UNIVERSITI PUTRA MALAYSIA

MICROWAVE EXTRACTION OF ESSENTIAL OILS FROM JASMINUM SAMBAC FLOWERS

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Thesis

MICROWAVE EXTRACTION OF ESSENTIAL OILS FROM *JASMINUM SAMBAC* FLOWERS

By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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Faculty: Science

The Microwave Extraction System (MES) is a high-speed method used to extract target compounds from various raw materials. It was originally developed for the extraction of flavors and fragrances from plant materials, which is generally carried out by conventional techniques that require a lot of energy or a long time or a combination of both. In this study, a microwave extraction system (MES) is developed for the extraction of essential oils from *Jasminum sambac* flowers. The important MES process parameters, i.e, time, temperature and microwave power are controlled to obtain rapidly the highest quantity and quality of oil. The performance of MES is compared with the Conventional Extraction Method (CEM) using various methods such as dry distillation (DD), wet distillation (WD), hydro distillation (HD) and steam distillation (SD) in terms of rapidity, quality and percentage of yield, chemical composition of oil and its efficiency. The MES is performed by DD and WD at irradiation power of about 450 W and temperature about 100 ^{o}C for 1 hour whereas CEM is performed by HD and SD at

extraction power of 450 W and temperature 100 ^{o}C for 8 hours. The absorption power of microwave irradiation by *Jasminum sambac* flowers are predicted for DD and WD. The project has successfully proved that MES is more efficient than CEM in terms of rapidity, quality and quantity of yield, energy use, easiness in operation and cost effectiveness. The MES provides a rapid extraction, with about 8 times faster than CEM. After 1 hour of microwave extraction, it is possible to collect sufficient essential oil which provides comparable yields to those obtained with 8 hours by CEM. Using MES, DD requires only 5 minutes to obtain its first oil droplet and WD requires 8 minutes whereas using CEM, both HD and SD allows 15 and 42 minutes respectively to obtain the first oil droplet. DD requires less time to obtain its first oil droplet compared to other methods because the absorption power for dry sample is higher than for wet sample. The DD technically gives higher percentage yield with 0.10 % which is about 5 times higher compared to SD and HD where both only contribute 0.02 %, respectively whereas WD contributes 0.06 %. Although more compounds are detected in the essential oil extracted by CEM, substantial higher amounts of highly odoriferous compounds are present in the MES extract. The relative amount of oxygenated fraction in essential oil is less than 30 % for CEM and more than 30 % for MES. In terms of the energy consumed, the energy consumption in MES is 8 times lower than in CEM. HD requires heating of 0.4 liter of water and 200 g of flowers to the extraction temperature, and evaporating the water and essential oil for 8 hours whereas DD only requires heating 200 g of fresh flowers and evaporating the in situ water and essential oil of the plant material for 1 hour. Furthermore, MES requires maximum power irradiation only to heat up the sample and water to its boiling point and thus obtain its first oil droplet whereas CEM requires maximum power irradiation throughout the process. Essentially, the MES provides an easily controlled system, rapid extraction process; high yield and purity extracts, more valuable essential oils, extracts with chemical compositions comparable to conventional method and allows a substantial saving of energy. These advantages not only reduce operating costs, but also result in a more environmentally friendly process.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENGEKSTRAKAN GELOMBANG MIKRO BAGI MINYAK PATI DARIPADA BUNGA *JASMINUM SAMBAC*

Oleh

NURSHAHIDAH BINTI OSMAN

November 2006

Pengerusi: **Profesor Kaida bin Khalid, PhD**

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Sistem Pengekstrakan Gelombang Mikro ataupun dikenali dengan *Microwave Extraction System* (MES) merupakan suatu kaedah berkelajuan tinggi yang digunakan untuk mengekstrak komposisi-komposisi sasaran daripada pelbagai bahan mentah. Sistem ini pada asalnya dimajukan untuk pengekstrakan bahan-bahan perisa dan pewangi daripada tumbuh-tumbuhan yang kebiasaannya dijalankan oleh kaedah-kaedah konvensional yang memerlukan penggunaan tenaga yang banyak atau masa pengekstrakan yang terlampau lama atau kedua-duanya sekali. Dalam kajian ini, sebuah sistem pengekstrakan gelombang mikro (MES) dibina untuk pengekstrakan minyak pati dari sampel bunga *Jasminum sambac*. Parameter-paramater proses MES yang penting seperti masa, suhu dan kuasa gelombang mikro dikawal untuk mencapai ekstrak minyak pati yang paling maksimum dan berkualiti tinggi dalam masa yang singkat. Prestasi MES dibandingkan dengan Teknik Pengekstrakan Biasa ataupun dikenali sebagai *Conventional Extraction Method* (CEM) menggunakan pelbagai kaedah seperti penyulingan kering (DD), penyulingan basah (WD), penyulingan hidro (HD) dan

penyulingan wap (SD) dari segi kepantasan, kualiti dan peratusan minyak, komposisi kimia minyak dan kecekapannya. MES dilakukan secara DD dan WD yang dijalankan pada kuasa sinaran 450 W dan suhu di sekitar 100 ^{o}C selama 1 jam manakala CEM dilakukan secara HD dan SD yang dijalankan pada kuasa pengekstrakan 450 W dan suhu 100 ^{o}C selama 8 jam. Kuasa serapan sinaran gelombang mikro oleh bunga *Jasminum sambac* diramal bagi DD dan WD. Projek ini telah membuktikan dengan jayanya bahawa MES lebih efisien berbanding CEM dari segi kepantasan, kualiti dan kuantiti minyak yang terhasil, penggunaan tenaga, kemudahan pengoperasian dan penjimatan kos. MES memberikan pengekstrakan yang cepat, lebih kurang 8 kali lebih pantas dari CEM. Selepas 1 jam pengekstrakan menggunakan gelombang mikro, adalah mungkin untuk mengumpul amaun minyak pati yang mencukupi bagi tujuan penyelidikan dan memberikan hasil ekstrak yang setara dengan 8 jam pengekstrakan menggunakan CEM. Didapati titisan minyak pertama yang jatuh dikesan antara minit kelima bagi DD dan minit kelapan bagi WD sementara HD dan SD masing-masing memerlukan 15 dan 42 minit untuk mencapai titisan minyak pati pertama. DD mengambil masa yang lebih singkat untuk mendapatkan titisan minyak pertama berbanding kaedah-kaedah lain kerana kuasa serapan bagi sampel kering lebih tinggi daripada kuasa serapan bagi sampel basah. Secara teknikal, DD menyumbangkan peratusan ekstrak minyak yang lebih tinggi iaitu sebanyak 0.1 % iaitu 5 kali ganda berbanding kaedah-kaedah CEM yang lain iaitu HD dan SD yang hanya menyumbangkan minyak sebanyak 0.02 % masing-masing manakala WD memberikan hasil sebanyak 0.06 %. Walaupun jumlah sebatian dikesan lebih banyak dalam ekstrak CEM, namun didapati lebih banyak sebatian pewangi yang wujud dalam ekstrak MES. Secara relatifnya, peratusan sebatian wangi di dalam minyak CEM adalah kurang dari 30

% sementara peratusan sebatian wangi di dalam minyak CEM adalah lebih dari 30 %. Dari segi penggunaan tenaga, MES menggunakan tenaga sebanyak 8 kali lebih rendah berbanding CEM. HD memerlukan pemanasan 0.4 liter air dan 200 g sampel bunga untuk mencapai suhu pengekstrakan 100 ^{o}C dan mengewap minyak pati di dalam sampel serta tambahan air yang dibuat selama 8 jam manakala DD hanya memerlukan pemanasan 200 g sampel bunga dan mengewap kandungan minyak pati dan air di dalam sampel selama 1 jam. Tambahan pula, MES memerlukan penggunaan sinaran kuasa maksimum hanya untuk memanaskan sampel dan air ke takat didihnya dan seterusnya mencapai titisan minyak pertama manakala CEM memerlukan sinaran kuasa maksimum sepanjang proses berjalan. Paling utama, MES merupakan suatu sistem kawalan yang amat mudah, proses pengekstrakan yang pantas, menghasilkan minyak yang lebih banyak, tulen serta berkualiti tinggi, memberikan komposisi minyak pati yang setara dengan CEM serta membenarkan penjimatan tenaga yang banyak. Kelebihan-kelebihan ini bukan sahaja menjimatkan kos malah menyumbangkan kepada suatu proses yang mesra alam.

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I certify that an Examination Committee has met on 17th November 2006 to conduct the final examination of Nurshahidah binti Osman on her Master of Science thesis entitled "Microwave Extraction of Essential Oils from *Jasminum sambac* Flowers" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the examination Committee are as follows:

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This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

NURSHAHIDAH BINTI OSMAN

Date:

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£	pound
eV	electron volts
T	temperature
T_W	temperature of water
T_R	room temperature
T_f	final temperature
T_i	initial temperature
ΔT_w	temperature rise of water
t	time
Δt_w	heating time of water
τ	relaxation time
ω	angular frequency
c	speed of light
c_o	velocity of propagating wave in free space ($c_o = 3.0 \times 10^8$ m/s)
π	pi ($\pi = 3.142$)
f	frequency
λ	wavelength
λ_0	free space wavelength
E	electric field strength (unit: V/m)
H	magnetic field strength or magnetic intensity (unit: A/m)
$\nabla \times H$	curl of H
∇	del operator

j	$\sqrt{-1}$
J	current density
σ	conductivity
ε	permittivity
ε'	dielectric constant
ε''	loss factor
$\tan \delta$	loss tangent or the ratio of ε'' to ε''
ε_o	permittivity of free space ($\varepsilon_o = 8.85 \times 10^{-12}$ F/m)
ε'_w	dielectric constant of water
ε''_w	loss factor of water
ε'_{mixs}	relative dielectric permittivity of mixture for solid materials
ε'_s	dielectric constant of solid materials
$\varepsilon''s$	loss factor of sample
ε^*	complex permittivity or dielectric properties of material
ε^*_r	relative permittivity
ε'_r	relative dielectric constant
ε''_r	relative loss factor
ε''_d	dipolar polarizations
ε''_e	electronic polarizations
ε''_a	atomic polarizations
ε''_i	interfacial (Maxwell-Wagner) polarizations
ε''_c	conductive losses
ε'_s	static permittivity
ε'_∞	permittivity for infinite frequency

ε'_J	dielectric constant of Jasmine (experimental)
ε''_J	loss factor of Jasmine (experimental)
ε^*_w	dielectric permittivity of water added to jasmine
ε^*_a	dielectric permittivity of air in jasmine
ε^*_{mixJF}	dielectric properties of mixture for fresh jasmine
ε^*_{mixJD}	dielectric properties of mixture for dry jasmine
ε^*_{mfixJW}	dielectric properties of mixture for wet jasmine
ε'_{mixJF}	dielectric constant of mixture for fresh jasmine (mixture model)
ε''_{mixJF}	loss factor of mixture for fresh jasmine (mixture model)
ε'_{mixJD}	dielectric constant of mixture for dry jasmine (mixture model)
ε''_{mixJD}	loss factor of mixture for dry jasmine (mixture model)
ε'_{mixJW}	dielectric constant of mixture for wet jasmine (mixture model)
ε''_{mixJW}	loss factor of mixture for wet jasmine (mixture model)
PA	absorption power or the energy developed per unit volume (W/m^3)
PA_w	absorption power per unit volume by water (W/m^3)
PA_s	absorption power per unit volume by sample (W/m^3)
PA_{JD}	absorption power per unit volume by dry Jasmine (W/m^3)
PA_{JW}	absorption power per unit volume by wet Jasmine (W/m^3)
Ein_s	electric field strength inside sample (V/m)
Eo_s	electric field strength outside sample (V/m)
Ein_{JD}	electric field strength inside dry jasmine (V/m)
Ein_{JW}	electric field strength inside wet jasmine (V/m)
Ein_w	electric field strength inside water (V/m)

Eo_w	electric field strength outside water (V/m)
d_p	penetration depth or power penetration
Z_o	wave impedance of free space (approximately 377 Ω)
P	polarization
h	specific heat of the material
h_w	specific heat of water
L	Latent heat of water
MC_J	moisture content of fresh jasmine flowers
MC_d	moisture content defined on a dry basis
MC_w	moisture content defined on a wet basis
SG	specific gravity
P_{obj}	density of object
ρ_{JF}	density of fresh jasmine
ρ_{JD}	density of dried jasmine
ρ_w	density of water (ρ_w = 1.000 g/cm^3)
m_w	mass of water
$m_{w(added)}$	Mass of water to submerge fresh jasmine
m_s	mass of sample
m_m	mass of moist material
m_d	mass of dry material
m_a	Mass of air
m_{JFa}	Mass of fresh jasmine to determine volume fraction of air
m_{JF}	Mass of fresh jasmine to extract
M_w	wet weight of the sample

V_s	volume fraction of sample
V_J	volume fraction of water in fresh jasmine ($V_J=(MC_J/(MC_J+((1-MC_J)(\rho_w/\rho_{JD}))))$)
Vol_w	Volume of water to submerge fresh jasmine ($Vol_w = m_{w(added)}/\rho_w$)
V_w	Volume fraction of Water ($V_w = Vol_w / Vol_w + Vol_{JF}$)
Vol_a	Volume of air ($Vol_a = m_a / \rho_w$)
V_a	Volume fraction of air ($V_a = Vol_a / Vol_a + Vol_{JFa}$)
Vol_{JFa}	Volume of fresh jasmine to determine volume fraction of air ($Vol_{JFa} = m_{JFa}/\rho_{JF}$)
Vol_{JF}	Volume of fresh jasmine to extract ($Vol_{JF} = m_{JF} / \rho_{JF}$)
SFME	Solvent Free Microwave Extraction
MAE	Microwave-Assisted Extraction
MES	Microwave Extraction System
CEM	Conventional Extraction Method
DD	Dry Distillation
WD	Wet Distillation
HD	Hydro Distillation
SD	Steam Distillation
GC	Gas Chromatography
MS	Mass Spectrometer
GC-MS	Gas Chromatography coupled to Mass Spectrometry
CAS	Chemical Abstract Number
m/z	mass to charge ratio
OC	oxygenated compound
TC	terpene compound

ppm	parts per million
H_2O	chemical description for water
CO_2	carbon dioxide
NaCl	natrium chloride
EM	Electromagnetic
VHF	Very High Frequency
ANA	Automated Network Analyser

CHAPTER 1

INTRODUCTION & LITERATURE REVIEW

1.1 Introduction

The initial surge in microwave technology development was driven by the military needs of the Second World War. The tremendous effort that went into development of radar during Second World War generated a great body of knowledge on the properties of microwaves and related technologies [1]. After the Second World War, a rapid growth of microwaves system has taken place in the area of telecommunication and navigation in both civilian and military. However, industrial, scientific, medical and domestic applications have developed at a slower pace. Most microwave applications fall in the range 300 MHz to 300 GHz [2].

By far, the most popular application of microwave power is in microwave oven for domestic and commercial cooking, heating and drying. Household and institutional microwave ovens operate at the frequency of 2450 MHz and the lower microwave frequency 915 MHz is currently only used for industrial applications. On the other hand, a greater variety of industrial applications of high microwave power has been demonstrated including applications in various industries such as rubber, food, textiles, plastics, foundry, building materials, paper, pharmaceuticals, cosmetics, and coal. The main advantages of microwave power in processing of materials are increased rate of