                | 3.36                 | 0.11                 | 0.1                 |                      |                     | 1.93                |                      |                         |
| 8.51  | 1192 | 1195     | Eugenol                              | 2.73         |                       |                     |                       |                      |                     |                    |                      | 7.78                 |                     |                      | 0.25                |                     |                      |                         |
|       |      | 1218     | <i>endo</i> -Fenchyl acetate         |              |                       |                     |                       |                      |                     |                    |                      | 0.1                  |                     |                      |                     |                     |                      |                         |
|       |      | 1223     | Citronellol                          |              |                       |                     |                       |                      |                     |                    |                      | 0.17                 |                     |                      |                     |                     |                      |                         |
|       |      | 1229     | <i>exo</i> -fenchyl acetate          |              |                       |                     |                       |                      |                     |                    |                      | 0.17                 |                     |                      |                     |                     |                      |                         |
|       |      | 1232     | Thymol methyl ether                  |              |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
|       |      | 1247     | ( <i>Z</i> )-Anethole                |              |                       |                     |                       |                      |                     |                    |                      | 0.17                 |                     |                      |                     |                     |                      |                         |
| 10.36 | 1242 | 1247     | <i>p</i> -Anisaldehyde               | 0.05         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
|       |      | 1249     | Geraniol                             |              |                       |                     |                       |                      |                     |                    |                      | 0.27                 |                     |                      |                     |                     |                      |                         |
| 10.54 | 1247 | 1254     | Phenyl ethyl acetate                 | 0.03         |                       |                     |                       |                      | 0.5                 |                    |                      |                      |                     |                      |                     |                     |                      |                         |
|       |      | 1282     | ( <i>E</i> )-Anethole                |              |                       |                     |                       |                      |                     |                    |                      | 34.95                |                     |                      |                     |                     |                      |                         |
|       |      | 1289     | Thymol                               |              |                       |                     |                       |                      | 33.4                |                    |                      | 4.47                 |                     |                      |                     | 32.88               |                      |                         |
|       |      | 1298     | Carvacrol                            |              |                       |                     |                       |                      | 1.2                 |                    |                      |                      |                     |                      |                     | 1.18                |                      |                         |
| 12.3  | 1295 | 1299     | Methyl ( <i>Z</i> )-cinnamate        | 7.37         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 13.97 | 1338 | 1348     | $\alpha$ -Cubebene                   | 0.03         | 0.22                  |                     |                       |                      |                     |                    |                      |                      | 0.2                 |                      |                     |                     |                      |                         |
| 14.4  | 1350 | 1356     | Eugenol                              | 13.9         | 59.45                 | 74.83               | 69.08                 | 77.39                |                     | 57.1               | 7.42                 | 9.07                 | 52.9                | 72.26                | 73.6                |                     | 70.96                | 56.4                    |
| 14.89 | 1362 | 1374     | $\alpha$ -Copaene                    | 0.32         | 2.48                  |                     |                       | 1.248                |                     | 0.6                |                      | 0.1                  | 2.3                 |                      | t                   |                     |                      | 2                       |
| 15.5  | 1378 | 1376     | Methyl ( <i>E</i> )-cinnamate        | 62.39        |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
|       |      | 1387     | $\beta$ -Bourbonene                  |              | 0.96                  |                     |                       | 0.716                |                     | 0.5                |                      | 0.05                 | 0.1                 |                      |                     |                     |                      |                         |
|       |      | 1387     | $\beta$ -Cubebene                    |              | 11.78                 |                     |                       |                      |                     | 0.1                |                      |                      |                     |                      |                     |                     |                      | 10.9                    |
|       |      | 1389     | $\beta$ -Elemene                     |              | 0.54                  |                     |                       |                      | 0.2                 |                    | 0.93                 | 0.12                 | 1.9                 |                      |                     |                     |                      |                         |
| 16.26 | 1398 | 1393     | Vanilline                            | 0.41         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 16.51 | 1404 | 1403     | Methyleugenol                        | 0.14         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 16.65 | 1408 | 1408     | ( <i>E</i> )- $\beta$ -Caryophyllene | 1.09         | 7.00                  | 2.2                 | 3.48                  | 3.16                 | 1.7                 | 14.2               | 1.68                 | 0.65                 | 1.6                 | 3.8                  | t                   | 3.55                |                      | 3.5                     |
| 17.01 | 1417 | 1419     | $\beta$ -cedrene                     | tr.          |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
|       |      | 1421     | $\beta$ -Ylangene                    |              |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     | 4.99                 |                         |
|       |      | 1430     | $\beta$ -Copaene                     |              |                       |                     | 0.32                  | 0.743                |                     |                    |                      |                      | 0.3                 |                      |                     |                     |                      |                         |
| 17.43 | 1426 | 1432     | <i>trans</i> - $\alpha$ -Bergamotene | 2.65         |                       |                     |                       |                      |                     |                    | 1.07                 |                      |                     |                      |                     |                     |                      |                         |
| 17.7  | 1433 | 1440     | ( <i>Z</i> )- $\beta$ -Farnesene     | 0.08         |                       |                     |                       |                      |                     | tr.                |                      |                      | 0.1                 |                      |                     |                     |                      |                         |
| 17.85 | 1437 | 1452     | $\alpha$ -Humulene                   | 0.59         | 0.53                  | 0.32                | 0.38                  |                      | 0.2                 | 0.1                |                      | 0.09                 | 0.2                 |                      |                     |                     |                      |                         |
| 18.36 | 1451 | 1454     | ( <i>E</i> )- $\beta$ -Farnesene     | 0.02         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      | 5.5                     |
| 18.86 | 1464 | 1465     | <i>cis</i> -Muurolo-4(14),5-diene    | 0.16         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 19.14 | 1473 | 1473     | Sesquisabinene                       | 0.79         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
| 19.39 | 1478 | 1476     | $\beta$ -Chamigrene                  | 0.65         |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     |                      |                         |
|       |      | 1478     | $\gamma$ -Muuroloene                 |              |                       | 0.51                | 0.11                  |                      |                     |                    |                      |                      | 0.1                 | 0.46                 |                     |                     |                      |                         |
|       |      | 1481     | $\gamma$ -Himachalene                |              |                       |                     |                       |                      |                     |                    |                      |                      |                     |                      |                     |                     | 2.62                 |                         |





- [1] R. S. Melo, A. M. A. Azevedo, A. M. G. Pereira, R. R. Rocha, R. M. B. Cavalcante, M. N. C. Matos, P. H. R. Lopes, G. A. Gomes, T. H. S. Rodrigues, H. S. Dos Santos, I. L. Ponte, R. A. Costa, G. S. Brito, F. E. A. Catunda Junior, V. A. Carneiro, *Molecules* **2019**, *24*, 1–17.
- [2] T. T. Anh, L. T. Duyen, L. M. Hang, T. D. Lam, L. G. Bach, D. C. Nguyen, T. Q. Toan, *Mater. Today: Proc.* **2019**, *18*, 4648–4658.
- [3] A. Silva Lima, M. N. Milhomem, O. Santos Monteiro, A. C. P. Arruda, J. A. M. de Castro, Y. M. L. Fernandes, J. G. S. Maia, L. M. Costa Junior, *Parasitol. Res.* **2018**, *117*, 59–65.
- [4] R. K. Joshi, *Acta Chromatogr.* **2017**, *29*, 111–119.
- [5] P.-C. Lin, J. J. Lee, I. J. Chang, *J. Food Drug Anal.* **2016**, *24*, 464–470.
- [6] B. R. Rajeswara Rao, S. K. Kothari, D. K. Rajput, R. P. Patel, M. P. Darokar, *Nat. Prod. Commun.* **2011**, *6*, 1705–1710.
- [7] L. O. Orafidiya, J. Kaloustian, E. O. Agbani, H. Portugal, A. Abudu, O. A. Igbenighu, L. Abou, C. Mikail, B. Ivaldi, G. A. Adetogun, *Int. J. Essent. Oil Ther.* **2007**, *1*, 103–109.
- [8] S. M. Kita, C. Vincent, J.-P. Schmit, A. Blanger, *Flavour Fragrance J.* **2000**, *15*, 339–341.
- [9] J. A. Pino, A. Rosado, V. Fuentes, *J. Essent. Oil Res.* **1996**, *8*, 139–141.
- [10] M. K. do Nascimento Silva, V. R. de Alencar Carvalho, E. F. F. Matias, *Pharmacogn. J.* **2016**, *8*, 4–9.
- [11] K. Cimanga, S. Apers, T. De Bruyne, S. Van Miert, N. Hermans, J. Totte, L. Pieters, A. J. Vlietinck, K. Kambu, L. Tona, *J. Essent. Oil Res.* **2002**, *14*, 382–387.
- [12] F. B. M. Mohr, C. Lermen, Z. C. Gazim, J. E. Gonçalves, O. Alberton, *Genet. Mol. Res.* **2017**, *16*, 1–10.
- [13] X. Ouyang, L. Wei, Y. Pan, S. Huang, H. Wang, G. B. Begonia, S. I. N. Ekunwe, *Med. Chem. Res.* **2013**, *22*, 1124–1130.
- [14] M. Brada, L. H. Khelifa, D. Ach, J. P. Wathelet, G. Lognay, *J. Essent. Oil-Bear. Plants* **2011**, *14*, 810–814.
- [15] L. Jirovetz, G. Buchbauer, M. P. Shafi, M. M. Kaniampady, *Eur. Food Res. Technol.* **2003**, *217*, 120–124.
- [16] X. Guo, *Trans. Chin. Soc. Agric. Eng.* **2019**, *35*, 299–307.
- [17] Venuprasad, H. Kumar Kandikattu, S. Razack, F. Khanum, *S. Afr. J. Bot.* **2014**, *92*, 151–158.
- [18] T. Buxton, S. Takahashi, A.-M. Eddy Doh, J. Baffoe-Ansah, E. O. Owusu, C.-S. Kim, *Pest Manage. Sci.* **2020**, *76*, 257–267.
- [19] R. P. Adams, *Identification of essential oil components by gas chromatography/mass spectrometry*, Allured publishing corporation Carol Stream, IL, **2007**, Vol. 456.
- [20] C. Marlatt, C. T. Ho, M. Chien, *J. Agric. Food Chem.* **1992**, *40*, 249–252.
- [21] R. E. Mokni, S. Hammami, S. Dall'Acqua, G. Peron, K. Faidi, J. P. Braude, H. Sebei, M. H. E. Aouni, *Nat. Prod. Commun.* **2016**, *11*, 1934578X1601100640.
- [22] <http://www.thegoodscentscompany.com> downloaded on 28/09/2022.
- [23] N. C. Huong, T. T. K. Ngan, T. T. Anh, X. T. Le, T. D. Lam, M. H. Cang, T. T. T. Huong, N. D. Y. Pham, *IOP Conf. Ser.: Mater. Sci. Eng.* **2020**, *736*, 1–6.
- [24] G. S. Cruz, V. Wanderley-Teixeira, L. M. da Silva, K. A. Dutra, C. A. Guedes, J. V. de Oliveira, D. M. A. F. Navarro, B. C. Araujo, L. A. C. Teixeira, *J. Essent. Oil-Bear. Plants* **2017**, *20*, 1360–1369.
- [25] R. S. Verma, A. Kumar, P. Mishra, B. Kuppasamy, R. C. Padalia, V. Sundaresan, *J. Essent. Oil Res.* **2016**, *28*, 35–41.
- [26] H. Mith, E. Yayi-Ladékan, S. D. Sika Kpoviessi, I. Yaou Bokossa, M. Moudachirou, G. Daube, A. Clinquart, *J. Essent. Oil-Bear. Plants* **2016**, *19*, 1413–1425.
- [27] S. M. Pessoa, N. A. Heredia Zarate, M. C. Vieira, C. A. L. Cardoso, N. R. Poppi, A. S. N. Formagio, L. R. Silva, *Rev. Bras. Plant. Med.* **2015**, *17*, 18–25.
- [28] F. Ocheng, F. Bwanga, M. Joloba, A. Softrata, M. Azeem, K. Pütsep, A.-K. Borg-Karlson, C. Obua, A. Gustafsson, *Evid. Based Complementary Altern. Med.* **2015**, *2015*, 1–10.
- [29] C. Giuliani, L. Lazzaro, R. Calamassi, L. Calamai, R. Romoli, G. Fico, B. Foggi, M. Mariotti Lippi, *Phytochemistry* **2016**, *130*, 128–143.
- [30] M. Leonardi, K. E. Ambryszewska, B. Melai, G. Flamini, P. L. Cioni, F. Parri, L. Pistelli, *Chem. Biodiversity* **2013**, *10*, 343–355.
- [31] L. Saint-Lary, C. Roy, J.-P. Paris, J.-F. Martin, O. P. Thomas, X. Fernandez, *Metabolomics* **2016**, *12*, 1–11.
- [32] W. A. König, C. Fricke, Y. Saritas, B. Momeni, G. Hohenfeld, *J. High Resolut. Chromatogr.* **1997**, *20*, 55–61.
- [33] M. Pierson, X. Fernandez, S. Antoniotti, *Sci. Rep.* **2021**, *11*, 11096.
- [34] T. K. T. Do, F. Hadji-Minaglou, S. Antoniotti, X. Fernandez, *TRAC Trends Anal. Chem.* **2015**, *66*, 146–157.
- [35] A. K. Djibo, A. D. Samaté, M. Nacro, *C.R. Chimie* **2004**, *7*, 1033–1037.
- [36] O. Ekundayo, I. Laakso, R. Hiltunen, *Flavour Fragrance J.* **1989**, *4*, 17–18.
- [37] L. Ntezurubanza, J. J. Scheffer, A. Looman, *Pharm. Weekbl Sci.* **1985**, *7*, 273–6.
- [38] R. K. Upadhyay, L. N. Misra, G. Singh, *Phytochemistry* **1991**, *30*, 691–693.
- [39] S. C. Gupta, S. N. Sobti, *Indian Perfum.* **1990**, *34*, 253–259.
- [40] Y. K. Sarin, S. G. Agarwal, R. K. Thappa, K. Singh, B. K. Kapahi, *J. Essent. Oil Res.* **1992**, *4*, 515–519.
- [41] C. C. Xaasan, A. D. Cabdulraxmaan, S. Passannanti, F. Piozzi, J. P. Schmid, *J. Nat. Prod.* **1981**, *44*, 752–753.
- [42] N. F. S. Morsy, K. S. M. Hammad, *J. Food Sci. Technol.* **2021**, *58*, 2599–2605.
- [43] S. Antoniotti, *Molecules* **2014**, *19*, 9203–9214.
- [44] C. Bouhlef, G. A. Dolhem, X. Fernandez, S. Antoniotti, *J. Agric. Food Chem.* **2012**, *60*, 1052–1058.
- [45] M. Lecourt, S. Antoniotti, *ChemSusChem* **2020**, *13*, 5600–5610.

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