HYDRODISTILLATION AND VOLATILE COMPOUNDS IDENTIFIED FROM DRIED POWDER JASMINE (*Jasminumsambac*L.) BY GC-MS

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ABSTRACT
Jasmine (*Jasminumsambac*L.) is one of scented Thai flower have been used for Thai traditional medicine. It has therapeutic properties and can be used as a cardiotonic agent, antidizziness, antidepressant, anti-inflammatotory and antiseptic. In the present study, hydrodistillation, the most common method was exploited to extract volatile oil from jasmine dried powder. Theyield of the extracts was 0.05% v/w. Chemical composition of the volatile oil were analysed by GC-MS, a process that integrated the features of gas chromatography and mass spectrometry to improve efficacy of volatile oil analysis. Volatile oil is complex mixture, constituted by terpenoid hydrocarbons, oxygenated terpenes. It was found that the main constituents of the volatile oil was (E,E)-7,11,15-Trimethyl-3-methylene-hexadeca-1,6,10,14-tetraene. Low yield of the jasmine volatile oils can be improved in future study by considered sample preparation and extraction process. GC-MS is selective, rapid and efficient for both the identification volatile oil components and composition variations.

Keywords: *Jasminumsambac*L., Hydrodistillation, GC-MS.

INTRODUCTION
Jasmine (*Jasminumsambac*L.), an aromatic flower is a major part of Thai traditional medicine (TTM) in many recipes such as Ya-hom (Teppajit, Nawakot, Tip-osod and Inthajak). Thai people believed that jasmine has been used for cardiotonic agent and antidizziness. Pharmacologilal studies have shown coronary vasodilator effect, cardiotropic activities, stimulate nerves system, anti-bacterial (*Streptococcus sanguinis* and *Pseudomonas aeruginosa*) and anti-fungal (*Aspergillus niger*). In clinical studies, (R)-(−)-linalool, an active compound in jasmine has sedative effect in patients and present unique aroma from flowers. Moreover, other chemical composition in volatile oil from jasmine are benzyl alcohol, benzyl acetate, jasmine lactone, methyl jasmine, geraniol, jasmine, jasmon, methyl benzoate, caryophyllene, cadinene, hexenyl benzoate, etc. During last centuries, Thai people use of these aromatic flowers as natural flavoring agent in Thai foods and Thai desserts. Nowadays, jasmine is developed to commercial product by extract with volatile organic solvent such as petroleum ether, hexane and liquid carbon dioxide. This product called concreteused in perfume, cosmetic, spa business and aroma therapy. Moreover, distillation is the common method for extracted volatile oil from aromatic plants. Hydrodistillation is commonly used to determine volatile oil for quality control in raw material of herbal medicine. There are several methods for extracting volatile oil from plant materials. Hydrodistillation is the most common method for volatile oil extraction. There are three types of hydrodistillation including water distillation, water and steam distillation and direct steam distillation. Many volatile oils from flowers, such as rose, jasmine, lavender, camomile, ylangylang, clove can be prepared by these
method. Although distillation is a simple method, there are many factors affecting the amount of volatile oil such as extracting method, duration time of distillation, distilled temperature. In case of plants which are unstable at high temperature, distillation must be done at low temperature with preferable solvent extraction. After the solvent has been evaporated, the wax residue called concrete was left. The commercial volatile oils are then separated from the insoluble waxes by dissolving in alcohol. Moreover, enfleurage is a method for extracting volatile oil from flowers by put petals of flower on a layer of animal fat. The fat saturated with volatile oil was extracted later. Gas Chromatography-Mass Spectrometry (GC-MS) is a process that integrated the features of gas chromatography and mass spectrometry to improve efficacy of qualitative and quantitative analysis within a test sample. The gas chromatograph applies which depend on the column (type, material, length, diameter, film thickness) as well as the phase properties. The mass spectrometer does this by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio. Applications of GC-MS include drug detection, plasma detection, fire investigation, environmental analysis, explosives investigation, and identification of unknown samples. Additionally, it can identify trace elements in materials that were previously thought to have disintegrated beyond identification.

The purposes of this study were to distill and identify the volatile compounds released from jasmine (Jasminum sambac L.). Since jasmine dried powder is a composition in many Thai traditional formulas, it is characterized to control the quality of raw material which active compound in volatile oil related to therapeutic effect. In addition, this study used for quality control of jasmine dried powder before apply to other formulation.

MATERIALS AND METHODS

MATERIALS
Distilled water for distillation from Puris® RO water system, model Expe-RO Ele 10-M, Korea. Hexane (Labscan) was purchased from TTK science, Bangkok, Thailand.

Preparation of flower samples
The dried flowers of Jasmine (Jasminum sambac L.) were purchased from Charoensuk Osot, Nakhon Pathom province, Thailand. Flowers were separated the contaminant such as gravel, insect, etc. Then ground and screened through a 40-mesh sieve. The obtained jasmine dried powders were packed in zipped-lock plastic bags and store at room temperature (25-30°C) for further experiments.

METHODS

Hydrodistillation
Jasmine dried powder (50 g) were accurately weigh and distilled in 500 mL of water in a 1000 mL round bottomed flask using Clevenger-type apparatus as described in Thai Herbal Pharmacopoeia, 2004. The distillation processes were performed in triplicate. Hydrodistillated condition set at the temperature of 130-150 °C and distillation rate 2-3 mL/min for 5 hours. Open the stopper of the apparatus until the volatile oil in the preparation line. Then stand for more than 1 hour at room temperature, the water and volatile oil were separated. The volume of the volatile oil was collected in millimeter and calculated percentage of the volatile oil content in sample using the expression:

\[
\% \text{ Volatile oil (v/w)} = \frac{V \times 100}{W}
\]

where \(V\) = the volume in mL of volatile oil \(W\) = the weight in g of dried powder

Volatile compound analysis using GC-MS
The components of the volatile oil were identified by GC-MS. The volatile oil from jasmine dried powder was dissolved in hexane and directly injected into the injection port of gas chromatograph (Agilent Technologies 7890A GC system) coupled with a mass spectrometer (Agilent Technologies 5975C inert XL EI/CI MSD with Triple-Axis Detector). The GC was operated on an Agilent J&W GC column HP-5 column (30 m x 0.32 mm, id. with 0.52μm film thickness) and helium was used as the carrier gas. The temperature program was started with an initial temperature of 150°C and held for 4 min at this temperature, then heated up to 170°C with a heating rate of 0.8°C/min and held for 1 min, heated up to 220°C with a heating rate of 3.0°C/min and held for 1 min, heated up to 240°C with a heating rate of 1.0°C/min and held for 1 min and heated up to 250°C with a heating rate of 5.0°C/min and held for 5 min as shown in Table 1 at a flow rate of 0.7 mL/min. The obtained mass spectra were preliminarily interpreted by comparing with those of Enhance Chemstation Version D00.00.38 (Agilent Technologies), the Mass Spectral Search Library of the National Institute of Standards and Technology (NIST, Gaithersburg, USA).
RESULTS AND DISCUSSION

Hydrodistillation

Hydrodistillation is commonly used for volatile oil extraction. This method is applied for the extraction of volatile oil from jasmine dried powder in this study. The physical appearances of the extracted oils were found to be yellow to brownish viscous solution to wax. The scent of the extracted oil obtained is similar to fresh jasmine flowers. The %yield of the extracts was very low (0.05% v/w). Since this method was indicated the dried powder being boiled in water, using heating mantle for 5 hours and relatively high temperature (130-150 °C), the compound can be destroyed during the process. Moreover, raw material (jasmine) was dried from oven before distillation process that affected to some volatile oil was lost. In case of jasmine dried powder, oils are difficult to extract from the surface of the powder unless the powder is wetted sufficiently enough for diffusion to take place. In this experiment used 50 g of jasmine dried powder per 500 mL of distilled water, this ratio is enough for wetted powder. Although hydrodistillation is an appropriate for extract volatile oil from many plants but this method has many disadvantage such as oil component like esters are sensitive to hydrolysis, tends to be a small operation (operated by one or two persons), it takes a long time to accumulate much oil, so good quality oil is often mixed with bad quality oil and slower process.

Volatile compound analysis using GC-MS

Analysis of volatile oil obtained hydrodistillation was done using gas chromatography couple with mass spectrometer method. The chromatogram generated by gas chromatography shows the composition of the volatile oil from jasmine dried powder is shown in Figure1. For mass spectrometer presents the percentage (%) of each component. There were found to be 12 volatile compounds identified from jasmine dried powder. The detected compounds are presented in Table2.

CONCLUSIONS

Hydrodistillation have been demonstrated for the extraction of volatile oil from jasmine dried powder. The %yield of the extracts was very low (0.05% v/w).The chemical compositions of the volatile oils from these flowers were identified by GC-MS, there were found to be 12 volatile compounds. The volatile oil contained many chemical groups, but they were mostly terpenes. It is difficult to distill volatile oil from dried powder as many factors can affect the quantity of the volatile oils. The quantity of volatile oil depended on raw material (jasmine dried powder) and method for extraction that affected to chemical composition in volatile oil. Therefore, it will be continue this study, especially process to prepare raw material and method of extraction in order to increase quality and quantity of volatile oil. GC-MS is a good method, due to its simplicity, rapidity and efficiency, for both the identification volatile oil components and composition variations.

ACKNOWLEDGEMENTS

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Fig. 1: The GC chromatogram of volatiles oil of jasmine dried powder

geraniol

(E,E)-7,11,13-trimethyl-5-methylene-hexadec-1,6,10,14-tetraene

9,12-Octadecadienoic acid (Z,Z)-, methyl ester

9,12,15-Octadecatrienoic acid, methyl (Z,Z,Z)-

9,12,15-Octadecatrienoic acid, ethyl ester, (Z,Z,Z)-
Fig. 2: Chemical structure of volatile oils from jasmine dried powder

Table 1: Working condition for GC-MS analysis

<table>
<thead>
<tr>
<th>Rate (°C/min)</th>
<th>Temp. (°C)</th>
<th>Hold (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-</td>
<td>150</td>
<td>4</td>
</tr>
<tr>
<td>0.8</td>
<td>170</td>
<td>1</td>
</tr>
<tr>
<td>3.0</td>
<td>220</td>
<td>1</td>
</tr>
<tr>
<td>1.0</td>
<td>240</td>
<td>1</td>
</tr>
<tr>
<td>5.0</td>
<td>250</td>
<td>5</td>
</tr>
</tbody>
</table>

Table 2: Volatile oil composition in jasmine dried powder

<table>
<thead>
<tr>
<th>RT</th>
<th>Constituent</th>
<th>Rate %</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.707</td>
<td>Geraniol</td>
<td>1.105</td>
</tr>
<tr>
<td>18.759</td>
<td>2(1H)-Azulene-4,5,6,7,8a-hexahydro-8a-methyl-(s)-</td>
<td>2.579</td>
</tr>
<tr>
<td>38.011</td>
<td>Hexadecanoic acid, methyl ester</td>
<td>3.925</td>
</tr>
<tr>
<td>42.284</td>
<td>Hexadecanoic acid, ethyl ester</td>
<td>4.004</td>
</tr>
<tr>
<td>43.998</td>
<td>(E,E)-7,11,15-Trimethyl-3-methylene-hexadeca-1,6,10,14-tetraene</td>
<td>42.203</td>
</tr>
<tr>
<td>47.065</td>
<td>9,12-Octadecadienoic acid,(ZZ)-, methyl ester</td>
<td>2.770</td>
</tr>
<tr>
<td>47.341</td>
<td>9,12,15-Octadecatrienoic acid, methyl ester (ZZ,Z)</td>
<td>10.323</td>
</tr>
<tr>
<td>49.842</td>
<td>Linoleic acid ethyl ester</td>
<td>2.231</td>
</tr>
<tr>
<td>50.091</td>
<td>9,12,15-Octadecatrienoic acid, ethyl ester (ZZ,Z)</td>
<td>7.645</td>
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<tr>
<td>54.127</td>
<td>9-Tricosene,(Z)-</td>
<td>15.487</td>
</tr>
<tr>
<td>55.096</td>
<td>Heneicosane</td>
<td>4.379</td>
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<tr>
<td>63.842</td>
<td>Hexacosane</td>
<td>3.348</td>
</tr>
</tbody>
</table>

REFERENCES


11. Rashid MFSA. Extraction of Essential Oils from Jasmine Flower Using Solvent Extraction Method. Faculty of Chemical & Natural Resources Engineering University College of Engineering & Technology Malaysia. 2006;17.