Volatile constituents from tea of roselle (Hibiscus sabdariffa L.)

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INTRODUCTION

The roselle, locally called ‘flor de Jamaica’, is the species Hibiscus sabdariffa L., which belongs to the botanical family Malvaceae and grows very well in Cuba. It is an annual shrub, which produces fleshy, red, edible calyces with a unique taste and flavor. The calyces contain large amounts of pectin, anthocyanin and ascorbic acid and can be used for making jellies, jams, sauces and beverages.1

There are few reports related to the aroma of roselle. More than 25 compounds were found in the seed oil, which had been analyzed by GC-MS and GC-FTIR.2 The volatiles of a tea from roselle calyces collected in Taiwan were analyzed and 37 volatiles were identified by GC-MS.3

The purpose of this study is to analyze the volatile constituents of roselle tea in order to determine the respective influence of its volatiles.

EXPERIMENTAL

Materials and Isolation of the volatile constituents

Samples of roselle were collected from a agricultural experimental station in Havana, in December 2001. The Likens-Nickerson steam distillation procedure was utilized to mimic the preparation of roselle tea. After addition of an internal standard (methyl undecanoate, 2 mg), calyces (200 g) were blended with distilled water (800 mL) and simultaneously distilled and extracted for 60 min in a Likens-Nickerson microapparatus with 25 mL of diethyl ether (previously redistilled and checked as to purity). The volatile concentrate was dried over anhydrous sodium sulfate and concentrated to 0.6 mL on a Kuderna-Danish evaporator with a 12-cm Vigreux column and then to 0.2 mL with a gentle nitrogen stream.

Analysis of the volatile constituents

The extract was analysed by HRGC using a Konik 4000A gas chromatograph equipped with a flame ionization detector (FID). The separations were performed using an SPB-5 column (30 m X 0.25 mm I.D., 0.25 µm film thickness, Supelco Inc., Bellefonte, PA). The initial temperature was 60 °C (2 min) and the column was then programmed at 4 °C/min to 250 °C (20 min). The carrier gas was helium at a flow-rate of 1 mL/min. The temperature of the injector and detector was 250 °C. The injection was made in the split mode (1:10 ratio). Linear retention indices (RI) were calculated against those of n-paraffins.4 These conditions were used for quantitative analysis, by the internal standard method. The recovery of the method was determined by the standard addition technique applied to a sample.

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Table 1. Volatile constituents of roselle tea.

<table>
<thead>
<tr>
<th>Compound</th>
<th>RI</th>
<th>Concentration (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,3-dimethylbutane</td>
<td>610</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>isobutanol b</td>
<td>622</td>
<td>0.15</td>
</tr>
<tr>
<td>2-pentancene b</td>
<td>689</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>2-methylbutanal b</td>
<td>715</td>
<td>0.06</td>
</tr>
<tr>
<td>3-methyl-1-butanol b</td>
<td>736</td>
<td>0.14</td>
</tr>
<tr>
<td>2-methyl-1-butanol b</td>
<td>739</td>
<td>0.06</td>
</tr>
<tr>
<td>isobutanoic acid b</td>
<td>744</td>
<td>0.01</td>
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<tr>
<td>2-ethylfuran</td>
<td>773</td>
<td>0.03</td>
</tr>
<tr>
<td>hexanal</td>
<td>802</td>
<td>0.12</td>
</tr>
<tr>
<td>2-furfural</td>
<td>830</td>
<td>0.13</td>
</tr>
<tr>
<td>2-methylbutanoic acid b</td>
<td>841</td>
<td>0.02</td>
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<tr>
<td>(Z)-2-hexenal</td>
<td>853</td>
<td>0.02</td>
</tr>
<tr>
<td>2-furfuryl alcohol b</td>
<td>854</td>
<td>0.02</td>
</tr>
<tr>
<td>(Z)-3-hexen-1-ol b</td>
<td>856</td>
<td>0.02</td>
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<tr>
<td>α-angelica lactone b</td>
<td>861</td>
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<tr>
<td>1-xylene b</td>
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<tr>
<td>heptanal b</td>
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<td>0.02</td>
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<tr>
<td>2-acetylfuran b</td>
<td>910</td>
<td>0.02</td>
</tr>
<tr>
<td>(E)-2-heptenoic acid b</td>
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<td>0.03</td>
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<tr>
<td>benzaldehyde b</td>
<td>961</td>
<td>0.03</td>
</tr>
<tr>
<td>5-methyl-2-furfural</td>
<td>963</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>2,2,6-trimethyl-4-vinyltetrahydrofuran b</td>
<td>969</td>
<td>0.13</td>
</tr>
<tr>
<td>pentyl propanoate b</td>
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<tr>
<td>methyl-2-furoate b</td>
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<td>0.03</td>
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<tr>
<td>1-octen-3-ol b</td>
<td>978</td>
<td>0.05</td>
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<tr>
<td>6-methyl-5-hepten-2-one b</td>
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<td>0.02</td>
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<tr>
<td>octanal</td>
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<tr>
<td>α-terpinene b</td>
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<td>α-cymene b</td>
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<td>limonene</td>
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<td>(Z)-β-ocimene b</td>
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<td>1-propylbenzene b</td>
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<td>(E)-β-ocimene b</td>
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<td>acetophenone b</td>
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<td>&lt; 0.01</td>
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<td>octanol b</td>
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<tr>
<td>cis-linalool oxide (furanoid form)</td>
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<td>0.36</td>
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<tr>
<td>trans-cymene b</td>
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<tr>
<td>cis-linalool oxide (furanoid form)</td>
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<tr>
<td>linalool</td>
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<td>0.58</td>
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<td>nonanal</td>
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<td>2-phenethyl alcohol b</td>
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<tr>
<td>myrcenol b</td>
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<td>cis-β-terpineol b</td>
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<td>(E)-2-nonenal b</td>
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<td>ethyl benzoate b</td>
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<tr>
<td>terpinen-4-ol b</td>
<td>1177</td>
<td>0.08</td>
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<td>1-cymene-8-ol b</td>
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<td>0.06</td>
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<tr>
<td>α-terpineol b</td>
<td>1189</td>
<td>0.55</td>
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</table>

RESULTS AND DISCUSSION

The volatile constituents of roselle tea fruit were obtained by simultaneous steam distillation-solvent extraction and analyzed by GC and GC-MS using fused silica capillary columns. Table 1 summarizes the qualitative and quantitative analyses of the volatiles according to order of elution on the SPB-5 column. The yield of total volatiles, estimated by the addition of a measured amount of internal standard to the calyces, was approximately 6 mg/kg of calyces.

Eighty-one volatile constituents were identified, 66 of them reported for the first time as constituents of roselle tea. The major constituents found in roselle tea were linalool (0.58 mg/kg) and α-terpineol (0.55 mg/kg). A rough survey of the chemical classes represented in the identified volatiles was as follows: Terpenoids comprise the largest class of volatiles (48.2 %); the composition of the other classes of compounds was as follows: fatty acids, 12.4 %; alcohols, 8.6 %; phenols, 6.6 %; esters, 3.9 %; furanoids, 3.8 %; and others, 16.3 %.

Some compounds present, e.g. 2-furfural and 5-methyl-2-furfural, are probably degradation products of ascorbic acid and sugars. It is well-known that 2-furfural and 5-methyl-2-furfural could be formed from ascorbic acid and sugar degradation.7,8

To be continued on following page.
Odor descriptors provided by an informal sensory panel included floral, acidic and caramel-like. The presence of linalool, α-terpineol and linalool oxides could be responsible for the floral notes, while the fatty acids could be responsible for the acidic notes observed in roselle tea. The impression of caramel-like odor due presumably to constituents such as 2-furfural, furfuryl alcohol and 5-methyl-2-furfural.

Taking together, these results indicate that the typical aroma character of roselle tea is the interaction of floral (linalool, α-terpineol and linalool oxides), with acidic (fatty acids) and caramel-like (sugar degradation products) notes contributing to the complexity of the flavor.

**BIBLIOGRAPHY**


<table>
<thead>
<tr>
<th>Compound</th>
<th>RIa</th>
<th>Concentration (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>methyl salicylateb</td>
<td>192</td>
<td>0.05</td>
</tr>
<tr>
<td>decanalb</td>
<td>205</td>
<td>0.05</td>
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<td>α-menthen-9-alb</td>
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<td>thymoquinoneb</td>
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<td>geraniolb</td>
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<td>decanolb</td>
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<td>(α-α-anetholeb</td>
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<td>1-methylnaphthaleneb</td>
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<td>indoleb</td>
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<td>methyl anthranilateb</td>
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<td>geranyl acetoneb</td>
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<td>β-santaleneb</td>
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<td>(α-α-phytol acetateb</td>
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</tbody>
</table>

*aRetention indices on SPB-5.

*bReported for the first time in roselle tea.