

EXTRACTION OF ESSENTIAL OIL FROM
PATCHOULI LEAVES USING MICROWAVE-
ASSISTED HYDRODISTILLATION AND
HYDRODISTILLATION METHODS

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MASTER OF SCIENCE

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I hereby declare that the work in this 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 Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Penggunaan minyak pati dalam industri minyak wangi, farmaseutikal dan kosmetik semakin meningkat dalam beberapa dekad yang lalu. Kaedah konvensional sedia ada seperti penyulingan hidro (HD) dan penyulingan wap mempunyai pelbagai kelemahan seperti masa pengekstrakan yang panjang, penggunaan tenaga yang tinggi, kos operasi yang tinggi dan pelepasan sisa toksik ke alam sekitar. Dalam kajian ini, kecekapan (MAHD) daripada konvensional HD dalam pengekstrakan minyak pati dari daun Nilam (Patchouli) telah dikaji. Tiga parameter utama iaitu masa pengekstrakan, jumlah pelarut dan gelombang ketuhar mikro telah dinilai dalam pengekstrakan minyak untuk MAHD manakala jumlah pelarut dan masa pengekstrakan dikaji untuk HD menggunakan eksperimen satu faktor-di-satu-masa (OFAT). Sebagai perbandingan antara kedua-dua kaedah MAHD dan HD, MAHD menunjukkan masa pengekstrakan yang lebih singkat untuk mendapatkan hasil optimum di mana 23.33 mg/g minyak pati yang diperolehi melalui MAHD manakala 9 mg/g daripada minyak yang diperolehi melalui kaedah konvensional selama 45 minit pengekstrakan dan 360ml pelarut. Parameter yang digunakan dalam OFAT telah dilanjutkan dengan menggunakan RSM untuk dioptimumkan. Keadaan yang optimum untuk menghasilkan hasil maksimum minyak pati 61.5 mg/g nilam menggunakan MAHD adalah 440.71 W dan 383.89 ml pelarut selama 75 minit pengekstrakan manakala apabila menggunakan kaedah konvensional, hanya boleh menghasilkan maksimum 35 mg/g daripada minyak nilam untuk pengekstrakan selama 75 minit dan 300 ml pelarut adalah kurang berbanding MAHD. GC-MS, FTIR dan LC-MS / QToF analisis telah digunakan untuk mengenal pasti dan mencirikan sebatian bioaktif dalam minyak pati Patchouli. Band FTIR di 3293 cm^{-1} untuk MAHD dan HD yang berkaitan dengan kumpulan alkohol menunjukkan kandungan patchouli alkohol dalam minyak. Selain itu, 29 kompaun telah dikenal pasti dalam ekstrak MAHD manakala 27 kompaun telah dijumpai di dalam ekstrak HD. Selain itu, analisis LC-MS/QTOF menunjukkan kehadiran tanin, alkaloid fenolik, flavonoid, sesquiterpenoids lactones dan triterpenoids dalam minyak nilam. MAHD adalah kaedah yang baik dan menjanjikan untuk mengekstrak minyak pati dari tumbuhan kerana ia menggunakan tenaga yang kurang dan menghasilkan jumlah minyak nilam yang tinggi pada masa pengekstrakan lebih pendek.

ABSTRACT

The use of essential oils in perfumery, pharmaceutical and cosmetics industries are increasing in the last few decades. The existing conventional methods such as hydrodistillation (HD) and steam distillation have various disadvantages such as long extraction time, high energy use, high operational cost and release of toxic wastes to the environment. In this study, the efficiency of microwave-assisted hydrodistillation (MAHD) over conventional HD in the extraction of essential oil from Patchouli (*Pogostemon cablin*) leaves were investigated. Three major parameters namely extraction time, volume of solvent and microwave power levels were evaluated in oil extraction for MAHD while volume of solvent and extraction time were studied for HD using one-factor-at-a-time (OFAT) experiments. In comparison between both MAHD and HD methods, MAHD showed shorter extraction time to obtain optimum yield where 23.33 mg/g of essential oil obtained through MAHD while 9 mg/g of oil obtained through conventional method for 45 minutes of extraction when using 360ml of solvent. The parameters used in OFAT were further optimized using Response Surface Methodology (RSM). The optimal conditions to produce maximum yield of 61.5 mg/g patchouli essential oil using MAHD were 440.71 W and 383.89 ml of solvent for 75 minutes of extraction while when using conventional method, only can yield maximum of 35 mg/g of patchouli oil for 75 minutes of extraction and 300 ml of solvent which is less amount compared to MAHD. Gas Chromatography-Mass Spectrometry (GC-MS), Fourier transform infrared transmission (FTIR) and Liquid chromatography-mass spectrometry Quadrupole Time of Flight (LC-MS/QTOF) analyses were employed to identify and characterize the bioactive compounds in the patchouli essential oil. FTIR bands at 3293 cm^{-1} for both MAHD and HD related to alcohol group show the abundance of patchouli alcohol in the oil. Moreover, 29 compounds were identified in MAHD extract while 27 compounds were found in HD extract. Besides, LC-MS/QTOF analysis indicated the presence of tannins, phenolic alkaloids, flavonoids, sesquiterpenoids lactones and triterpenoids in the patchouli oil. MAHD is a good and promising method to extract essential oils from plants because it consumes less energy and yield high amount of patchouli oil at shorter extraction time.

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LIST OF SYMBOLS

ml	millilitre
%	Percentage
W	Watt
Min	minutes
cm ⁻¹	wavenumber
g	gram
MHz	Mega Hertz
GHz	Giga Hertz
tan δ	Dissipation factor
ϵ'	The measure of the ability to absorb microwave energy
ϵ''	Efficiency of converting microwave energy to heat energy
wt	weight
ρ	density
α	alpha
Y	Predicted yield of patchouli oil
Φ_0	Intercept term
ϕ_i	Linear coefficient
ϕ_{ii}	Squared coefficient
ϕ_{ij}	Interaction coefficient
m	metre
mm	millimetre
μm	micrometre
μL	microlitre
°C	Celsius
eV	electronvolt
keV	Kiloelectron volt
mg	milligram
g	gram
ppm	Parts-per million
s	seconds
RM	Ringgit Malaysia

LIST OF ABBREVIATIONS

MAHD	Microwave-assisted hydrodistillation
HD	Hydrodistillation
FTKKP	Faculty of Chemical and Process Engineering Technology
OFAT	One-factor-at-a-time
RSM	Response surface methodology
GC-MS	Gas chromatography-mass spectrometry
FTIR	Fourier Transform Infrared Transmission
LC-MS/QTOF	Liquid Chromatography-Mass Spectrometry Quadrupole-Time of Flight
MAE	Microwave-assisted extraction
SEM	Scanning Electron Microscopy
FDA	Food and Drug Administration
CCD	Central Composite Design
IR	Infrared
ANOVA	Analysis of variance
P-VALUE	Probability value
2D	Two dimensions
3D	Three dimensions

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