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Research Article

HYDRODISTILLATION AND VOLATILE COMPOUNDS IDENTIFIED FROM DRIED POWDER JASMINE (*JasminumsambacL.*) 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-inflammatory 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 recipies such as Ya-hom (Teppajit, Nawakot, Tip-osod and Inthajak)[1]. Thai people believed that jasmine has been use forcardiotonic agent andantidizziness. Pharmacoloigcal studies have shown coronary vasodilator effect, cardiotropicactivities, stimulate nervours system, anti-bacterial (*Streptococcus sanguinis and Pseudomonas aeruginosa*) and anti-fungal (*Aspergillusniger*). In clinical studies, (R)-(-)-linalool, an active compound in jasmine has sedative effect in patients and present unique aroma from flowers[2]. Moreover, other chemical composition in volatile oil from jasmine are benzyl alcohol, benzyl acetate[3], jasmine lactone, methyl jasmonte, geraniol, jasmine, jasmone, methyl benzoate, caryophyllene, cadinene, hexenyl benzoate, etc[2]. 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 productby extract with volatile organic solvent such as petroleum ether, hexane and liquid carbondioxide. This product called concreteused in perfume, cosmetic, spa business and aroma therapy[4]. 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[5]. 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 on 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[6].

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[7]. 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[8].

The purposes of this study were to distill and identify the volatile compounds released from jasmine (*JasminumsambacL.*). 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.

MATERAILS 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 (*Jasminumsambac*L.) were purchased from CharoensukOsot, NakhonPathom 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 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[9]. 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 Table1 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 hoursand 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[10]. 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.

The volatileoil contained a mixture of acyclic diterpenes (alcohols and hydrocarbons) and various straight-chain fatty acid derivates (alcohols, esters, and both saturated and unsaturated hydrocarbons inchain length carbon atoms) as shown in Figure2. The majority compounds were(E,E)-7,11,15-Trimethyl-3-methylene-hexadeca-1,6,10,14-tetraene(42.203 %). Geraniol is one of chemical constituents in jasmine[11] was also detected in low abundance (1.105 %). While linalool, an active compound in jasmine[2,4,12] and present unique aroma from flowers was not detected since raw material, jasmine was dried by hot air oven in 50-60 °C for 6 hours as a results, linalool may be lost from this process.

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 extractionthat affected to chemical composition in volatile oil. Therefore, it will becontinue 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.

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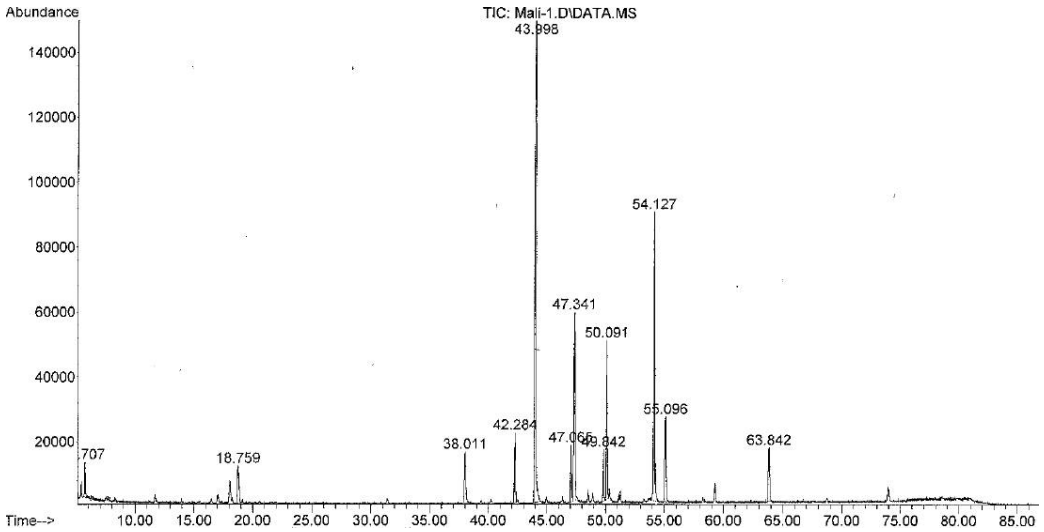
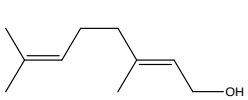
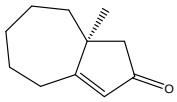


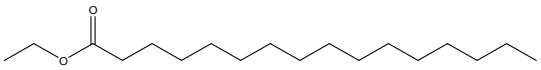
Fig. 1: The GC chromatogram of volatiles oil of jasmine dried powder



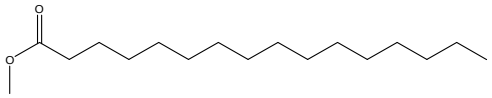
geraniol



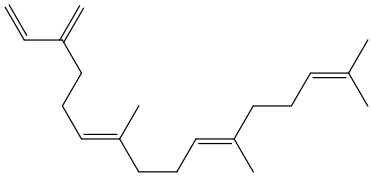
2(1H)-Azulenone,4,5,6,7,8,8a-hexahydro-8a-methyl-, (s)-



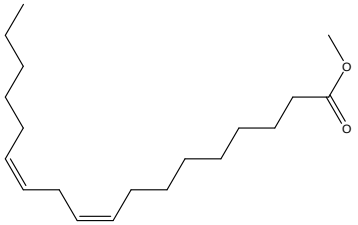
hexadecanoic acid, ethyl ester



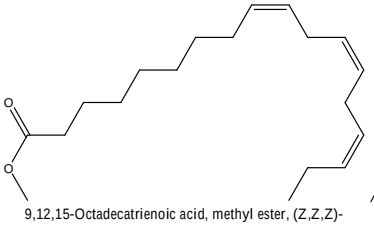
hexadecanoic acid, methyl ester



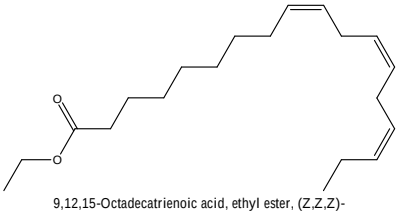
(E,E)-7,11,15-trimethyl-3-methylene-hexadeca-1,6,10,14-tetraene



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



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



9,12,15-Octadecatrienoic acid, ethyl ester, (Z,Z,Z)-

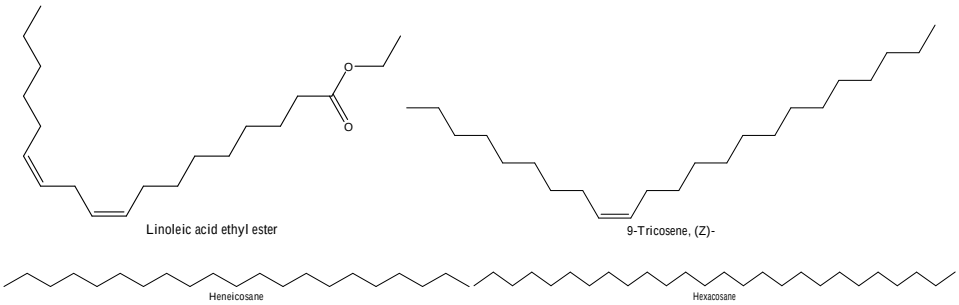


Fig. 2: Chemical structure of volatile oils from jasmine dried powder

Table 1: Working condition for GC-MS analysis

Rate (°C/min)	Temp. (°C)	Hold (min)
-	150	4
0.8	170	1
3.0	220	1
1.0	240	1
5.0	250	5

Table 2: Volatile oil composition in jasmine dried powder

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

REFERENCES

1. National Drug System Development committee, Ministry of public health. National List of Essential Medicines. 2013.
2. Ito Y, Sugimoto A, Kakuda T and Kubota K. Identification of potent odorants in Chinese jasmine green tea scented with flowers of Jasminumsambac. J Agric Food Chem. 2002;50:4878-84.
3. Thanapad S and Charoenpuchitnun S. The study of volatile Oils from ThaiFlowers. Faculty of Pharmacy, Mahidol University. Bangkok 1981.
4. Paibon W, Yimnoi CA, Tembab N, Boonlue W, Jampachaisri K and Nuengchamnong N. Comparison and evaluation of volatile oils from three different extraction methods for some Thai fragrant flowers. International Journal of Cosmetic Science. 2011;150–6.
5. Handa SS, Khanuja SPS, Longo G and Rakesh DD. Extraction Technologies for Medicinal and Aromatic Plants. International Center for Science and High Technology Trieste. 2008;115-21.
6. Pensuk W, PadumanondaT and Pichaehsoonthon C. Comparison of the chemical constituents in Micheliaalba flower oil extract by steam distillation, hexane extraction and enfleurage method. J Thai Tradit Altern Med. 2007;5:30–9.
7. Bramer SEV. An Introduction to Mass Spectrometry. Widener University. Department of Chemistry. 1998;26.
8. Sahil K, Prashant B, Akanksha M, Premjeet S and Devashish R. Gas Chromatography-Mass Spectrometry: Applications. International Journal of Pharmaceutical & Biological Archives. 2011; 2(6):1544-60.
9. Ministry of public health, Thailand .Thai Herbal Pharmacopoeia. 2004;3:159-60.

10. Ozek T. Distillation Parameters for Pilot Plant Production of LaurusnobilisEssential oil. Rec Nat Prod. 2012;6(2):135-43.
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.
12. Samakradhamrongthai R, Utama-Ang N and Thakeow P. Identification of volatile compounds released from dry scented Thai flowers and their potential application in flower-mixed tea. As. J Food Ag-Ind. 2009;2(04):525-34.