

**“OPTIMIZATION OF HYDRO DISTILLATION CONDITIONS FOR THE  
PRODUCTION OF ESSENTIAL OIL FROM *Alpinia galanga*”**

**SHAHRIL BIN MOHAMAD**

**A thesis submitted in fulfillment of the requirements for the award of the degree  
of Bachelor of Chemical Engineering**

**Faculty of Chemical & Natural Resources Engineering  
University College of Engineering & Technology Malaysia**

**NOVEMBER 2006**

I declare that this thesis entitled “**OPTIMIZATION OF HYDRO DISTILLATION CONDITIONS FOR THE PRODUCTION OF ESSENTIAL OIL FROM *Alpinia galanga***” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : .....

Name of Candidate : Shahril bin Mohamad

Date : .....

## **DEDICATION**

Special Dedication of This Grateful Feeling to My...

*Beloved Parents;  
Mohamad bin Ismail  
Fazilah bte Abdullah*

For Their Love, Support and Best Wishes.

## ACKNOWLEDGEMENT

*Bismillahirrahmanirahim,*

I am so thankful to Allah S.W.T for giving me patience and strength throughout this project and the research is successfully completed. With the mercifulness from Allah therefore I can produce a lot of useful ideas to this project.

To my beloved father and mother, Mohamad bin Ismail and Fazilah bte Abdullah. I am grateful to have both of you in my life and giving me full support through life. I pray and wish the both of you health and in Allah's mercy always. You are the precious gift from Allah to me.

I am indebted to my supervisor, Prof. Dr Mashitah bte Mohd Yusoff for her advice, insightful comments and generous support. Thank for your guidance.

I would like to dedicate my appreciation to all the lecturers that involved in this project for their guidance and advice. Without your cooperation and sacrifices this research will not have been completed and published.

I would also like to thank my beloved sisters for their encouragement and besides giving some idea to this project. A special thanks too to all my beloved friends who have accompanied me throughout this project.

## ABSTRACT

Essential oils from the rhizomes of *Alpinia galanga* are primarily used in the perfumery industry and have a very high commercial value due to its therapeutic properties. As essential oils are composed of heat-sensitive chemical compounds, the use of conventional steam distillation technique would inevitably inflict thermal degradation to the natural fragrance. In this experimental work, hydro distillation method was employed due to its milder extracting condition and lower operating cost. Three different mesh sizes were used, named herein as powder, cubes (1cmx1cmx1cm) and slices, respectively. The extract compositions were compared using gas chromatography analysis. Besides mesh size, further studies also revealed that the composition and yield of essential oils were influenced by the different types of solvents used. The most optimum yield which is 0.79% was extracted from powder sample with n-hexane as solvent. The low yield of essential oils can be improved in future studies by carrying out the research on a larger scale.

## ABSTRAK

Kegunaan utama minyak pati daripada rizom *Alpinia galanga* ialah di dalam pembuatan minyak wangi dan ianya mempunyai nilai komersil yang tinggi disebabkan oleh ciri-ciri terapinya. Minyak pati terdiri daripada komponen yang sensitif pada haba, oleh itu penggunaan pengekstrakan wap air sebagai salah satu cara untuk mengekstrakkan minyak pati secara tidak langsung membawa kepada kesan degradasi haba terhadap bau semulajadi minyak lengkuas. Di dalam kajian ini, pengekstrakan minyak ini dilakukan menggunakan kaedah penyulingan hidro kerana ia didapati sesuai untuk tujuan pengekstrakan minyak ini dan kos menggunakan cara ini lebih rendah. Tiga jenis saiz yang berlainan digunakan iaitu serbuk, dadu dan keping. Sampel minyak pati yang didapati daripada kajian ini akan dibandingkan menggunakan kaedah analisis gas kromatografi. Selain daripada faktor saiz, kajian berikutnya membuktikan pengesanan komponen dan kuantiti minyak ini adalah dipengaruhi oleh faktor utamanya iaitu penggunaan pelarut-pelarut yang berlainan. Hasil minyak pati yang paling optimum iaitu 0.79% diekstrak menggunakan sampel serbuk dengan n-heksan sebagai pelarut. Hasil minyak lengkuas yang rendah ini dapat dipertingkatkan pada kajian akan datang menggunakan skala yang lebih besar.

## TABLE OF CONTENT

CHAPTER	TITLE	PAGE
	TITLE PAGE	i
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENT	iv
	ABSTRACT (ENGLISH)	v
	ABSTRAK (BAHASA MELAYU)	vi
	TABLE OF CONTENT	vii
	LIST OF TABLES	xiii
	LIST OF FIGURES	xiv
	LIST OF SYMBOLS	xv
	LIST OF APPENDICES	xvi
<b>1</b>	<b>INTRODUCTION</b>	<b>1</b>
	1.1 Introduction	1
	1.2 Problem Statement	2
	1.3 Objectives	3
	1.4 Scopes of Work	3
<b>2</b>	<b>LITERATURE REVIEW</b>	<b>4</b>
	2.1 <i>Alpinia galanga</i>	4
	2.1.1 History	4
	2.1.2 Description	6

2.1.3	Morphology	6
2.1.4	Background Information	6
2.1.5	Product of <i>Alpinia galanga</i>	7
2.1.5.1	Essential Oil	7
2.1.5.2	Uses	8
2.2	Separation Process Principle	9
2.2.1	Classification of Separation Process	10
2.2.2	Evaporation	12
2.2.2.1	Processing Factors	12
2.2.3	Boiling and Condensation	15
2.2.3.1	Boiling	15
2.2.3.2	Condensation	15
2.2.3.3	Condensation of Mixed Vapors	16
2.2.4	Distillation	16
2.2.4.1	Distillation of the Oil	18
2.2.4.2	Principle of Hydro Distillation	18
2.2.4.2.1	Hydro Distillation Methods	19
2.2.5	Extraction	20
2.2.5.1	Liquid Extraction	20
2.2.5.2	Leaching	21
2.2.5.3	Factors Influencing Yield	22
2.3	Analysis	23
2.3.1	Introduction	24
2.3.2	Gas Chromatography	24
2.3.2.1	Practical Aspects	24
2.3.2.2	Principle of Process	24
2.3.2.3	Basic Components of a GC	26
2.3.2.3.1	Injectors	26
2.3.2.3.2	Carrier Gas	27
2.3.2.3.3	Column	27
2.3.2.3.4	Oven	28



	2.3.2.3.5 Detector	29
	2.3.2.3.6 Recorder	30
	2.3.2.4 Samples and Samples Preparation	30
<b>3</b>	<b>METHODOLOGY</b>	<b>32</b>
	3.1 Introduction	32
	3.2 Plant Material	32
	3.2 Sample Preparation	33
	3.3 Isolation of Essential Oils	34
	3.5 Analysis of Essential Oils	35
	3.6 Yield Calculation	36
<b>4</b>	<b>RESULT AND DISCUSSION</b>	<b>37</b>
	4.1 Yield of Essential Oil	37
	4.2 Determination of Oil Quality	38
	4.2.1 Chemical Composition of Essential Oils	38
<b>5</b>	<b>CONCLUSION AND RECOMMENDATIONS</b>	<b>39</b>
	5.1 Conclusion	39
	5.2 Recommendation	40
	<b>REFERENCES</b>	<b>42</b>

**LIST OF TABLES**

<b>TABLE NO</b>	<b>TITLE</b>	<b>PAGE</b>
<b>2.1</b>	Taxonomy of <i>Alpinia galanga</i>	7
<b>2.2</b>	Comparison of Similarities between Extraction and Distillation	11
<b>3.1</b>	Experimental Condition	35
<b>4.1</b>	Yield of Essential Oils	37

**LIST OF FIGURES**

<b>NO</b>	<b>TITLE</b>	<b>PAGE</b>
<b>2.1</b>	<i>Alpinia galanga</i>	5
<b>2.2</b>	Leaves	5
<b>2.3</b>	Flowers	5
<b>2.4</b>	Rhizomes	5
<b>2.5</b>	Rhizomes	5
<b>2.6</b>	Chemical Structure of 1,8-cineol	9
<b>2.7</b>	Simple Distillation	18
<b>2.8</b>	Gas Chromatography System	27
<b>3.0</b>	Powdered Sample	33
<b>3.1</b>	Cubed Sample	33
<b>3.2</b>	Sliced Sample	33

**LIST OF SYMBOLS**

g	-	gram
ml	-	mililiter
L	-	liter
kg	-	kilogram
°C	-	Celcius
%	-	percent

**LIST OF APPENDICES**

<b>LIST</b>	<b>PAGE</b>
Appendix A	43
Appendix B	52

## CHAPTER 1

### INTRODUCTION

#### 1.1 Introduction

The genus *Alpinia* belongs to the family *Zingiberaceae* with *Alpinia galanga* Willd.(syn. *Languas galanga* Stunz and *Alpinia officinarum* Hance syn. *Languas officinarum* Hance) as the most important species. *Alpinia galanga* locally known as *Lengkuas* but generally called greater galangal is a rhizomatous herb distributed in various parts of India and throughout South East Asia. It is widely cultivated in small garden plots on rather wet ground in Malaysia, India, Indo China, Indonesia, and the Philippines.

The plant grows to a height of 1.8 m and has long, blade-like leaves. The flowers are green and white with red tips. Rhizomes is built up from cylindrical subunits (circular cross-section), whose pale-reddish surface is characteristically cross-striped by reddish-brown, small rings.

The rhizome most importantly is the useful plant part and it is warm, sweet, and spicy plus, it is high in starch. The fresh or dried rhizome is often used as a spice or flavoring agent in native dishes and as medicament or part of medicaments in Asian folk medicine for various applications, such as post-partum protective medicine, for the treatment of stomachache, diarrhea, dysentery, and sinus.

An essential oil can be defined as the volatile material present in plants. It consists of a complex mixture of terpenes, oxygenated terpenes, sesquiterpenes and oxygenated sesquiterpenes. From previous study, the essential oil from the rhizomes of *Alpinia galanga* made up only 1 to 1.5% of its dry weight. In addition, the main constituents of galangal oil are 1,8-cineole,  $\alpha$ -pinene, eugenol, camphor, methyl cinnamate, sesquiterpenes,  $\alpha$ -bergamotene, trans- $\beta$ -farnesene and  $\beta$ -bisabolene.

In this experiment, the analytical procedure for the essential oils of *Alpinia galanga* comprises two steps; extraction by using hydro distillation and analysis by using Gas Chromatography with Flame Ionization Detector (GC-FID). Further, the GC-FID condition was also being optimized by setting the right temperature for column and detector and pressure for carrier gas.

The aim of the present work is to optimize the hydro distillation condition for the production of essential oil from *Alpinia galanga* by manipulating the distillation temperature and the surface area of the samples of rhizomes.

## 1.2 Problem Statement

Previous studies have reported that the marker compound for the essential oil from *Alpinia galanga* is 1,8-cineole. This compound constitutes about 40 to 47% of the yields. The problem occurs when obtaining the yields of the essential oil. The concentration of the cineole seems to change when using different methods of distillation. In steam distillation method, cineole constitutes about 28 to 29% of the yields while in hydro distillation, cineole constitute about 40 to 41% which is much greater than the 18% using steam distillation.

These differences between the methods used maybe due to the temperature setting and the duration of the raw materials' exposure to the heat during distillation in

each method. For steam distillation, the raw material is directly heated by steam whereby the temperature is greater than 100°C for about 3 to 4 hours. However, for hydro distillation, the raw material is immersed in water and being heated at 80°C or lower for about 5 to 8 hours.

### **1.3 Objectives**

The objectives of this study are to determine optimal conditions for the distillation of essential oil from *Alpinia galanga* by hydro distillation and to determine the quality of the oil by using GC-FID.

### **1.4 Scope of Work**

In order to achieve the objective of this research, the scope of work has been identified as follows:

- i. Optimizing the hydro distillation conditions for the improvement of yields based on temperature, surface area of samples of rhizomes from *Alpinia galanga* during the distillation of essential oils.
- ii. Optimization of GC-FID conditions, such as column temperature, detector temperature, and carrier gas pressure for the determination of the quality of essential oil.



## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 *Alpinia galanga*

##### 2.1.1 History

*Alpinia galanga* (known as greater galangal) was described by Hance, in the *Journal of the Linnéan Society* (1871). The plant was obtained from Hainan, an island directly south of China, but it is also found to grow on the adjacent mainland, as the root is largely exported from Shanghai and other Chinese ports. *Alpinia galanga* grows in Java. The name galangal is said to be derived from the Arabic *Khanlanjan*, which, in turn, is perhaps the perversion of a Chinese word, signifying mild ginger. Galangal has long been an article of commerce with the Eastern nations, and has been known in Northern Europe since the twelfth century. Figure 2.1 shows the *Alpinia galanga* plant. Figures 2.2 and 2.3 depicts the leaves and flowers of the plant, while figures 2.4 and 2.5 depicts rhizomes of the plant.



**Figure 2.1** *Alpinia galanga*



**Figure 2.2 and Figure 2.3** Leaves and Flowers



**Figure 2.4 and Figure 2.5** Rhizomes

### 2.1.2 Description

Greater galangal, is a very popular flavouring agent in whole South East Asia cuisine and particular that of Thailand. In the Middle Ages, it was used as a medicine, spice and an aphrodisiac. Its origin is in South East Asia, probably southern China; it is now cultivated in India, Indochina, Thailand, Malaysia and Indonesia. The ginger-like rootstock (rhizome) is the useful plant part. It is warm, sweet and spicy. Fresh galangal has a distinct fragrance and the dried galangal is more spicy and sweet-aromatic, almost like cinnamon.

### 42.1.4 Morphology

Greater galangal grows to a height of 1.8 m and has long, elegant, blade-like leaves. The flowers are green and white with red tips. Rhizome is built up from cylindrical subunits (circular cross-section), whose pale-reddish surface is characteristically cross-stripped by reddish-brown, small rings. The interior has about the same colour as the skin and is hard and woody in texture.

### 2.1.4 Background Information

Greater galangal is scientifically known as *Alpinia galanga*. The genus name *Alpinia* is in memory of an Italian Botanist, Prospero Alpina (1533-1617), and the alternative (younger) genus name *Languas* is based on Malay *lengkuas* “galanga”, which in turn relates to the former mentioned Chinese *liang-jiang* (Southern Chinese form *liang-kiang*).

**Table 2.1 Taxonomy of *Alpinia galanga***

Scientific Classification	
Kingdom	<i>Plantae</i>
Division	<i>Magnoliophyta</i>
Class	<i>Liliopsida</i>
Order	<i>Zingiberales</i>
Family	<i>Zingiberaceae</i>
Genus	<i>Alpinia</i>
Species	<i>galanga</i>

### **2.1.5 Product of *Alpinia galanga***

There is one value added product of *Alpinia galanga* which is the essential oil. The product is distinguished by the techniques it been derived, it appearances, odour, and flavour.

#### **2.1.5.1 Essential Oil of *Alpinia galanga* (Galangal Oil)**

Galangal oil is produced from fresh or dried rhizomes of *Alpinia galanga*. The rhizome contains up to 1.5% essential oil (1,8 cineol, alpha-pinene, eugenol, camphor, methyl cinnamate and sesquiterpenes). In dried galangal, the essential oil has it quantitatively different composition than in the fresh one. Whereas alpha-pinene, 1,8-cineol, alpha-bergamotene, trans-beta-farnesene and beta-bisabolene seem to contribute to the taste of fresh galangal equally, the dried rhizome shows lesser variety in aroma components (cineol and farnesene, mostly). The resin causing the pungent taste

(formerly called galangol or alpinol) consists of several di-arylheptanoids and phenylalkanones. Furthermore, the rhizome is high in starch.

There are several techniques used to obtain galangal oil. The most popular techniques are hydro distillation and steam distillation.

#### **2.1.5.2 Uses**

The rhizomes have been used as flavors in native dishes and ingredients in many traditional medicines to treat various ailments that are listed below:

##### ***i. Malay tradition***

In Malay tradition galangal is used to treat a lot of disease such as stomachache, diarrhea, dysentery, and sinus. It is also used as post-partum protective medicine.

##### ***ii. Chinese medicine***

In traditional Chinese herbal medicine, galangal is a warming herb used for abdominal pain, vomiting, and hiccups, as well as for diarrhea due to internal cold. When used for hiccups, galangal is combined with codonopsis and Ju ling.

##### ***iii. Indian tradition***

In India and southwestern Asia, galangal is considered stomachic, anti-inflammatory, expectorant, and a nervine tonic. Galangal is used in the treatment of hiccups, dyspepsia, stomach pain, rheumatoid arthritis, and intermittent fever.

##### ***iv. Western herbalism***

Galangal was introduced into Europe by Arabian physicians well over a thousand years ago. In line with the Chinese and Indian herbal traditions, galangal is mainly

used in the West for gas, indigestion, vomiting, and stomach pain. An infusion can be used to alleviate painful canker sores and sore gums. Galangal has long been recommended as a treatment for seasickness, which is not surprising given the well-established ability of its relative ginger to relieve motion sickness.

**v. *Candidiasis***

Galangal can be used with other antifungal herbs as part of a regimen to treat intestinal candidiasis.

**vi. *Dosage***

At a moderate dosage, galangal is a warming and gently stimulating herb for a weakened digestive system, but at a higher dosage it can be an irritant.

**vii. *Other medical uses***

Altitude sickness.

## **2.2 Separation Processes Principles**

Separation processes is defined as any set of operations that separate solutions of two or more components into two or more products that differ in composition (Noble & Terry, 2004). Separation is achieved by exploiting the differences between chemical and physical properties of the substance through the use of a separating agent (mass or energy). There are three primary functions of separation processes:

**i. *Purification***

In purification, undesired components in a feed mixture are removed from the desired species.

**ii. Concentration**

In concentration, a higher proportion of desired components that are initially dilute in a feed stream can be obtained.

**iii. Fractionation**

In fractionation, a feed stream of two or more components is segregated into product streams of different components, typically pure streams of each component.

### **2.2.1 Classification of Separation Processes**

Separation processes deal mainly with the transfer and change of energy and the transfer and change of materials, primarily by physical means but also by physical-chemical means. Important separation process, which can be combined in various sequences in a process are:

**1) Evaporation**

This refers to the evaporation of a volatile solvent such as water from a nonvolatile solute such as salt or any other material in solution.

**2) Drying**

In this operation volatile liquids, usually water, are removed from solid material.

**3) Distillation**

This is an operation whereby components of a liquid mixture are separated by boiling because of their differences in vapor pressure.

**4) Absorption**

In this process a component is removed from a gas stream by treatment with a liquid.

**5) Membrane separation**

This process involves the separation of a solute from a fluid by diffusion of this solute from a liquid or gas through a semipermeable membrane barrier to another fluid.

**6) Liquid-liquid extraction**

In this case a solute in a liquid solution is removed by contacting with another liquid solvent that is relatively immiscible with the solution.

**7) Adsorption**

In this process a component of a gas or liquid stream is removed and adsorbed by a solid adsorbent.

**8) Ion exchange**

Certain ions in solution are removed from a liquid by ion-exchange solid.

**9) Liquid-solid leaching**

This involves treating a finely divided solid with a liquid that dissolves out and removes a solute contained in the solid.

**10) Crystallization**

This concerns the removal of a solute such as a salt from a solution by precipitating the solute from the solution.

**11) Mechanical-physical separation**

These involve separation of solids, liquids, or gases by mechanical means, such as filtration, settling, centrifugation, and size reduction.



## 2.2.2 Evaporation

The objective of evaporation is to concentrate a solution consisting of a non-volatile solute and a volatile solvent. In the overwhelming majority of evaporations the solvent is water. Evaporation is conducted by vaporizing a portion of the solvent to produce a concentrated solution of thick liquor. Evaporation differs from drying in that the residue is a liquid, sometimes a highly viscous one, rather than a solid; it differs from distillation in that the vapor usually is a single component, and even when the vapor is a mixture, no attempt is made in the evaporation step to separate the vapor into fraction; it differs from crystallization in that emphasis is placed on concentrating a solution rather than forming and building crystals. In certain situation, for example, in the evaporation of brine produce a common salt, the line between evaporation and crystallization is far from sharp. Evaporation sometimes produces a slurry of crystal in a saturated mother liquor.

Normally, in evaporation the thick liquor is the valuable product, and the vapor is condensed and discarded. In one specific situation, however the reverse is true. Mineral-bearing water often is evaporated to give a solid-free product for the boiler feed, for special process requirements, or for human consumption. This technique is often called *water distillation*, but technically it is evaporation. Large-scale evaporation processes have been developed and used for recovering potable water from seawater. Here the condensed water is the desired product. Only a fraction of the total water in the feed is recovered, the remainder is returned to the sea.

### 2.2.3.1 Processing Factors

The physical and chemical properties of the solution being concentrated and of the vapor being removed bear greatly on the type of evaporator used and the pressure

and temperature of the process. Some of the properties which affect the processing methods are:

*i. Liquid characteristic*

The practical solution of an evaporation problem is profoundly affected by the character of the liquor to be concentrated. It is the wide variation in liquor characteristics (which demands judgment and experience in designing and operating evaporators) that broadens this operation from simple heat transfer to a separate art. Some of the most important properties of evaporating liquids are as follows.

*ii. Concentration in the liquid*

Usually, the liquid feed to an evaporator is relatively dilute, so its viscosity is low, similar to that of water, and relatively high heat-transfer coefficients are obtained. As evaporation proceeds, the solution may become very concentrated and quite viscous, causing the heat-transfer coefficient to drop markedly. Adequate circulation and/or turbulence must be present to keep the coefficient from becoming too low.

*iii. Solubility*

As solutions are heated and the concentration of the solute or salt increases, the solubility limit of the material in solution may be exceeded and crystal form. This may limit the maximum concentration in solution which can be obtained by evaporation. In most cases the solubility of the salt increases with temperature. This means that when a hot, concentrated solution from an evaporator is cooled to room temperature, crystallization may occur.

*iv. Temperature sensitivity of material*

Many products, especially food and other biological materials, may be temperature-sensitive and degrade at higher temperature or after prolonged heating. Such products include pharmaceutical products; food products such as milk, orange juice,

and vegetable extract; and fine organic chemicals. The amount of degradation is a function of the temperature and the length of time.

**v. *Foaming or frothing***

In some cases materials composed of caustic solutions, food solutions such as skim milk, and some fatty-acid solutions form a foam or froth during boiling. This foam accompanies the vapor coming out the evaporator and entrainment losses occur.

**vi. *Pressure and temperature***

The boiling point of the solution is related to the pressure of the system. The higher the operating pressure of the evaporator, the higher the temperature at boiling. Also, as the concentration of the dissolved material in solution increases by evaporation, the temperature of boiling point may rise. This phenomenon is called *boiling-point rise* or *elevation*. To keep the temperature low in heat-sensitive materials, it is often necessary to operate under 1 atm pressure, that is, under vacuum.

**vii. *Scale deposition and materials of construction***

Some solutions deposit solid materials called *scale* on the heating surfaces. These could be formed by decomposition products or by decreases in solubility. The result is that the overall heat-transfer coefficient decreases and the evaporator must eventually be cleaned. The materials used in construction of the evaporator should be chosen to minimize corrosion.

## **2.3.2 Boiling and Condensation**

### **2.3.3.1 Boiling**

Heat transfer to a boiling liquid is very important in evaporation and distillation as well as in other kind of chemical and biological processing, such as petroleum processing, control of the temperature of chemical reactions, evaporation of liquid foods, and so on. The boiling point is usually contained in a vessel with a heating surface of tubes or vertical or horizontal plates which supply the heat for boiling. The heating surfaces can be heated by electrically or by a hot or condensing fluid on the other side of the heated surface.

In boiling, the temperature of the liquid is the boiling point of this liquid at the pressure in the equipment. The heated surface is, of course, at a temperature above the boiling point. Bubbles of vapor are generated at the heated surface and rise through the mass of liquid. The vapor accumulates in a vapor above the liquid level and is withdrawn.

### **2.3.3.2 Condensation**

Condensation of a vapor to a liquid and vaporization liquid to a vapor both involve a change of phase of a fluid with large heat-transfer-coefficients. Condensation occurs when a saturated vapor such as steam comes in contact with a solid whose surface temperature is below the saturation temperature, to form a liquid such as water. Normally, when a vapor condenses on a surface such as vertical or horizontal tube or other surface, a film of condensate is formed on the surface and flows over the surface by the action of gravity. It is this film of liquid between the surface and the vapor that forms the main resistance to heat transfer. This is called film-type condensation.

Another type of condensation, dropwise condensation, can occur, where small drops are formed on the surface. These drops grow and coalesce, and the liquid flows from the surface. During this condensation, large areas of tube are devoid of any liquid and are exposed directly to the vapor. Very high rates of heat transfer occur on these bare areas.

Dropwise condensation occurs on contaminated surfaces and when impurities are present. Film-type condensation is more dependable and more common. Hence, for normal design purpose, film-type condensation is assumed.

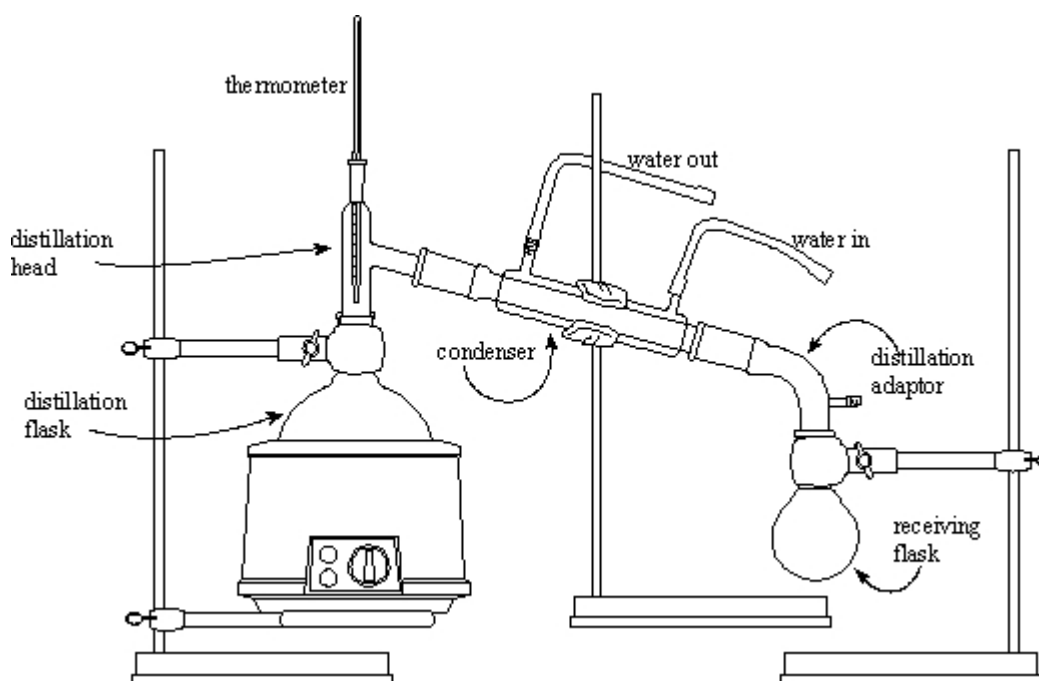
### **2.3.3.3 Condensation of mixed vapors**

If the vapor contains two or more volatile components (unless it is azeotropic mixture), the condensation temperature is no longer constant at a given pressure. Concentration gradients exist in both the vapor and liquid phases as the higher-boiling-point component or components tend to condense, enriching the vapor in lower-boiling-point material. If the coolant temperature is low enough, all the vapor may eventually be condensed; the composition of the condensate will then be the same as that of the original vapor. In other cases some of the low-boiling-point material may not be condensed and must be vented from the condenser.

### **2.3.4 Distillation**

The separation process known as distillation is a method for separating the various components of a liquid solution which depends upon the distribution of these components between a vapor phase and a liquid phase. All components are present in both phases. The vapor phase is created from the liquid phase by vaporization at the boiling point.

The basic requirement for the separation of components by distillation is that the composition of the vapor be different from the composition of the liquid with which it is in equilibrium at the boiling point of the liquid. Distillation is concerned with solutions where all components are appreciably volatile, such as ammonia-water or ethanol-water solution, where both components will be in the vapor phase. In evaporation, by contrast, of solution of salt and water, for example the water is vaporized but the salt is not. The process of absorption differs from distillation in that one of the components in absorption is essentially insoluble in the liquid phase. An example is absorption of ammonia from air by water, where air is insoluble in the water-ammonia solution.



**Figure 2.7 Simple distillation**

#### **2.3.4.1 Distillation of the Oil**

Essential oils are the volatile organic constituents of fragrant plant matter. They are generally composed of a number of compounds, including some that are solids at normal temperatures, possessing different chemical and physical properties. The aroma profile of the oil is a cumulative contribution from the individual compounds. The boiling points of most of these compounds range from 150 to 300°C at atmospheric pressure. If heated to this temperature, labile substances would be destroyed and strong resinification would occur. Hydro distillation permits the safe recovery of these heat-sensitive compounds from the plant matter.

Hydro distillation involves the use of water or steam to recover volatile principles from plant materials. The fundamental feature of hydro distillation is that it enables a compound or mixture of compounds to be distilled and subsequently recovered at a temperature substantially below of the boiling point of the individual constituents.

#### **2.3.4.2 Principle of Hydro distillation**

During hydro distillation, water and essential oil form a heterogeneous system of immiscible liquids. By the principle of distillation mutually immiscible liquids, the total vapor pressure of the mixture at its boiling point will be equal to the sum of their partial vapor pressures. Hence, the vapor pressure exerted by each component is less than its vapor pressure if present alone at its boiling point. Therefore, the boiling temperature for any two-phase liquid will always be lower than the boiling point of either of the pure liquids at the same total pressure. Thus, in the case of an essential oil, the constituent compounds distill at temperatures below 100°C when boiled with water at atmospheric pressure.

### **2.3.4.2.1 Hydro distillation Methods**

Hydro distillation of plant material may be carried out by the one of the following techniques (Guenther, 1972): Water distillation, water and steam distillation, and steam distillation.

#### ***1. Water distillation***

In this method, the raw material to be distilled is charged in the still. Water is added to immerse the charge, leaving sufficient vapor space. The quantity of water should be adequate for the material to move freely in boiling water, thus avoiding, localized over-heating and subsequent charring of the material. The water is boiled under direct fire or by steam jacket or closed steam coil. It may be necessary to add more water as the distillation proceeds to prevent any dry material from being exposed to direct heating. The vapor is condensed and the oil is separated from water, taking advantage of their mutual immiscibility and difference in specific gravity. This method is normally used where the raw materials tend to agglutinate and form a large compact lumps through which steam cannot penetrate.

#### ***2. Water and Steam Distillation***

Here the plant material is supported on a perforated grid inside the still. The lower part of the still is filled with water to a level below the grid. The water is heated to generate steam. The steam usually wet and at low pressure, rises through the charge carrying the essential oil. The advantage of this method over water distillation is that the raw material is not in contact with boiling water. The exhausted plant material can be handled easily as it does not form slurry with water.

#### ***3. Steam distillation***

This is the most widely used industrial method for the isolation of the essential oil from plant material. Here the steam is produced outside the still, usually in a steam boiler. Steam at optimum pressure is introduced into the still below the charge



through a perforated coil or jets. Steam distillation is relatively rapid and is capable of greater control by the operator. The steam pressure inside the still could be progressively increased as distillation proceeds for complete recovery of high-boiling constituents. The still can be emptied and recharge quickly. With the immediate reintroduction of steam, there is no unnecessary delay in the commencement of the distillation process. Oils produced by this method are of more acceptable quality than those produced by other methods.

In steam distillation, the raw material is grinded and charged in still of optimum dimensions. The still is attached to a heat exchanger (condenser) and a separator. Direct steam admitted from the bottom of the still. The steam, which rises through the charge, carries along with the vapors of the volatile oil. The oil vapor-steam mixture is cooled in the condenser. The oil is separated from the water in the separator and collected in a glass or stainless steel bottles. The oil thoroughly dried and stored airtight in full containers in a cool dry place protected from light.

### **2.3.5 Extraction**

#### **2.3.5.1 Liquid extraction**

In liquid extraction, sometimes called solvent extraction, a mixture of two components is treated by a solvent that preferentially dissolves one or more of the components in the mixture. The mixture so treated is called *raffinate*, and the solvent-rich phase is called extract. The component transferred from *raffinate* is the *diluent*. The solvent in the extract leaving the extractor usually recovered and reused. There are, oddly enough, a number of such similarities between extraction and distillation;

**Table 2.2 Comparison of Similarities between Extraction and Distillation**

<b>Extraction</b>	<b>Distillation</b>
Addition of solvent	Addition of heat
Solvent mixer	Reboiler
Removal of solvent	Removal of heat
Solvent separator	Condenser
Solvent-rich solution saturated with solvent	Vapor at the boiling point
Solvent-rich solution containing more solvent than the required to saturate it	Superheated vapor
Solvent-lean solution containing less solvent than that required to saturate it	Liquid below the boiling point
Solvent-lean solution saturated with solvent	Liquid at the boiling point
Two-phase liquid mixture	Mixture of liquid and vapor
Selectivity	Relative volatility
Change of temperature	Change of pressure

### 2.3.5.2 Leaching

Many biological and inorganic and organic substances occur in a mixture of different components in a solid. In order to separate the desired solute constituents or remove an undesirable solute component from the solid phase, the solid is contacted with a liquid phase. The two phases are in intimate contact and the solutes can diffuse from the solid to liquid phase, resulting in a separation of the components originally in the solid. This separation process is called *liquid-solid leaching* or simply *leaching*. In leaching, when an undesirable component is removed from a solid with water, the process is called *washing*.

### 2.3.5.3 Factors influencing the rate of extraction

The selection of the equipment for an extraction process is influenced by the factors which are responsible for limiting the extraction rate. There are four important factors to be considered:

*i. Particle size*

Particle size influences the extraction rate in a number of ways. The smaller the size, the greater is the interfacial area between the solid and liquid, and therefore the higher is the rate of transfer of material and the smaller is the distance the solute must diffuse within the solid as already indicated.

*ii. Solvent*

The liquid chosen should be a good selective solvent and its viscosity should be sufficiently low for it to circulate freely. Generally, a relatively pure solvent will be used initially, although as the extraction proceeds the concentration of solute will increase and the rate of extraction will progressively decrease, first because of the concentration gradient will be reduced, and secondly because the solution will generally become more viscous.

*iii. Temperature*

In most cases, the solubility of the material which is being extracted will increase with temperature to give a higher rate of extraction. Further, the diffusion coefficient will be expected to increase with rise in temperature and this will also improve the rate of extraction.

*iv. Agitation of the fluid*

Agitation of the solvent is important because this increases the eddy diffusion and therefore the transfer of material from the surface of the particles to the bulk of the

solution. Further, agitation of suspension of fine particle prevents sedimentation and more effective use is made of the interfacial surface.

## **2.4 Analysis**

### **2.4.1 Introduction**

There are different methods to analyze volatile components in essential oils. The usual method is by using chromatography. Chromatography is an analytical technique based on the separation of molecules due to differences in their structure and/or composition. Chromatography involves moving a sample through the system to be separated into its various components over a stationary phase. The molecules in the sample will have different interactions with the stationary support, leading to separation of similar molecules. Chromatographic separations can be divided into several categories based on the mobile and stationary phases used, including thin-layer chromatography, gas Chromatography (GC), paper chromatography and high-performance liquid chromatography (HPLC).

### **2.4.2 Gas Chromatography**

GC is a physical separation technique in which components of a mixture are separated using a mobile phase of inert carrier gas and a solid or liquid stationary phase contained in a column. The separation is based on the interactions of the vaporized components in a mixture with the stationary phase as they are moved along by the mobile phase.

Since GC is a gas-based separation technique, it is limited to components that have sufficient volatility and thermal stability.

#### **2.4.2.1 Practical Aspects of Gas Chromatography Theory**

To understand GC and effectively use its practical applications, a grasp of some basic concepts of general Chromatographic theory is necessary. Chromatographic principles, including retention, resolution, sensitivity and other factors, are important for all types of Chromatographic separation. A compound is vaporized, introduced into the carrier gas and then carried onto the column. The sample is then partitioned between the gas and the stationary phase. The compounds in a sample are slowed down to varying degrees due to the sorption and desorption on the stationary phase. The elution of the compound is characterized by the partition ratio  $kD'$ , which is a dimensionless quantity also called the capacity factor. The partition ratio can also be thought of as the ratio of the time required for the compound to flow through the column (the retention time) to the elution time of an unretained compound. The value of the capacity factor is dependent on several elements of the Chromatographic system, including the chemical nature of the compound; the nature, amount and surface area of the stationary phase; the column temperature; and the gas flow rate. Capacity factor is essential for separation by GC because separation is only possible if the compounds in the sample have different capacity factors.

#### **2.4.2.2 Principle of Process**

In all types of chromatographies, a mixture is separated by distributing the components between a stationary phase and a mobile phase. The mixture is initially placed on the stationary phase (a solid or a liquid) and then the mobile phase (a gas or a liquid) is allowed to pass through the system. Efficient separation of compounds in GC