

**OPTIMIZATION OF ESSENTIAL OIL EXTRACTION FROM
*ZINGIBER OFFICINALE***

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

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OCTOBER 2006

I declare that this thesis entitled “Optimization of Essential oil extraction from *Zingiber officinale*” 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.

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Date : 18 October 2006

DEDICATION

Special dedicated of my grateful feelings to my parents, Mr. Amiril Bin Sayuti & Maimunah Ahmad for their love and encouragement. I also dedicated this to my loving sisters. Lastly not forget to thank a special person for me, Mohd Harry Bin Ramle who understand and help me in everything I do.

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ABSTRACT

The production of essential oils from natural sources is a highly profitable nowadays. The herbaceous perennial of cooking ginger has been used in culinary uses and home remedies since ancient time. Cooking ginger is also known scientifically as *Zingiber Officinale spp.* The extraction process of ginger is conducted by undergoes the sample of rhizome which part of the plant contains the active compound of essential oil and pungent compound using hydrodistillation method. The components of the essential oil will be decomposed and will create new components that is unidentified if the extraction process undergoes in high temperature. Hence this study is focused to optimize the condition of hydrodistillation which are the size of sample and time of experiment conducted to extract the essential oil from ginger to yield better essential oil without decompose its components. Then the essential oil obtained will be diluted in n-hexane before analyzed it by Gas Chromatography using the Flame Ionization Detector, (GC-FID). The result obtained from the analysis will be compared between the data gained from the analysis of the reference compound of cineol and compare the result between the data obtained with the reference data of cineol and hydrocarbon. Qualitative analysis shows the powder size yields more essential oil than slices size while quantitative analysis shows the samples of Eoil1 and Eoil2 did yield the marker compound of 1, 8-cineol.

ABSTRAK

Pengeluaran minyak pati daripada sumber semulajadi pada masa kini menjanjikan pulangan yang lumayan kepada para pengusaha. Tumbuhan herba yang hidup meliar seperti halia telah menjadi bahan ramuan dalam masakan dan ubat-ubatan tradisional sejak zaman dahulu lagi. Halia secara saintifiknya dikenali sebagai *Zingiber officinale spp.* Proses pengekstrakan rizom iaitu bahagian tumbuhan halia yang mengandungi sebatian bahan kimia yang aktif yang mudah meruap iaitu minyak pati halia dan sebatian bahan kimia yang mempunyai rasa atau bau yang tajam boleh dijalankan menggunakan kaedah hydrodistillation. Minyak pati ini adalah begitu peka terhadap perubahan suhu. Komponen-komponen yang terkandung di dalam minyak pati ini akan mengurai dan berubah menjadi komponen-komponen yang lain yang tidak dapat dikenalpasti jika proses pengekstrakan ini dijalankan pada suhu yang tinggi. Dengan itu, kajian ini dijalankan adalah untuk mencapai kondisi 'hydrodistillation' yang sesuai berdasarkan saiz sampel dan masa untuk menjalankan pengekstrakan minyak pati daripada halia ini tanpa menguraikan komponen-komponennya. Seterusnya minyak pati yang diperolehi ini akan dicairkan bersama bahan kimia n-hexane sebelum dianalisis menggunakan 'Gas Chromatography' dilengkapi oleh 'Flame Ionization Detector', (GC-FID). Data yang diperolehi daripada analisis tadi akan dibandingkan antara data yang didapati daripada analisis komponen rujukan seperti cineol dan hidrokarbon. Daripada analisis kualitatif, pengekstrakan menggunakan sampel saiz sebuk mengeluarkan minyak pati lebih banyak daripada sampel saiz yang dihiris. Manakala analisis kuantitatif menunjukkan sampel Eoil1 dan Eoil2 berjaya mengekstrak kompaun rujukan 1, 8-cineol.

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LIST OF ABBREVIATIONS AND SYMBOLS

cm	Centimeter
m	Meter
μL	Micro liter
g	Gram
°C	Degree Celcius
%	Percentage
-	No
GC	Gas Chromatography
KI	Kovat Indices
FID	Flame Ionization Detector
α	Alpha
β	Beta
δ	Sigma

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Ginger is a tropical herbaceous perennial. It grows from an aromatic tuberlike rhizome (underground stem) which is warty and branched. It has upright stems and narrow medium green leaves arranged in two on each stem which plant can grows about 1.2m tall. The stem is surrounded by the sheathing bases of two rank leaves. The leaves are 1.9cm wide and 17.8cm long (Skinner, D, 2003). The flower either white or yellowish-green and is rarely seen. It grows well in humid climate where it can absorb more sunlight.



Figure 1.0 Ginger plant (Yamasaki, K, 2003)

1.1.1 Origin and Current Location of Ginger

Ginger is originated in the tropical Asia. However, nowadays it has been cultivated as a commercial crop in Latin America, Africa as well as South East Asia. Fifty percent of the worldwide ginger production is in India but the best quality gingers come from Jamaica. The most important factor in dried ginger production is to control the level of pungency, aroma, and flavor of its final product.

1.1.2 Usage of Ginger

For thousand of years, ginger has been widely used as a culinary herb, condiment, spice, home remedy and medical agent. Extraction of ginger have been extensively studied for several biological activities including antibacterial, anti convulsion, analgesic, antiulcer, gastric antisecretory, and antitumor. Ginger was also reported by Mowrey and Clayson (1982) that it gave an excellent result in treating nausea. A study by Bone (1990) showed that acetone and methanol extract of ginger strongly inhibits gastric ulceration.

1.2 Problem Statement

The use of volatile plant oil, including essential oils, for psychological and physical well being has dated back thousand of years in China and Egypt. With such a long history, it came as a complete surprise that only during the earlier part of the twentieth century; a French Chemist by the name of Rene-Maurice Gattefosse discovered the potential medicinal use of essential oil.

Many researches have been done to discover the use of ginger in various fields, especially in the medical sector. The pungent components in ginger, specifically

gingerols are proven beneficial in treating health problems such as in countering liver toxicity by increasing bile secretion (Foster, 2000).

Furthermore, ginger flavor containing aromatic and pungent components are usually preferred in the flavor industries but recovery on both components simultaneously has not been possible by conventional separation processes. For instance, hydrodistillation is able to recover the pungent components but it is disable to recover the pungent of components, since the pungent components, gingerols is thermally degraded to produce volatile aldehyde and ketones (Yonei & Ohinata, 1995).

Generally, there are few problems occur in obtaining essential oil from ginger as it is highly sensitive to thermal changes. It can easily decomposed if it is undergoes in high temperature. However this proposed of study is more focused on the yield produced by hydrodistillation with conditions that will maintain its chemical constituents.

Therefore this study is focused on the effectiveness of using hydrodistillation with given conditions is certainly appropriate as it might yield better essential oil.

1.3 Objectives

The objective of this study is to obtain yield better essential oil from *Zingiber officinale* by optimizing the size of sample and time using hydrodistillation method. *Zingiber officinale* is consists with many chemical constituents, as for that a marker compound of 1, 8- cineol will be identify in each essential oils obtained to achieve the objective of this study.

1.4 Research Scope

In order to achieve the objective of this study, there are two scopes that have been identified in this study:

- i. To study the effect of size of the sample during hydrodistillation process

This study is done to discover which size of sample might be better in yielding better essential oil of ginger.

- ii. To study the effect of time to conduct the extraction process

The focus of this study is to observe the optimum time needed to obtain essential oil using hydrodistillation with better yield of essential oil without decompose the marker compound.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

2.1.1 Background Information

Ginger is scientifically known as *Zingiber officinale*. The name was given by the English botanist, William Roscoe (1753-1831) in an 1807 publication. A number of medical plants are included in the ginger family. There is about 150 species altogether, typically tropical perennials with large rhizomes. The ginger family is found abundantly in Indo-Malaysia, consisting of more 1200 plant species in 53 genera. Additionally, about 85 species of aromatic herbs from East Asia and tropical Australia are included in the genus *Zingiber* (Foster, 2000). The scientific classification of ginger is further detailed in Table 2.0.

Table 2.0 Taxonomy of Ginger (Farlex, 2004)

Kingdom	<i>Plantae</i>
Subkingdom	<i>Viridaeplantae</i>
Phylum	<i>Tracheophyta</i>
Subphylum	<i>Spermatophytina</i>
Intraphylum	<i>Angiosperma</i>
Division	<i>Magnoliphyta</i>
Class	<i>Liliopsida</i>
Order	<i>Zingiberales</i>
Family	<i>Zingiberaceceae</i>
Genus	<i>Zingiber</i>
Species	<i>Officinale</i>
Scientific name	<i>Zingiber officinale</i>
Common name	Ginger

Different species of Zingiber are shown in Figure 2.0, 2.1, 2.2 and 2.3:

**Figure 2.0** *Zingiber Officinale* (Skinner, D, 2002)



Figure 2.1 *Zingiber collinsii* (Skinner, D, 2004)



Figure 2.2 *Zingiber corallinum* (Skinner, D, 2001)



Figure 2.3 *Zingiber eborinum* (Skinner, D, 2003)

2.1.2 History of Ginger

Ginger has been cultivated for so long that its exact origin is unclear. However, for millennia it has been cultivated in Asia, specifically in China and India. In the second century, ginger was recorded as a subject of a Roman tax after being imported via the Red Sea to Alexandria. Then, tariff duties if ginger were found on the record of Marseilles in 1228 and in Paris by 1296. Ginger was later known in England through the 11th century Anglo-Saxon leech books. Details of ginger were more discovered in 13th century work “Physicians of Myddvai”, a collection of recipes and prescriptions written by a physician, Rhiwallon and his three sons. Ginger became familiar to the English by the 13th and 14th century and considered the most popular spice next to pepper. At that time, a pound of ginger was equivalent to a price of one sheep (Foster, 2000).

2.1.3 Essential oil of ginger

Ginger oil is produced from fresh or dried rhizomes. The phytochemicals of ginger consist of volatile oils (bisabolene, cineol, phellandrene, citral, borneol, citronellol, geranial, linalool, limonene, zingiberol, zingiberene, camphene), oleoresin (gingerol, shogaol), Phenol (gingeol, zingerone), Proteolytic enzyme (zingibain), Vitamin B6, Vitamin C, Calcium, Magnesium, Phosphorus, Potassium, Linoleic acid. The pungency of ginger is due to gingerol which is the alcohol group of the oleoresin. Ginger owes its aroma to about 1 to 3% of volatile oils; these are bisabolene, zingiberene and zingiberol (Kurt, 2006).

Ginger oil can vary in color from pale yellow to darker amber color and the viscosity also ranges from medium to watery. It is a strong smelling oil-spicy, sharp, and warm.

2.1.4 Marker compound

The marker compound of 1, 8-cineole is also known as 1, 8-epoxy-para-menthane, 1, 3, 3-trimethyl-2-oxabicyclo [2.2.2] octane, cineole, and eucalyptol. The molecular weight is 154.2493200 and the molecular formula is $C_{10}H_{18}O$. It is a colorless liquid in appearance and has eucalyptus camphor odor description. The melting point and boiling point of 1, 8-cineole are $2^{\circ}C$ and $176-177^{\circ}C$ respectively. It has 0.922 pounds per gallon for specific gravity. It is insoluble in water but it is slightly soluble in alcohol fixed oil water. It has stability of flammable and incompatible with acids, bases, strong oxidizing agents. 1, 8-cineole is harmful by ingestion and by inhalation or through skin contact. It may act as an irritant (Cartwright, H.M, 2006).

1, 8-cineole is used as perfumery and because of its stability it is widely used in cosmetic manufacture such as cream, lipstick, soap, lotion, shampoo, and bath foam. The naturally occurring organic compounds produced by living organisms have interested mankind for centuries. The individual organic compounds that occur in the native plants exhibit biological activities that may be used to treat various maladies. Recent clinical research has demonstrated 1, 8-cineole's effectiveness in reducing inflammation and pain, and in promoting leukemia cell death (Moteki, H *et al*, 2002). The price market of 1, 8-cineole is USD36 for 5ml.

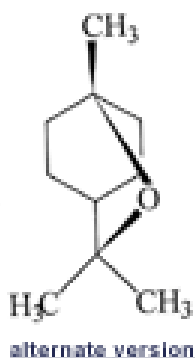


Figure 2.4 1, 8-cineole (Cartwright, H.M, 2006)

2.1.5 Medical Uses of Ginger

The important active components of the ginger root are thought to be volatile oils and pungent phenol compounds (oleoresins). Essential oil of ginger can be varying in color from pale yellow to a darker amber color. It is a strong smelling oil-spicy and sharp. The component contained in ginger give it's a distinct odor and generally used as a stimulant of the peripheral circulation, a useful diaphoretic, promote perspiration in feverish conditions and used externally for muscle sprains (Mowrey & Clyson, 1982).

Gingerols also have been shown to be inhibitors of prostaglandin biosynthesis. Based on a study done by Danish researches at Odense University on the anticoagulant

properties of ginger, it was found that ginger was a more potent blood and emdash; clotting agent than garlic or onion (Foster, 2000).

Furthermore, in a study, acetone extract of ginger at 100 mg/kg p.o. significantly inhibited serotonin (5-HT) induced hypothermia. The active responsible was found to be shogaols. Shogaols, [6]-dehydrogingerdione, [8] - and [10]-gingerols were also found to have an anticathartic action (Yamahara, J *et al*, 1990).

.The warming essential oil is often used to help treat arthritis, colic, diarrhea, and heart conditions. In addition to these medicinal uses, ginger continues to be valued around the world as an important cooking spice and is believed to help the common cold, flu-like symptoms, headaches, and even painful menstrual periods (Chaiyakunapruk, N, 2006). Today, ginger root is widely used as a digestive aid for mild stomach upset and is commonly recommended by professional herbalists to help prevent or treat nausea and vomiting associated with motion sickness, pregnancy, and, sometimes, chemotherapy for cancer. Native to Asia where its use as a culinary spice spans at least 4,400 years, ginger grows in fertile, moist, tropical soil.

2.2 Extraction of Essential oil

Essential oil can be extracted using variety of methods, although some are not commonly used today. Concerning of this study is emphasizing on the extraction of *Zingiber officinale* using hydrodistillation. Hydrodistillation of plant material can be carried out of the following techniques (Guenther, E, 1972): Water distillation, Water and Steam distillation, and Steam distillation which is the most popular method used to extract essential oil from ginger.

2.2.1 Distillation of the Oil

Essential oils are very volatile organic constituent of fragrant plant matter. They are 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 150-300°C at atmospheric pressure in Table 2.1 (Guenther, E, 1972). If heated to this temperature, labile substances would be destroyed and strong resinification would occur.

Majority of essential oils are produced by distillation. Distillation is the process of heating a liquid until it boils, capturing and cooling the resultant hot vapors, and collecting the condensed vapors. Interactions between the components of the solution create properties unique to the solution, as most processes entail non-ideal mixtures, where Raoult's law does not hold. Such interactions can result in an azeotrope. At an azeotrope, the solution contains the given component in the same proportion as the vapor, so that evaporation does not change the purity, and distillation does not effect separation (McMahon, 2005).

Distillation is for the identification and the purification of organic compounds. It is used to purify a compound by separating it from a non-volatile or less-volatile material. When different compounds in a mixture have different boiling points, they separate into individual components when the mixture is carefully distilled. The odors occur because many of organic chemicals are volatile.

Interactions between the components of the solution create properties unique to the solution, as most processes entail non-ideal mixtures, where Raoult's law does not hold. Such interactions can result in an azeotrope. At an azeotrope, the solution contains the given component in the same proportion as the vapor, so that evaporation does not change the purity, and distillation does not effect separation (Sandra, 2001).

Table 2.1 Boiling points of some common essential oil components, °C

Constituents	BP	(at atm pressure)	BP
<i>alpha</i> -Pinene	154.75	Citral	228.00
Camphene	159.50	Geraniol	229.65
Myrcene	171.50	Carvone	230.84
<i>alpha</i> -Phellandrene	175.79	Thymol	231.32
Cineole	176.40	Safrole	234.50
<i>p</i> -Cymene	176.80	Cuminic aldehyde	235.50
Dipentene	177.60	Carvacrol	237.70
Fenchone	193.53	Anethole	239.50
Linalool	198.30	Cinnamic aldehyde	251.00
<i>beta</i> -Tujone	201.00	Eugenol	252.66
Citronellal	206.93	Caryophyllene	260.50
Borneol	212.00	Isoeuganol	266.52
<i>l</i> -Menthol	216.00	Zingiberene	269.50
<i>alpha</i> -terpinoel	217.50	Dillapiol	285.00
Dihydrocarvone	222.40	Coumarin	301.72
Bornyl acetate	223.00	<i>alpha</i> -Santalol	301.99
Citronellol	223.42	<i>beta</i> -Santalol	309.00

2.2.2 Methods for Hydrodistillation

2.2.2.1 Steam distillation

This method is the most widely used in essential oil manufacture from plant material. The steam is produced outside the still, usually in a steam boiler. Steam at optimum pressure is introduced into the still below the charged through a perforated coil or jets. Steam distillation is relatively rapid and is capable of greater control by 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 recharged 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 (Ravindran, P.N, & Babu, K.N, 2005).

2.2.2.2 Water distillation

In this method, the raw material to be distilled is letting immersed in water is charged in the still. The ratio of the mixture must be sufficient for leaving enough vapor space. The quantity of the water should be adequate for the material to leave freely in boiling water, thus localized overheating and subsequent charring of the material. It may be necessary to add more water ad the distillation proceeds to prevent any dry material from being exposed to direct heating. The vapor is condensed and oil is separated from water, taking advantage of the mutual immiscibility and difference in specific gravity. This method is normally used where the raw materials tend to agglutinate and form large compact lumps through which steam cannot penetrate. The advantage of this method is suitable to conduct a small-scale distillation of essential oil (Ravindran, P.N, & Babu, K.N, 2005).

2.2.2.3 Water and Steam distillation

Here the plant is supported on perforated grid inside the still. The lower part of the still is filled with water to a level below a grid. The water is heated to generate steam. The steam usually is wet and at low pressure, rises through the charge carrying essential oil. The advantage of this method is that the raw material is not contact with boiling water. The exhausted plant material can be handled easily as it does not form slurry with water (Ravindran, P.N, & Babu, K.N, 2005).

2.2.3 Boiling Point

The boiling point is the temperature at which the vapor pressure of the liquid phase of a compound equals the external pressure acting on the surface of the liquid. The external pressure is usually the atmospheric pressure. The vapor pressure of the liquid will increase as the temperature of the liquid increases, and when the vapor pressure equals the atmospheric pressure, the liquid will boil. Different compounds boil at different temperatures because each has a different, characteristic vapor pressure where compounds with higher vapor pressures will boil at lower temperatures (Sandra, 2001)

2.2.4 Principle of Hydrodistillation

Hydrodistillation involves the use of water or steam to recover volatile principles from plant materials. The fundamental feature of hydrodistillation is that it enables a compound or mixture of compounds to be distilled and subsequently below that of the boiling point of the individual constituent. The advantage of using hydrodistillation is it permits the safe recovery of these heat-sensitive compounds from the plant matter (Gilbert, J.C & Martin, S.F, 2002).

During hydrodistillation, water and essential oil form a heterogeneous system of immiscible liquids. By the principle of distillation of 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 compound distill at temperature below 100°C when boil with water at atmospheric pressure (McCabe, W.L, et al, 2003)

2.2.5 Simple Hydrodistillation (Lab-Scale)

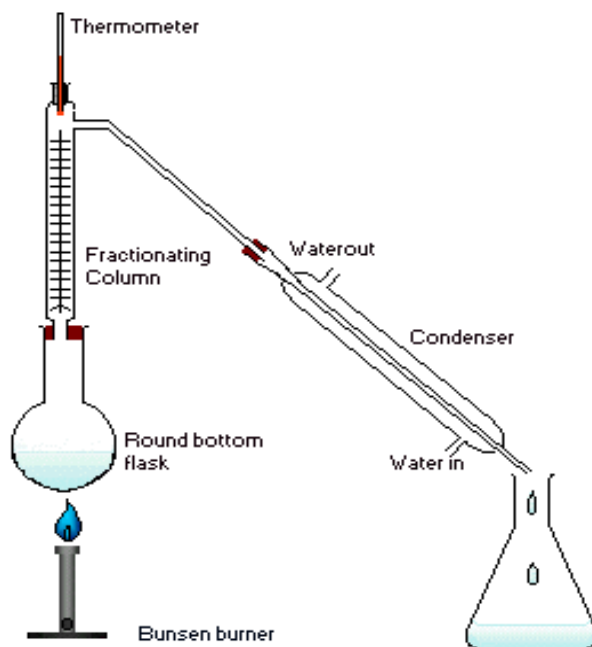


Figure 2.5 Diagram of simple distillation set-up with a fractionating column (Wikipedia, 2006)

Hydrodistillation is a special type of distillation (a separation process) for temperature sensitive materials like natural aromatic compounds. Many complex organic compounds tend to decompose at sustained high temperatures. Separation by normal distillation would then not be an option, so water or steam is introduced into the distillation apparatus. By adding water the boiling point of the compounds is depressed, allowing them to evaporate at lower temperatures, preferably below the temperatures at which the deterioration of the material becomes appreciable. After distillation the vapors are condensed as usual, usually yielding a two-phase system of water and the organic compounds, allowing for simple separation.

Ginger boils at 256°C while water boils at 100°C . So, by gently heating the mixture, the most volatile component will concentrate to a greater degree in the vapor leaving the liquid. Some mixtures form azeotrope, where the mixture boils at a lower

temperature than either component. A mixture of 40% ginger and 60% water boils at 80°C. The mixture is put into the round bottomed flask and the fractioning column is fitted into the top. As the mixture boils, vapor rises up the column. The vapor condenses on the glass platforms, known as trays, inside the column, and conducts back down into the liquid below, refluxing distillate. The column is heated from the bottom. The hottest tray is at the bottom the coolest is at the top. At steady state conditions the vapor and liquid on each tray is at equilibrium. Only the most volatile of the vapors stays in gaseous form all the way to the top. The vapor at the top of the column then passes into the condenser, which cools it down until it liquefies. The condensate that was initially very close to the azeotrope composition becomes gradually richer in water. The process continues until all there is no more oil produced (Wikipedia, 2006).

In laboratory distillation, several types of condensers are commonly found. The Liebig condenser is simply a straight tube within a water jacket, and is the simplest (and relatively least expensive) form of condenser. The Graham condenser is a spiral tube within a water jacket, and the Alhin condenser has a series of large and small constrictions on the inside tube, each increasing the surface area upon which the vapor constituents may condense. Being more complex shapes to manufacture, these latter types are also more expensive to purchase. Condensers are usually sold by the mm: 100, 200, and 400 mm are common lengths, and are connected to the other vessels with ground glass fittings (Feist, 2000).

2.3 Analyzing Essential oil

2.3.1 Gas Chromatography Analysis

Gas chromatography (GC) is used as analytical tool to discover how many components are in a mixture. Besides that, it is also used to separate small amounts of material and to determine whether a desired component is present. For instances, GC is

used I petrochemical (refinery gas), pharmaceutical (alkaloid street drugs) and environmental (chlorinated pesticides) analysis (Davisson, 2005).

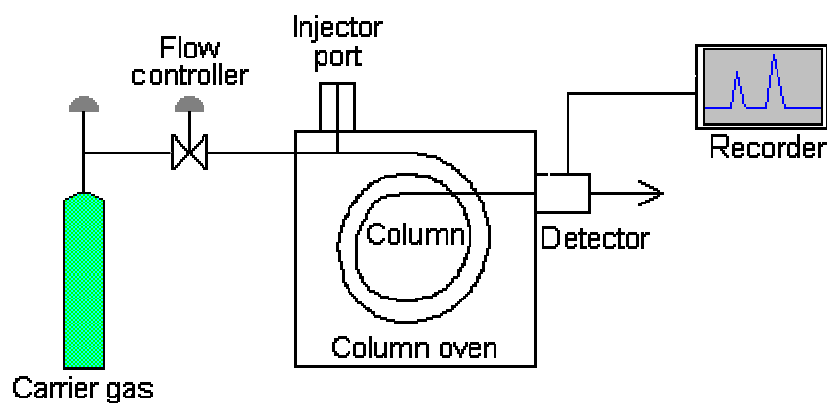


Figure 2.6 Gas Chromatography

2.3.2 Principle of process

In all types of chromatography, a mixture is separated by distributing the component between 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 is dependent on the compounds traveling through the column at different rates (Feist, 2000).

The rate at which a compound travels through a particular GC system depends on the factors listed below:

i. Volatility of compound

Low boiling (volatile) components travel faster through the column than high boiling point components.

ii. Polarity of compounds

Polar compounds move more slowly, especially if the column is polar.

iii. Column temperature

Raising the column temperature speeds up all the compounds in a mixture.

iii. Column packing polarity

Usually, all compounds move slower on polar columns, but polar compounds will show a larger effect.

iv. Flow rate of the gas

Through the column, speeding up the carrier gas flow increases the speed with which all compounds move through the column.

v. Length of the column

The longer the column, the longer it will take all compounds to elute. Longer columns are employed to obtain better separation.

2.3.3 Injection System of a Gas Chromatography

The sample has to be vaporized prior to analysis by GC; this is a limiting factor for many inlets. The first and most common method is to introduce a small volume of sample with syringe. This injection may be done by hand or by an automatic sampler.

Next is Headspace sampling which involves taking a sample of the gas above a liquid sample (the headspace) and injecting into the chromatograph.

Third is purge and trap which is a variation on headspace analysis. A gas is bubbled through the sample and the analyte is trapped in a special kind of filter (or in a cold trap), this concentrates the analyte, the trap is then heated to desorb the analyte off of the trap and into the column (Brammer, 1996).

2.3.4 Column Selection

Capillary column are capable of more efficient separation. They enable more complex mixtures to be separated or resolved. As for packed column, they have extremely high surface area, which is an advantage for large amounts of analyte like when separating gases. Capillary column, however, have much greater resolution so they are more widely used for analysis.

The most important consideration is the stationary phase (this is the liquid that is coated onto the inside of a capillary column or on the packing material of a packed column). The stationary phase is selected to separate the compounds of interest. For instance, a polar column will retain polar molecules longer; therefore it is better for separating polar compounds. Likewise, a non-polar column is used for non-polar analyte. Other stationary phases are designed to interact with different types of functional groups (Brammer, 1996).

2.3.5 Detectors Used in a Gas Chromatography

The differences and advantages of each are vital in selecting an analysis technique. Thermal conductivity is the amount of the heat conducted by the effluent. When the analyte exits the column and passes through the detector it changed the amount

of the heat conducted by the effluent. This detector is sensitive to almost any compound (this may be an advantage or disadvantage) (Chasteen, T.G, 2006).

Flame ionization detector (FID) is essentially a small H_2 flame that the column effluent flows into. When an organic compound (or anything that burns) enters the detector the combustion produces ions that are easily detected. This detector is very sensitive and responds to most compounds (anything that burns). However, it does not respond to air or water.

The Flame Ionisation Detector

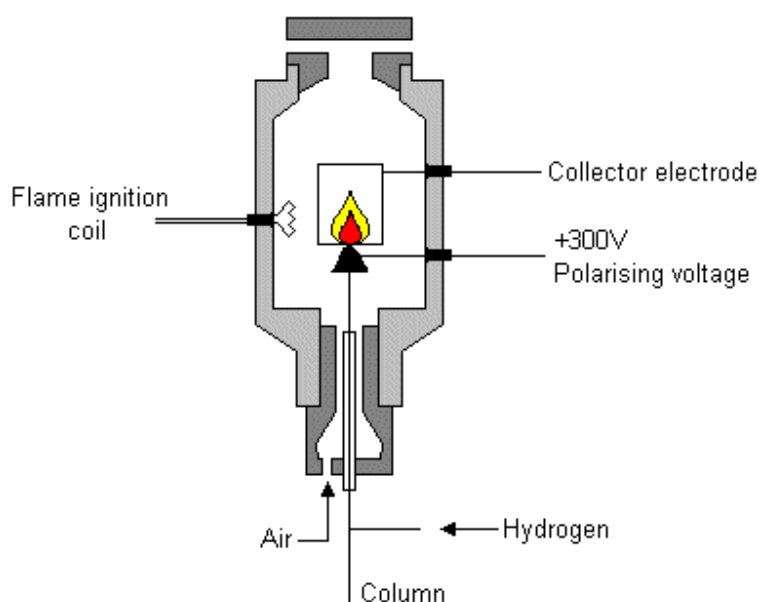


Figure 2.7 FID (Chasteen, T.G, 2006)

Thermionic Emission Detector is a slight variation of FID. The flame is adjusted so that the combustion temperature is very low and most organic compounds do not undergo combustion to form ions. A special bead (rubidium silicate) is inserted into the flame to catalyze the combustion of the compounds with nitrogen or phosphorus. This detector is only sensitive to nitrogen and phosphorus containing compounds

Electron Capture Detector (ECD) is considered the strangest and most difficult detector to understand, but it is very useful for environment analysis. In this detector, electrons are created by a radioactive source, then they travel across the detector and are detected. The effluent flows through the detector. When a compound that absorbs electrons enters the detector, the electron current is decreasing. Due to the way it works this detector is extremely sensitive to certain compound classes (particularly chlorinated hydrocarbons).

Fourier Transform Infrared (FT-IR) and Mass Spectrometry (MS) detectors are providing with structural information about the analyte. This is very useful for identification of unknowns, but the technique are not usually as sensitive as other detectors (Chasteen, T.G, 2006).

CHAPTER 3

METHODOLOGY

3.1 Introduction

The experiment was conducted focusing on the achievement of the conceptual study, laboratory experimental work, analyzing and completion the project. The detailed procedure of the experimental work is discussed through out this chapter. The purpose of this experimental work is to understand the requirement for the experiment. This will include the two major steps which are the requirement laboratory testing (lab-scale experiment) and data analysis. Besides that, the analysis is being done to get the data for the study. From the experiment conducted all the necessary result has been identified.

Two different experimental had been done during the optimization of essential oil extraction from ginger based on the hydrodistillation conditions which are:

- i. Condition I: Size of the sample
- ii. Condition II: Time to conduct the extraction process

3.2 Procedure of Experimental Study

This procedure was applied for both experiments conducted.

- Substances:
- Old-ginger (raw material)
 - Sodium sulfate anhydrous (chemical substance)
 - Ice slurry (water bath)
- Operating conditions:
- Heating temperature: 80-90°C
 - Water bath temperature: 0-5°C
 - Oven temperature: 50°C

3.3 Apparatus

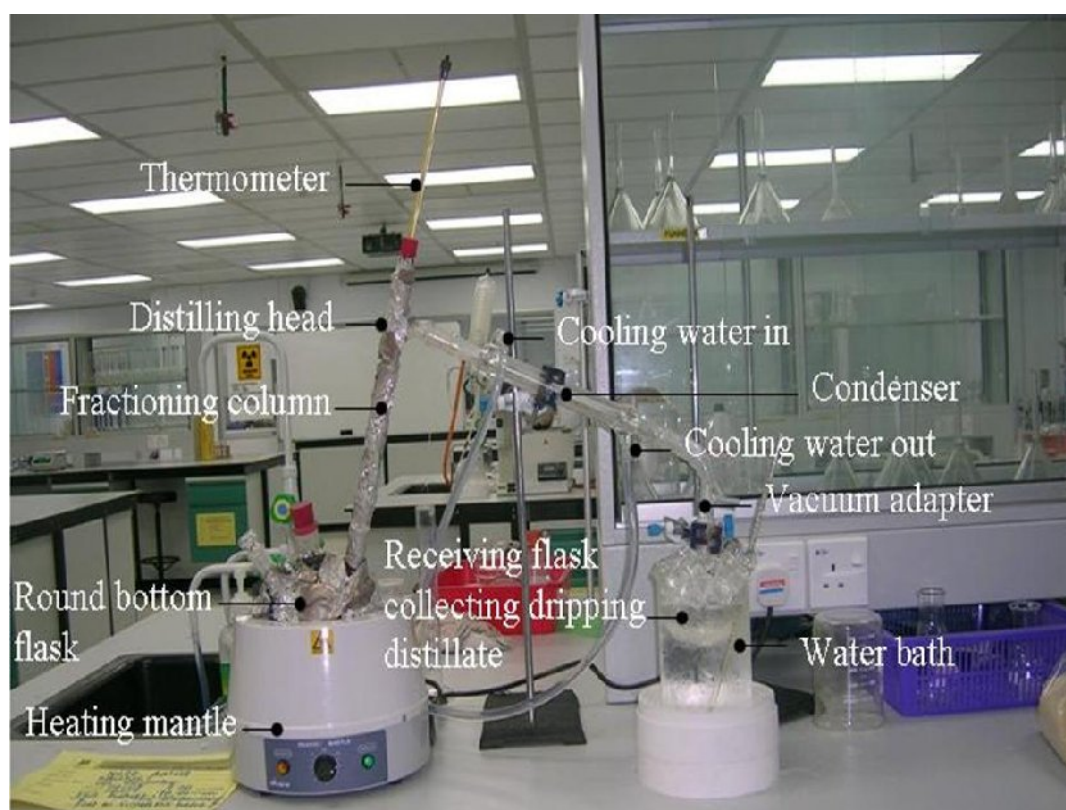


Figure 3.0 Simple distillation set-ups

The schematic diagram of the experimental apparatus used for both experimental studies is shown in Figure 3.3. It consist of heating mantle, round bottom flask (1L or