

TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENT	iii
ABSTRACT (English)	iv
ABSTRACT (Thai)	v
LIST OF TABLES	ix
LIST OF ILLUSTRATIONS	x
ABBREVIATIONS AND SYMBOLS	xiii
CHAPTER I INTRODUCTION AND LITERATURE REVIEW	1
1.1 General information on vetiver grass	1
1.1.1 Morphology of vetiver grass	2
1.1.2 Vetiver ecotypes	6
1.2 Literature reviews	12
1.3 Introduction of chromatography	18
1.3.1 Thin layer chromatography	20
1.3.2 Classical liquid chromatography	23
1.3.3 Gas chromatography	27
1.3.3.1 Sample inlet system	28
1.3.3.2 Column oven	30
1.3.3.3 Temperature-programmed analysis	31
1.3.3.4 Detectors	32
1.4 Gas chromatography-mass spectrometry	35
1.4.1 Instrumental aspects of GC-MS	36
1.4.2 Analyte ionization	37

	Page
1.4.3 Mass analysis	39
1.4.4 GC-MS interfacing	42
1.5 Solid phase microextraction and applications	44
1.5.1 Desorption	46
1.5.2 Preparation of GC	47
1.5.3 Injector temperature	47
1.5.4 Selection of fiber, sampling vial, and vial septa	48
1.6 The scope and purposes of this research	50
CHAPTER II EXPERIMENTAL	51
2.1 Apparatus chemicals and samples	51
2.2 Investigation of aroma components by SPME-GC-MS	54
2.3 Separation and purification of aroma components	60
CHAPTER III RESULTS AND DISCUSSIONS	67
3.1 Investigation of aroma components in scented vetiver root by SPME-GC-MS	68
3.1.1 At room temperature	68
3.1.2 At higher temperature	73
3.2 Isolation and purification of aroma component in scented vetiver root	80
3.2.1 Analysis of the dichloromethane crude extract by GC-MS	80
3.2.2 Separation of the aroma fraction using classical column chromatography	92

	Page
3.2.3 Separation of fraction A6 using thin layer chromatography	123
3.2.4 Separation and isolation of the aroma components by preparative gas chromatograph	126
CHAPTER IV CONCLUSION	129
REFERENCES	130
CURRICULUM VITAE	133



ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่
Copyright© by Chiang Mai University
All rights reserved

LIST OF TABLES

Table	Page
1.1 Comparing the differences between <i>Vetiveria zizanioides</i> and <i>Vetiveria nemoralis</i>	8
1.2 Samples of 28 ecotypes in Thailand	10
1.3 Stationary phase selection	22
1.4 Proton affinity of some frequently used reagent gases for CI	39
1.5 Commercial available SPME fibers for GC and GC-MS	49
2.1 Conditions of GC-MS	58
2.2 Conditions of GC-FID	62
3.1 Mass spectral data of all volatile components obtained at room temperature	71
3.2 Mass spectral data of all volatile components obtained at higher temperature	75
3.3 Mass spectral data of labeled components in dichloromethane crude extract	83
3.4 Mass spectral data of labeled components obtained by CC	101
3.5 Comparison the detail of separated components obtained from CC	116

LIST OF ILLUSTRATIONS

Figure	Page
1.1 Vetiver grass	2
1.2 Morphology of vetiver grass	5
1.3 Differential migration of three components of a mixture	19
1.4 Schematic of gas chromatograph	27
1.5 Schematic diagram of a splitter injection for capillary columns	29
1.6 Schematic diagram of a splitless injection for capillary columns	30
1.7 Schematic diagram of the essential of a thermal conductivity detector	32
1.8 A schematic diagram of a flame ionization detector	34
1.9 Geometries of double-focusing sector instrument	40
1.10 Schematic diagram of linear quadrupole instrument	42
1.11 Schematic diagram of the open split coupling for GC-MS	43
1.12 SPME fiber with holder	44
1.13 SPME headspace sampling	45
2.1 SPME sampling and analysis procedure at room temperature	56
2.2 SPME sampling and analysis procedure at higher temperature	57
2.3 Extraction separation and isolation of aroma components in scented vetiver root	65
3.1 Comparison of SPME-GC-MS chromatogram of volatile components at room temperature	69
3.2 SPMEGC-MS chromatogram of volatiles at higher temperature	74

Figure	Page
3.3 Chemical structures of some volatile components in raw scented vetiver root extracted by SPME at higher temperature	79
3.4 GC-MS chromatogram of dichloromethane crude extract of scented vetiver root	81
3.5 Expansion of GC-MS chromatogram of Fig. 3.4 and its labeled components	82
3.6 Comparison of GC-MS chromatograms of 8 separated fractions of scented vetiver root crude extract	93
3.7 Expansion of GC-MS chromatogram and its labeled components of fraction A1	95
3.8 Expansion of GC-MS chromatogram and its labeled components of fraction A2	96
3.9 Expansion of GC-MS chromatogram and its labeled components of fraction A3	97
3.10 Expansion of GC-MS chromatogram and its labeled components of fraction A4	98
3.11 Expansion of GC-MS chromatogram and its labeled components of fraction A5	98
3.12 Expansion of GC-MS chromatogram and its labeled components of fraction A6	99
3.13 Expansion of GC-MS chromatogram and its labeled components of fraction A7	99

Figure	Page
3.14 Expansion of GC-MS chromatogram and its labeled components of fraction A8	100
3.15 GC-FID chromatograms of each fraction obtained from TLC	124
3.16 GC-MS chromatogram of component C6 obtained from preparative GC	127
3.17 Mass spectrum of component C6 obtained from GC-MS	127

ABBREVIATIONS AND SYMBOLS

°C	degree celsius
μL	microlitre
μm	micrometer
amu	atomic mass unit
AR	analytical reagent
B	magnetic field
BE	reversed geometry
C ₁₄	carbon fourteen
C ₇ H ₈	toluene
CC	classical column liquid chromatography
CI	chemical ionisation
cm	centimetre
DC	direct current
E	elementary charge
EB	Nier-Johnson geometry
EI	electron impact
ESR	electrostatic analyzer
et al.	and other
EtOAc	ethyl acetate
EtOH	ethanol
eV	electron volt
F	fluorescence

FID	flame ionisation detector
g	gram
GC	gas chromatography
GLC	gas liquid chromatography
HP	high pure
HPLC	high performance liquid chromatography
HPTLC	high performance thin layer chromatography
hrs	hours
Hz	hertz
i. d.	diameter
IR	infrared
kJ/mol	kilojoule per mole
LC	liquid chromatography
m	metre
m/z	a value of mass divided by charge
m ² /g	squaremeters per gram
min	minute
mL	millilitre
mm	millimetre
MS	mass spectrometry
nm	nanometer
NMR	nuclear magnetic resonance
PA	proton affinity

PAH	polyaromatic hydrocarbon
PC	paper chromatography
PCI	positive-ion chemical ionisation
PDMS	polydimethylsiloxane
R	radius
rf	radiofrequency
s	sinister
SPME	solid phase microextraction
TCD	thermal conductivity detector
TLC	thin layer chromatography
UV	ultra violet
V	volt

ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่
Copyright© by Chiang Mai University
All rights reserved