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RESEARCH TITLE :

PILOT SCALE OF THE SOLVENT EXTRACTION OF VALUE ADDED PRODUCT FROM ZINGIBER OFFICINALE ROSCOE : ESSENTIAL OILS AND OLEO RESIN

(PENGEKSTRAKAN PELARUT BERSKALA LOJI PANDU BAGI PRODUK NILAI TAMBAH DARIPADA ZINGIBER OFFICINALE ROSCOE : ESSENTIALS OIL & OLEORESIN)

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ABSTRACT

Ginger is scientifically known as *Zingiber Officinale Roscoe*. The herbaceous perennial has been popular in culinary uses and home remedies since thousands of years. This study is focused on oleoresin production as it is the total extract of the natural herb, representing both volatile and non volatile components. One of the advanced technology in extracting essential oils and oleoresins is by using the multi-purpose extractor. In this research, the extractor was utilized to its maximum capability to prove whether it is effective in achieving least residual solvent whilst obtaining a high quality flavor ginger oleoresin within a short limit of time. The best solvent to enhance ginger extraction was also investigated. The process began with extraction of ginger using solvent in the extraction vessel for a varied time limit. Then, the mixture of oil and solvent was pumped to the evaporation vessel where the solvent was vaporized. Finally, purification occurred when the last remaining solvent was separated in the thin film evaporator. Three important processes identified as extraction, distillation and purification carried out batch wisely in a pilot plant ensures the production of a high quality and quantity yield. *n*-hexane was discovered as the best solvent to improve ginger's extraction time. The highest yield obtained from the lab study was 19.2 ml and from the Ginger Extraction Pilot Plant was 30.5 ml at 4 hours of extraction time. Apart from being effective, this study might as well discover potential savings in its operational cost.

ABSTRAK

Halia secara saintifiknya dikenali sebagai *Zingiber Officinale Roscoe*. Tumbuhan herba ini telah menjadi ramuan di dalam masakan dan ubat-ubatan tradisional sejak ribuan tahun dahulu. Kajian ini fokus terhadap penghasilan oleoresin memandangkan oleoresin merupakan ekstrak yang mengandungi kesemua komponen yang mudah dan sukar meruap. Salah satu teknologi terkini dalam pengekstrakan minyak dan oleoresin ialah penggunaan ekstraktor serbaguna. Alat ini digunakan untuk menguji keefektifannya dalam mencapai oleoresin halia berkualiti tinggi yang memiliki kurang kesan pelarut dalam suatu tempoh yang pendek. Selain itu, penggunaan pelarut terbaik untuk pengekstrakan minyak halia turut dikaji. Proses bermula apabila halia direndam di dalam bekas pengekstrak untuk suatu tempoh masa tertentu. Seterusnya, campuran minyak dan pelarut tersebut dipam ke bekas pemeruapan untuk mengasingkan pelarut dari minyak. Akhirnya, penyucian berlaku di pemeruap filem nipis bagi menanggalkan sisa-sisa terakhir pelarut dari sampel minyak. Tiga proses utama dikenal pasti sebagai ekstraksi, distilasi dan penyucian dijalankan secara satu persatu di dalam suatu loji pandu bagi memastikan penghasilan produk berkualiti dan berkuantiti tinggi. *n*-heksana ternyata dapat mempercepatkan proses pengekstrakan minyak halia ini. Hasil paling banyak yang diperoleh dari ujikaji makmal ialah 19.2 ml, manakala melalui pengekstrak serbaguna ialah 30.5 ml untuk jangka masa 4 jam pengekstrakan. Selain efektif, kajian ini juga ekonomikal melalui penjimatan kos operasinya.

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cm	-	Centimeter
d	-	Depth
e.g.	-	Example
h	-	Height
Hz	-	Hertz
kW	-	Kilowatt
L	-	Liter
m	-	Meter
max.	-	Maximum
ml	-	Milliliter
mm	-	Millimeter
P	-	Pressure
T	-	Temperature
V	-	Volt
w	-	Width
°C	-	Degree Celsius
°F	-	Degree Fahrenheit
%	-	Percentage
<	-	Less than

CHAPTER 1

INTRODUCTION

1.1 Origin and Current Location of Ginger

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

1.2 Physical Structure of Ginger Plant

Ginger is obtained from a plant scientifically known as *Zingiber Officinale Roscoe*. It is an herbaceous perennial with upright stems, growing from one to three feet in height. The stem is surrounded by the sheathing bases of the two ranked leaves. It also possesses a club like spike of yellowish, purple lipped flowers with showy greenish yellow bracts beneath.

1.3 Usage of Ginger

For thousand of years, ginger has been widely used as a culinary herb, condiment, spice, home remedy and medicinal agent. Ginger extracts have been extensively studied for several biological activities including antibacterial, anti convulsant, analgesic, antiulcer, gastric antisecretory and antitumor. Mowrey and Clayson (1982) reported that ginger gave an excellent result in treating nausea. In addition, gingerol, (one of the primary pungent principles of ginger) has been found to be helpful in countering liver toxicity by increasing bile secretion. A study by Bone (1990) showed that acetone and methanol extracts of ginger strongly inhibits gastric ulceration.

1.4 Usual Methods of Obtaining Ginger Essential Oil and Oleoresin

Oleoresin and ginger oil are two value added products gained from the dry rhizome of *Zingiber Officinale Roscoe*. Ginger oil is obtained by steam distillation while the oleoresin is recovered from solvent extraction. Ginger oleoresin is known to have constituents responsible for its medicinal effects such as gingerol and shogaol.

Supercritical fluid extraction utilized carbon dioxide under high pressure and low temperature and has advantages over steam distillation in terms of obtaining better yield, shorter extraction time, lower energy consumption and better sensory properties of the extract. This technique is preferred because there is no thermal degradation, and the aromatic profile is closer to the profile in the plant. Extraction of ginger using high pressure carbon dioxide has been done by Yonei and Ohinata (1995). They found that 6-gingerol could be easily concentrated by the two stage separation proposed in the study.

Based on the information gathered, ginger extraction using multi-purpose extractor is thus proposed to study whether it is effective in producing high yield of oleoresin as well as low time consumption.

1.5 Research Background / Problem Statement

Generally, there are a few problems that occur in ginger extraction. However, this proposed study is more focused on the yield produced by the multi-purpose extractor or also known as Ginger Extraction Pilot Plant; ginger oleoresin.

Many researches have been conducted to discover the use of ginger in various field, especially in the medicinal 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). However, these components are volatile and can be thermally decomposed.

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

In addition, the removal of the last remaining quantity of solvent in ginger extraction has yet to be solved. An effective method to purify the yield is still being searched.

Therefore, in the Ginger Extraction Pilot Plant, a short path distillation is applied to help in producing high purity oleoresin as well as possessing high content of the pungent components. A study on the effectiveness of using this equipment is certainly appropriate as it might be an excellent alternative for ginger extraction in future.

1.6 Objective

The objective of this research is to study on the effectiveness of using the multi-purpose extractor in ginger extraction.

1.7 Research Scope

In order to achieve the objective of this research, three scopes have been identified in this research.

i. To study on the effects of extraction time on yield

The focus of this study is to observe the optimum time needed in extracting ginger oleoresin using the multi-purpose extractor.

ii. To observe and compare the effects of using different solvents (*n*-hexane, ethanol and diethylene glycol) in ginger extracting process.

This study is conducted to discover the type of solvent that can enhance the production of yield within a specified range of time.

iii. To achieve least residual solvent whilst obtaining a high quality flavor oleoresin.

The effectiveness of using multi-purpose extractor to yield pure ginger oleoresin without traces of solvent is investigated.

1.8 Contribution of The Study

The multi-purpose extractor is expected to improve the quality of yield (ginger oleoresin).Among the outputs expected from the project are stated as follow:

i) An effective and efficient method in removing the last few percents of solvent which is a problem that has yet to be solved satisfactorily up until now.

ii) Application of advanced technology in ginger extracting process.

iii) Potential savings in the operational cost.

CHAPTER 2

LITERATURE REVIEW

2.1 Separation Processes

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 substances 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.

The analysis of separation processes are divided into two fundamental categories:

i. Equilibrium- based processes

ii. Rate-based processes

Equilibrium-based processes are those in which the degree of separation in each stage is governed by a thermodynamic equilibrium relationship between the phases. The common processes related to this category are:

a. Distillation

b. Extraction and leaching

In distillation, a different temperature at each stage alters the vapor phase equilibrium between typically binary mixtures. The desire of a new equilibrium between the two phases at the temperature of each stage is the driving force for separation. The end result is the separation of two liquids with dissimilar boiling temperatures.

In extraction, a species is removed from a liquid in which it is dissolved by means of another liquid for which it has a higher affinity. As for leaching, a species is removed from a solid phase by means of another liquid for which it has a stronger affinity.

Rate-based processes are limited by the rate of mass transfer of individual components from one phase into another under the influence of physical stimuli (such as concentration, temperature, pressure, external force). Among the processes that are included in this category are:

a. Gas absorption

In gas absorption, a vapor is removed from its mixture with an inert gas by means of a liquid in which the vapor is soluble.

b. Desorption or stripping

In desorption, a volatile gas is removed from liquid by means of a gas in which the volatile gas is soluble.

c. Adsorption

In adsorption, a species from a fluid stream is removed by means of a solid adsorbent in which the species has a higher affinity.

d. Ion exchange

Ion exchange is similar to adsorption except the species removed from solution is replaced with a species from a solid resin matrix in order to maintain electroneutrality.

e. Membrane separations

In membrane separations, the processes depend on differences in permeability between components of a feed stream, due to size and chemical selectivity for the membrane material.

2.2 Extraction

Extraction is the removal of one or more solutes from a liquid by transferring the solute into a second liquid phase, for which the solute has a higher affinity (Noble & Terry, 2004). Separation through extraction depends on differences in both solute solubility and density of the two phases.

There are a few advantages of extraction separations. Firstly, extraction can be performed at ambient temperature. Therefore, it is relatively energy efficient and can be applied to separations involving thermally unstable molecules. Close boiling mixtures or substances that cannot withstand the temperature of distillation, even under a vacuum, usually are separated from impurities by extraction, which utilizes chemical differences instead of vapor pressure differences. Besides that, extraction processes can accommodate changes in flow rates and the solvent can be recovered and recycled for reuse. It offers greater flexibility in terms of operating conditions too, since the type, amount of solvent and operating temperature can be varied.

Nevertheless, extraction also has its disadvantages. In this process, the solvent must be recovered for reuse (usually by distillation), and the combined operation is more complicated and often more expensive than ordinary distillation without extraction (McCabe, Smith & Harriott, 2001).

2.2.1 Definition of Extraction Terms

Solute (A) : The species to be removed from the liquid diluent stream

Diluent (D) : The component containing the solute which is to be removed

Solvent (S) : The second liquid stream which will remove the solute from the diluent

Raffinate (R) : The exiting phase which has a high concentration of diluent (and less A)

Extract (E) : The exiting phase which has a high concentration of solvent (and more A)

2.2.2 Extraction Equipment

Liquid extraction requires the two phases be brought into intimate contact with each other to permit transfer of the solute from the diluent to the solvent, and then separated. However, the two phases involved often have similar densities. Due to this, they are usually difficult to mix and even more difficult to separate. In addition, liquids have sufficiently high viscosities that they require pumps to maintain flow. Therefore, most extraction processes include mechanical energy for pumping, mixing and separating the liquids (McCabe, Smith & Harriott, 2001).

2.2.3 Methods of Extracting Essential Oils and Oleoresins

There are several methods that have been used to extract essential oils and oleoresins. Among them:

i. Distillation
ii. Steam distillation
iii. Hydrodistillation
iv. Water and steam distillation
v. Cold pressing
vi. Florasols/ phytols
vii. Solvent extraction
viii. Carbon dioxide extraction

Majority of essential oils are produced by distillation (McMahon, 2005). Water is heated to produce steam, which carries the most volatile chemical of the aromatic material with it. Then, the steam is chilled in a condenser and the distillate is collected. The oil usually floats on top of the hydrosol (the distilled water component) and can be separated off.

In steam distillation, an outside source of steam is used. The steam is piped into the distillation unit, sometimes at high pressure. The steam then passes through the aromatic material, and exits into the condenser.

Other than that, there is hydrodistillation. Hydrodistillation is the oldest and most common method of extracting essential oil since it is economically viable and safe (Nurul Azlina Mohamed, 2004). The botanicals are fully submerged in water and the steam produced contains the aromatic plant molecules. This technique is effective for powders such as spice powder and ground wood as well as very tough materials like roots, woods or nuts.

In water and steam distillation, the botanicals are placed in a basket over boiling water, thus exposing the plant material to the steam vapors. This method only works for distilling leafy materials.

As for cold pressing, this process is usually applied to obtain citrus oil such as orange essential oil. Machines score the rind and capture the resulting oil.

Next is extraction using florasols/phytols. This method uses a new type of benign gaseous solvents which is florasol. Extraction occurs at below ambient temperature and therefore, there is no thermal degradation of products. It also utilizes the selectivity of the solvent and produces free flowing clear oil free of waxes.

Other than that is solvent extraction. This technique is used to extract very delicate aromatics which cannot withstand distillation. For instances,jasmine and linden blossom. In this process, the blossoms are washed repeatedly with solvent (usually hexane).The solvent dissolves all non aromatic waxes, pigments and highly volatile aromatic molecules. Then, the solution is filtered and the filtrate subjected to low pressure distillation to recover the solvent for further use. The remaining waxy mass is called concrete. The concretes are later processed to remove the remaining waxy materials which dilute the essential oil by warming and stirring them with alcohol (usually ethanol). Then, the solution is further agitated and freezed at very low temperatures (around -30°F) to remove most of the wax. Next, the purified solution is cold filtered leaving only the wax free material

In extraction using supercritical carbon dioxide (CO_2), the process is conducted at relatively low temperatures. Solvent removal from the extract is quite easy and any alteration of heat sensitive components and the loss of volatile components are minimized. Based on a research, the extract of ginger obtained by high pressure CO_2 contains both aromatic and pungent components and resembles the flavor profile of the starting material (Yonei & Ohinata, 1995). Another study on extraction of Australian

ginger root with CO_2 and ethanol entrainer revealed that the recovery of oleoresin was greater with entrainer, high solvent/ feed ratio and higher pressure, but showed little variation with temperature over the range involved (Badalyan, Wilkinson & Byung-Soo Chun, 1998). Apart from that, the effectiveness of using CO_2 extraction comparing to steam distillation in extracting various plant materials such as clove, cumin, sandalwood and ginger has also been done. It was found that the yields of CO_2 extraction were 10% to 360% larger than the yields of the steam distillations, while the extraction time is only ½ to 1/10 of the time needed for distillation (Naik, Lentz & Maheshwari, 1989).

In order to calculate the yield produced by these extraction methods, the following equation is done:

$$\text{Yield (\%)} = \frac{\text{Weight of ginger oil or oleoresin collected (g)}}{\text{Initial weight of sample (g)}} \times 100\ \%$$

Source: (Nurul Azlina Muhamad, 2004)

2.2.4 Short Path Distillation

Apart from the extraction methods above, there is another one of the latest preferred technique in extraction which is called short path distillation. Short path distillation is a thermal separation process for thermal sensitive products. It is applied when distillation at atmospheric pressure causes thermal decomposition. Besides that, it is also used for high boiling products when distillation at atmospheric pressure causes difficulties with respect to energy losses and availability of construction material. In order to achieve minimal thermal stress on the distilled products, two factors have to be considered which are short residence time and low evaporation temperatures.

2.2.4.1 Principle of Process

Short path distillation is a continuous separation process working under vacuum conditions. In this process, evaporation takes place from a heated wiped film. Lower pressure in short path evaporator is obtained by the short distance for the vapors on their way from the evaporator surface to the condenser. In addition, the cross section area of flow is equal to the evaporator and condenser causing minor pressure drop between evaporator and condenser (Fischer, 1995).

In the fine vacuum range (for example; between 1 and 0.001 mbar), a pressure reduction to 1/10 results in a great decrease of the evaporation temperature, so far that during the short residence time, no thermal decomposition of the product can occur.

There are several evaporators that have similar function in short path distillation such as:

i. Short path evaporator with internal condenser
ii. Wiped film evaporator with externally located condenser
iii. Pot still distillation unit
iv. Thin film evaporator

The Short Path Evaporator with internal condenser combines evaporator and condenser in one apparatus. Therefore, pressure losses caused by piping are eliminated. The distance between evaporator surface and condensation is extremely short resulting in minimum pressure drop. The apparatus also has a roller wiper system which is useful in mixing continuously the product film on the surface of the evaporator. Besides that, it avoids any concentration gradient inside the thin film. There is also an increase of the evaporator surface due to the surface of the rollers. In addition, it possesses its self cleaning characteristic.

In a wiped film evaporator, the condenser is located externally. It is connected to the evaporator by nozzles for vapour transfer. The vacuum pump system is connected to the condenser. Thus, the operating pressure of this design is limited to some millibars due to the pressure drop through the connection between the evaporator and condenser.

In a still pot distillation, there are several disadvantages for heat sensitive products such as:

a. Long residence time caused by batch process
b. Poor vacuum at the place of evaporation, since drops in pressure and static height of the liquid have to be added to the suction pressure of the vacuum pump set.
c. The ultimate pressure is not determined by size of the vacuum pump set but is limited by the conductivity of the piping and the static height of the liquid in the evaporator vessel.

As for the thin film evaporator, the product runs down on a heated cylindrical surface on a cylindrical pipe. The liquid film is mixed continuously by a rotating wiper system. This wiper system which usually consists of graphite rollers enables film uniformity, film mixing, low material hold up and self cleaning. Even small amounts of material are pressed out of the rollers' interior by centrifugal force, resulting in no product residues to cause thermal decomposition. Condensation occurs in a condenser placed outside, which is connected to a vacuum system. The pressure drop of the connection between evaporator and condenser thus limits the obtainable vacuum to some mbar.

2.2.4.2 Applications of Short Path Distillation

Short path distillation has been extensively utilized in extracting several materials such as to obtain vitamin concentrates from natural vitamin E in vegetable oils, increased

carotene concentration from red palm oil, omega-3-fatty acid-ester from fish oils and Paprika oleoresin from paprika (Fischer, 1995). Additionally, in a study on purification of meadowfoam monoestolide from polyestolide, cosmetic applications using meadow foam estolide require products of high purity and low colour. Therefore, short path distillation was applied and the desired result has been successfully achieved (Isbell & Cermak 2004).Apart from that, in the production of cocoa butter equivalent through enzymic interesterification of palm oil midfraction, short path distillation was used to separate the unreacted fatty acids from the interesterified products. The product obtained was also practically without free fatty acids (Undurraga, Markovits & Erazo).

2.3 Ginger

2.3.1 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 of ginger were found on the records 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 a 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).

2.3.2 Location

Ginger's origin is believed to be in tropical Asia, but now it is grown as a commercial crop in Latin America, Africa and South East Asia. Among the major world producers of ginger are India, Fiji, Jamaica, Nigeria, Sierra Leone and China. Out of the total worldwide production, India contributes fifty percent from it (Floridata, 2003).

2.3.3 Background Information

Ginger is scientifically known as *Zingiber Officinale Roscoe*. The name was given by an English botanist, William Roscoe (1753-1831) in an 1807 publication. A number of medical plants are included in the ginger family. There is about 1500 species altogether, typically tropical perennials with large rhizomes. A few examples of the species are shown in Figure 1, 2, 3 and 4. 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 2.1.

Table 2.1: Taxonomy of ginger (Farlex, 2004)

Scientific Classification	
Kingdom	Plantae
Division	Magnoliophyta
Class	Liliopsida
Order	Zingiberales
Family	Zingiberaceae
Genus	Zingiber
Species	Officinale

Different species of *Zingiber* are shown in Figure 2.1, 2.2, 2.3 and 2.4:

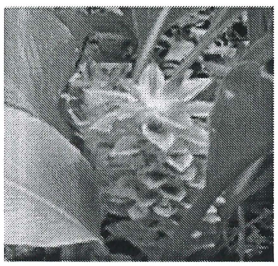


Figure 2.1 *Zingiber Officinale Roscoe* (Farlex, 2004)

Figure 2.2 *Zingiber Rubens New Orleans* (Farlex, 2004)

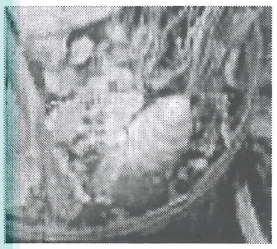


Figure 2.3 *Zingiber Eborinum Tomwood* (Farlex, 2004)

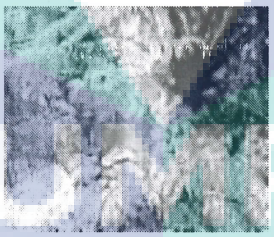


Figure 2.4 *Zingiber Zerumbet* (Farlex, 2004)

2.3.4 Morphology of Ginger

The ginger plant is an herbaceous perennial with upright stems, growing from one to three feet in height. This is illustrated in Figure 2.5.The stem is surrounded by the sheathing bases of the two ranked leaves. The leaves are green and narrow medium with ¾ inches (1.9cm) wide and 7 inches (17.8cm) long. It also possesses a club like spike of yellowish, purple lipped flowers with showy greenish yellow bracts beneath. These flowers are rarely seen as most gingers in cultivation are sterile cultivars grown only for the edible rhizome. On the other hand, the rhizomes are thick and scaly (underground stems of the plant that are warty and branched) as seen in Figure 2.6.

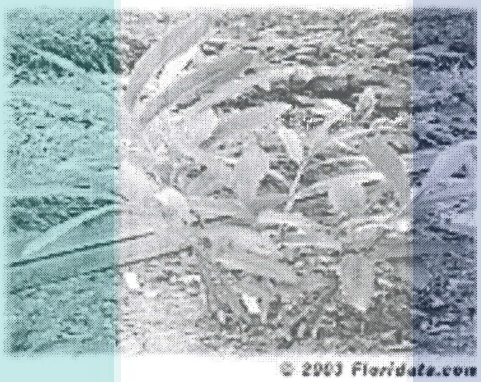


Figure 2.5 Ginger plant (Floridata, 2003)

Figure 2.6 Ginger rhizome (Floridata, 2003)

2.3.5 Products of Ginger

There are two value added products of ginger which are ginger oil and ginger oleoresin. These products are distinguished by the techniques they are derived, their appearances, odour and flavour.

2.3.5.1 Ginger Oil

Ginger oil is produced from fresh or dried rhizomes. When comparing the two sources, oil from the dried rhizomes will have less of the low boiling point volatile components. This is because the components are evaporated during the drying process. In order to improve yield, unpeeled rhizomes are preferred.

Steam distillation is a technique used to obtain ginger oil. In steam distillation, dried rhizomes are ground to a coarse powder and loaded into a still. Next, steam is passed through the powder, entraining the volatile components, which are later condensed with cold water. Upon cooling, the oil is separated from the water. Ginger oil appears to be pale yellow to yellow in colour. Its odor can be described as spicy, woody, terpy, warm, ginger citrus. It has a boiling point of 254°C.

The major components in ginger oil are zigerberene (20-37%), ar-curcumene (5-20%), -and- farnesene, -bisabolene and-sesquiphellandrene. However, the low boiling point components such as geranial and neral that impart aroma characteristic to the product. For example, Australian oils have a high citral content, up to 27%, averaging 19%, imparting a lemony aroma to the product (Plotto, 2004).

2.3.5.2 Ginger Oleoresin

Ginger oleoresin is obtained through extraction of the unpeeled and dried powdered rhizome of *Zingiber Officinale Roscoe*. The oleoresin contains gingerols (6-,8-, and 10-gingerol) which are components responsible for pungency. Structure of gingerols is shown in Figure 2.7. These components are readily decomposed to the less pungent shogaols and zingerons upon heating. Therefore, solvent extraction is a preferred technique when pungency of product is desired.

The oleoresin appears to be dark brown or very dark amber viscous liquid which usually deposits a grainy mass at the bottom of container. It has a warm, spicy and sweet odor which can be further described as ginger earthy, musty, citrus. Its flavour is equally warm, but pungent biting, leaving a peculiar cooling aftertaste.

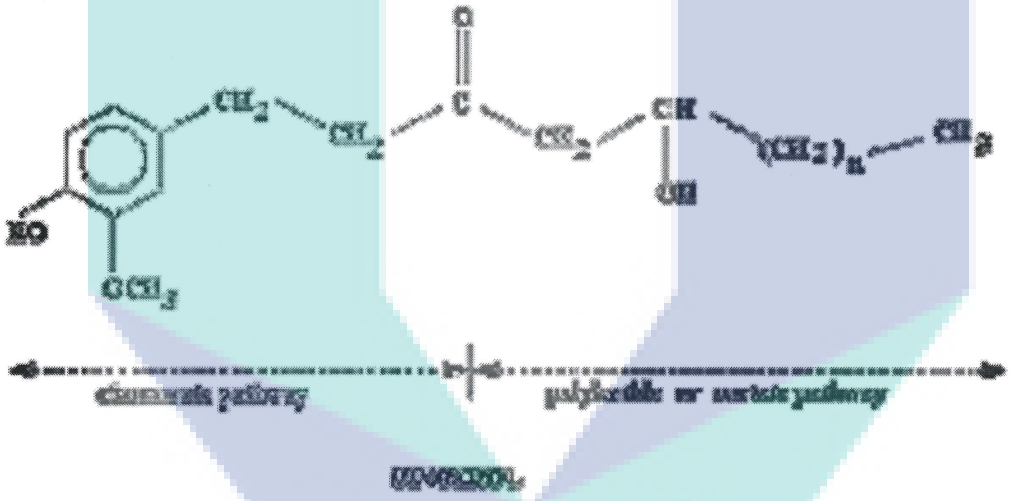


Figure 2.7 Structure of gingerol

2.3.6 Key Constituents in Ginger

i) Volatile oils: bisabolene, cineol, phellandrene, citral, borneol, citronellol, geranial, linalool, limonene, zingiberol, zingiberene, camphene

ii) Oleoresin: gingerol, shogaol
iii) Proteolytic enzyme (zingibain)
iv) Vitamin B6
v) Vitamin C
\vi) Calcium
vii) Magnesium
viii) Phosphorus
ix) Potassium
x) Linoleic Acid

2.3.7 Medical Uses of Ginger

Ginger extracts have been extensively studied for various purposes in the medical field. It has been found to be antibacterial, anticonvulsant, antiemetic, analgesic, antiulcer, antitumor and gastric antisecretory. The components contained in ginger (oleoresin; gingerol and shogaol) give it 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.

Gingerols also have been shown to be inhibitors of prostaglandin biosynthesis. Based on a study done by Danish researchers 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). Other than that, studies also prove that gingerol helps in countering liver toxicity by increasing bile secretion (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 shogoal. Shogaol, [6]-dehydrogingerdione, [8]- and [10]-gingerol were also found to have an anticathartic action (Huang, 1990).

2.4 Multi-Purpose Extractor (Ginger Extraction Pilot Plant)

Ginger Extraction Pilot Plant as illustrated in Figure 2.8 is designed for extraction of value added products from *Zingiber Officinale Roscoe*; ginger oil and oleoresins. It is developed to achieve high yield and low energy consumption. Besides that, the extract produced tends to be richer in high volatility components than that obtained by traditional methods (e.g steam distillation, solvent extraction).It is operated batch wisely; starting from extraction in the extraction vessel and ending at the solvent recovery vessel.The plant consists of:

i. Extraction vessel
ii. Evaporation vessel
iii. Thin film evaporator
iv. Condenser
v. Solvent recovery vessel

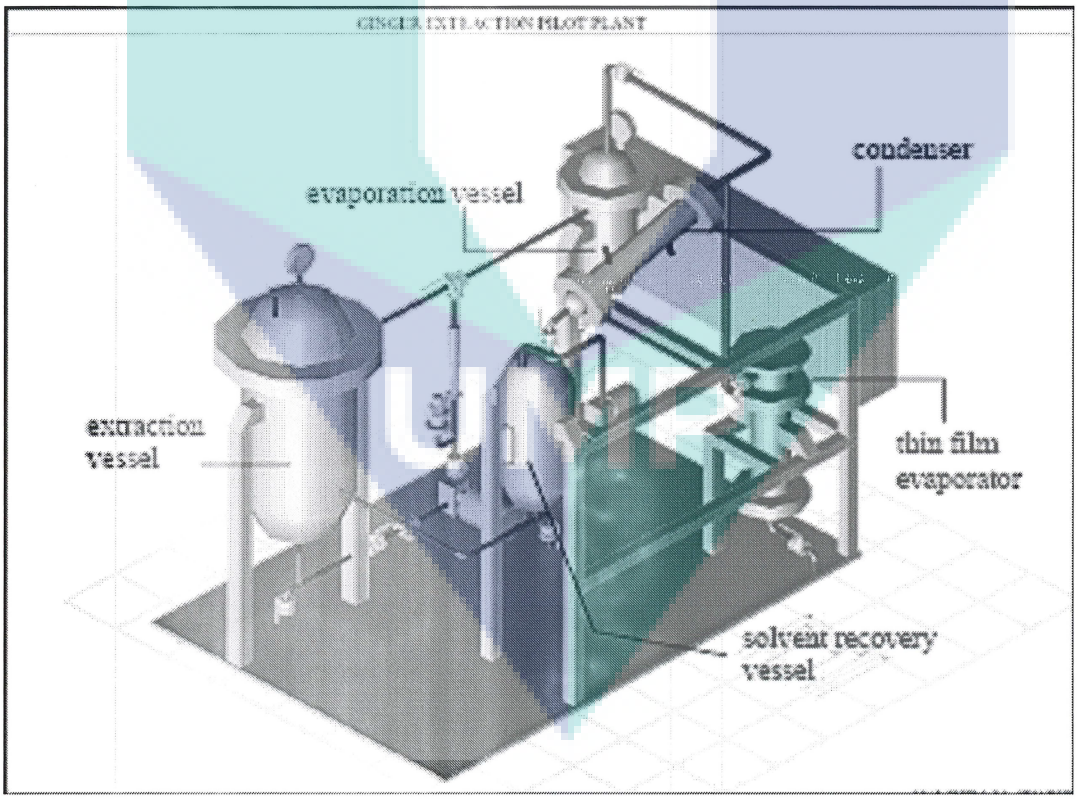


Figure 2.8 Ginger Extraction Pilot Plant

The extraction vessel capacity is 25L max. It is made of stainless steel 304 and provided with a basket for ginger. In this vessel, the ginger and solvent is mixed using a blower mixer. The blower mixer is created by process pump that discharges pressure through the inlet nozzle. An electrical clamp heater with the capacity of 8 kW is used as a heating medium and the setting temperature max. is 65°C.

Next is the evaporation vessel. The vessel is made of stainless steel 304. It is where evaporation occurs for the solvent. The heating medium is an electrical clamp heater with the capacity of 3.5 kW. The maximum is 150°C for setting temperature .

Thin film evaporator functions as a short path distillation unit where crude ginger oil that is mixed with small quantity solvent is separated to get the pure ginger oil and oleoresin. It is complete with variable speed motor, to rotate the rotor which creates a thin film at the wall in order to achieve separation. The shell side is made of stainless steel 304 while the rotor is made of graphite. A clamp heater with the capacity of 4 kW is used as heating medium. Aside from that, the max setting temperature is 100°C.

The condenser is a shell and tube exchanger with counter current design system. The shell and tube are both made of stainless steel 304. Cooling water is utilized as cooling medium with maximum temperature 32°C.

Lastly is the solvent vessel which is made of stainless steel 304.It is an empty vessel to recover the solvent from the condenser.

Apart from that, the equipment is designed following the listed utilities below:

i) Cooling water inlet temperature: max. 32°C
ii) Electricity voltage: 415 V
iii) Phase: 3 phases
iv) Frequency: 50 Hz

2.5 Rotary Evaporator (Lab Scale)

Rotary evaporators are also known as 'rotavaps' and commonly found in organic laboratories such as shown in Figure 2.9. They are used to remove solvents from reaction mixtures and can accommodate large volumes of liquid (up to 3 Liters). It is usually utilized to separate solvents such as hexane, acetone and ethanol from the essential oils produced in solvent extraction.

This equipment consists of several components such seen in Figure 2.10. The main parts of a rotary evaporator include a water bath, a speed motor, a condenser and a vacuum supply. A typical rotary evaporator has a water bath that can be heated in either a metal container or crystallization dish to keep the solvent from freezing during the evaporation process. Water or silicon oil is used as the heating medium. Besides that, the evaporator normally uses a variable speed sparkless induction motor that spins at 0- 220 rpm and provides high constant torque (Toreki, 2005).This enables the flask containing solution to rotate continuously according to the speed set as well as enhances the evaporation of solvent. Vacuum is used to evaporate the solvent while the condenser condenses the vapour trapped to liquid that is later collected for easy reuse or disposal. Most labs use a simple water aspirator vacuum on their rotary evaporators and therefore, a rotary evaporator cannot be used for air and water-sensitive materials unless special precautions are taken.

A vacuum is usually applied to the setup and this shows that the boiling points of the solvents are going to be significantly lower than at ambient pressure. Since the flask is rotated during the evaporation process, the surface area is larger which increases the evaporation rate. These two factors combined make it a very useful tool in synthetic chemistry to remove solvents. Apart from that, the need for lower temperatures also avoids overheating of the target compounds (Toreki, 2005).

Below are the technical specifications for a rotary evaporator and its water bath (Table 2.2):

Table 2.2: Technical specifications for components of a rotary evaporator

Rotary Evaporator	Water Bath
Speed range:20-190 rpm Vacuum: <1 mmHg Lift distance: 150 mm Dimension: (w x d x h) – 385 x 335 x 470 - 610mm, excluding glassware	Temperature range: ambient to 95°C Capacity: 3.5 Liters Heater power: 1300W Dimension: (w x d x h) – 260 x 280 x 200mm

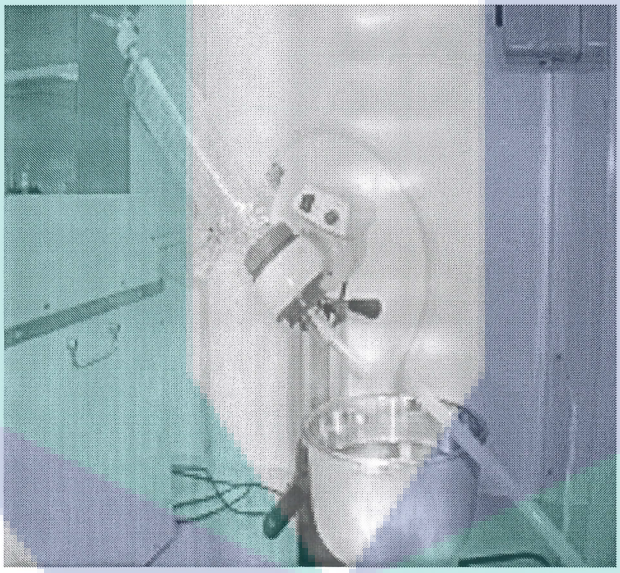


Figure 2.9 Rotary evaporator and its water bath

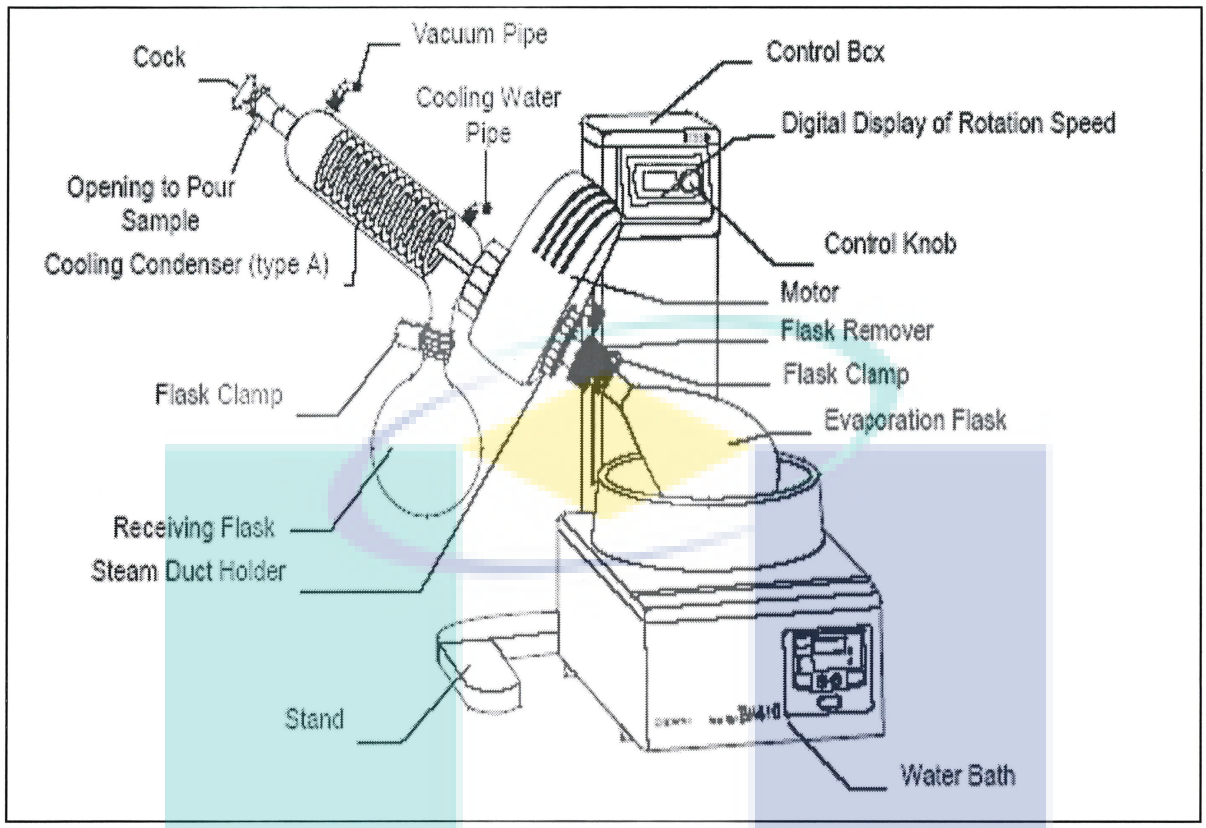


Figure 2.10 Components of a rotary evaporator (Toreki, 2005)

2.6 Analysis

There are different methods to analyze volatile components in essential oils. Two of the usual methods are:

i. Gas Chromatographic (GC) analysis
ii. High Performance Liquid Chromatography (HPLC) analysis

2.6.1 Gas Chromatographic Analysis

GC is used as an analytical tool to discover how many components are in a mixture (Figure 2.11). 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 in petrochemical (refinery gas), pharmaceutical (alkaloid street drugs) and environmental (chlorinated pesticides) analysis (Davisson, 2005).

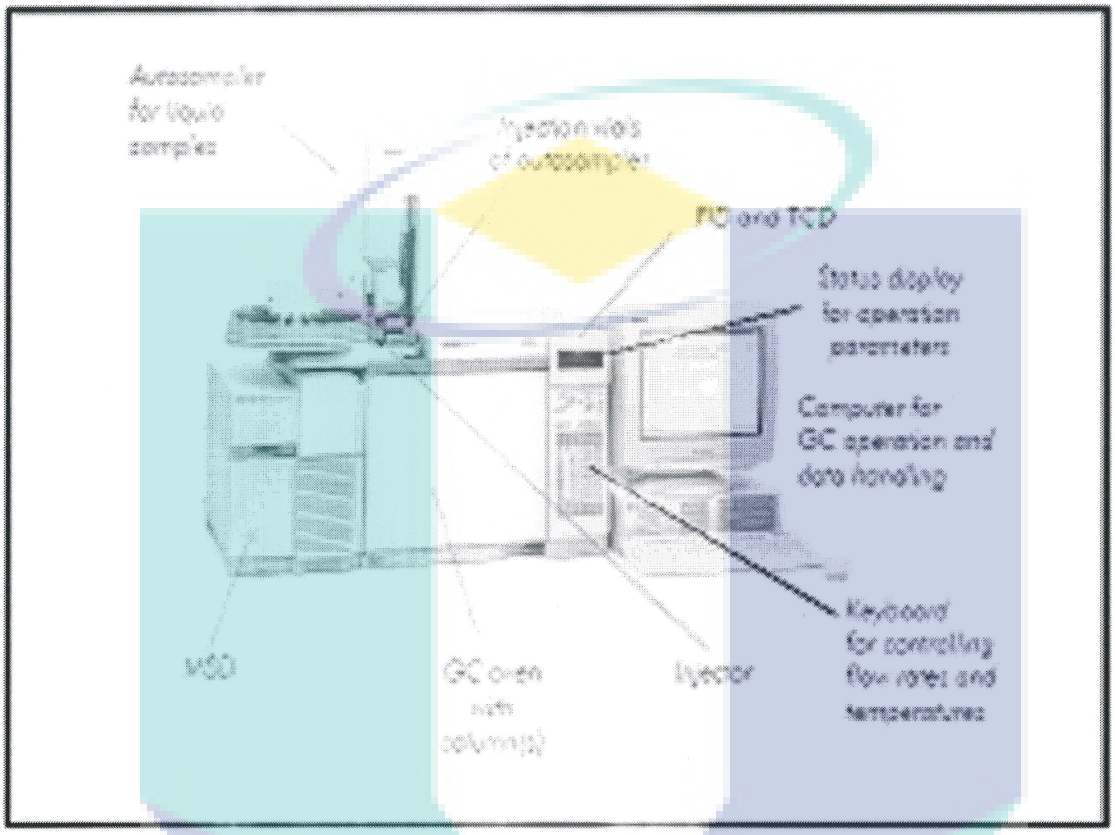


Figure 2.11 Gas Chromatography setup (Bramer, 1996)

2.6.1.1 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 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:

a. **Volatility of compound**: Low boiling (volatile) components travel faster through the column than high boiling components.

b. **Polarity of compounds**: Polar compounds move more slowly, especially if the column is polar.

c. **Column temperature**: Raising the column temperature speeds up all the compounds in a mixture.

d. **Column packing polarity**: Usually, all compounds move slower on polar columns, but polar compounds will show a larger effect.

e. **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.

f. **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.

2.6.1.2 Injection Systems 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 a 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 it 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 on 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 (Bramer, 1996).

2.6.1.3 Column Selection

Capillary columns are capable of more efficient separation. They enable more complex mixtures to be separated or resolved. As for packed columns, they have extremely high surface area, which is an advantage for large amounts of analyte like when separating gases. Capillary columns, however, have much greater resolution so they are more widely used for analysis. Illustrations are shown in Figure 2.12.

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 analytes. Other stationary phases are designed to interact with different types of functional groups (Bramer, 1996).

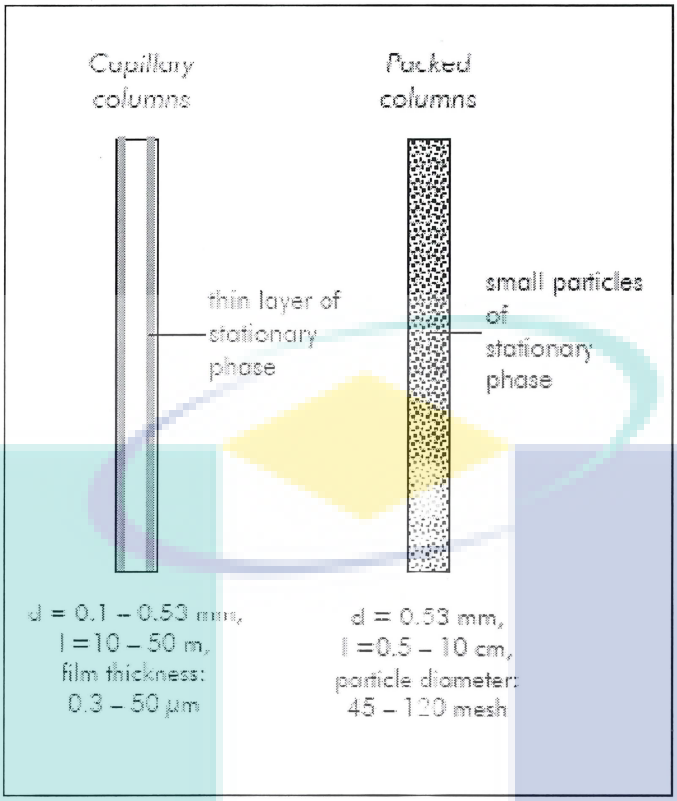


Figure 2.12 GC columns (Bramer, 1996)

2.6.1.4 Detectors Used In a Gas Chromatography

The differences and advantages of each is vital in selecting an analysis technique. Thermal Conductivity is the amount of heat conducted by the effluent. When the analyte exits the column and passes through the detector it changes the amount of heat conducted by the effluent. This detector is sensitive to almost any compound (this may be an advantage or a disadvantage).

Flame Ionization Detector (FID). The 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 and water.

Thermionic Emission Detector. This is a slight variation on the 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 compounds with nitrogen or phosphorous. This detector is only sensitive to nitrogen and phosphorous containing compounds.

Electron Capture Detector (EC). This is considered the strangest and most difficult detector to understand, but it is very useful for environmental 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 decreases. 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. These detectors also provide structural information about the analyte. This is very useful for identification of unknowns, but the techniques are not usually as sensitive as other detectors.

2.6.2 High Performance Liquid Chromatography Analysis

HPLC is useful for the analysis of compounds that are not volatile enough for GC analysis (Figure 2.13). The mobile phase is a liquid and separation is dependent upon the solubility of an analyte in the mobile phase.

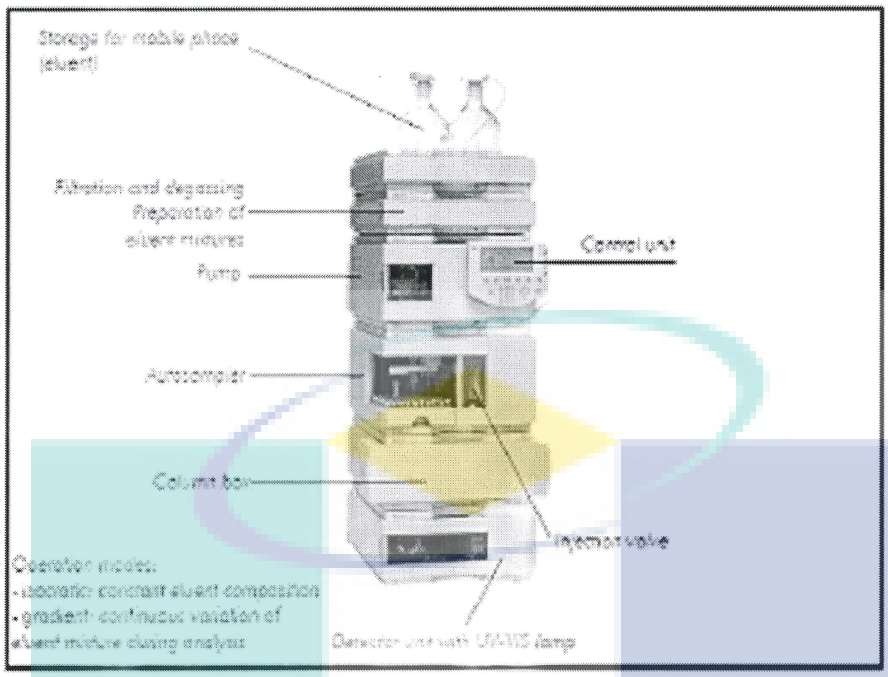


Figure 2.13 High Performance Liquid Chromatography setup (Bramer, 1996)

Typical types of liquid chromatography include:

a. Normal phase - use organic solvent for the mobile phase
b. Reverse phase - use an aqueous mobile phase
c. Ion chromatography - separation of ions

HPLC is utilized for several purposes such as Biotechnological (amino acids), Pharmaceutical (basic drugs) and Environmental (polycyclic aromatic hydrocarbons) analysis. Apart from that, HPLC has been used to analyze pungent components in ginger extraction using Supercritical Carbon Dioxide (Yonei & Ohinata, 1995). For sample preparations, ginger extracts were dissolved in hexane/ethyl acetate solution and pre-treated with sep-pack silica after the addition of methyl p-hydroxybenzoate. The instrument and analytical conditions are as follows:

i. Apparatus: HPLC system with a pump, UV-8000 detector
ii. Column: YMC-Pack A-014 Sil(S-5, 120 A, 6 mm X 300 mm)
iii. Detector: UV 280 NM

iv. Eluting solution: Hexane/ethyl acetate (9/1) for 16 minutes followed by hexane/ethyl acetate (7/3) for 16 minutes

v. Flow rate: 2 mL min^{-1}

Based on the experiment done by Yonei and Ohinata (1995), the chromatogram showed that 6-,8-, and 10- gingerols and 6- shogaol were confirmed as the pungent components. Among these components, 6-gingerol content was the largest as the peak was the highest. This also indicated that 6- gingerol is the representative of the pungent components in the extract

2.6.2.1 Column Efficiency

Column efficiency is highly dependent upon the packing size (5-10m diameter is common). The smaller the packing the higher the pressure required for a certain flow rate (typical pressures are several thousand PSI).

2.6.2.2 Column Selection

A typical column is made from a piece of 1/4" stainless steel tubing, 6 to 12 inches long and packed with 5 to 20m particles for the stationary phase.

Narrow bore and capillary columns are sometimes used as they have advantage of greater separation but less sample can be injected This makes the injection technique difficult and it provides less material for detection.

Guard Columns are used to protect the analytical (regular) column. This is a very short column, anything that would stick to the analytical column strongly enough to ruin it will be trapped on the guard column.

2.6.2.3 Detectors

Important considerations are: sensitivity (amount of analyte required for detection) and selectivity (what does it detect and not detect). Common detectors are:

UV-VIS. This is a UV-VIS spectrometer. The eluent flows into a very small cuvette that is part of a spectrometer. This cuvette is small so the path length is short (reduces sensitivity). This detector may be designed for a fixed wavelength, variable wavelength, or simultaneous detection at many wavelengths (using a diode array). The tradeoff between these different detectors is price. This detector responds to most compounds (anything that absorbs light at the set wavelength).

Fluorescent detectors. Detects fluorescence signal. Usually more sensitive than UV-VIS and more selective.

Electrochemical. This detector can be extremely sensitive and selective. However, it is also more difficult to use than UV-VIS or Fluorescent detectors.

Refractive index. This detects changes in the refractive index of the mobile phase as things elute. It is a very universal detector, but it is not very sensitive and it is difficult to align. (It is roughly equivalent to the thermal conductivity detector for GC)

Conductivity. This measures the resistance of the mobile phase. It is very useful for detecting ions (in ion chromatography). It is extremely sensitive for some ionic compounds.

HPLC/MS. This is an area of current development. The mass spectrometer provides structural information about the analyte as the peaks elute. The instrumentation is rather complex and expensive.

CHAPTER 3

METHODOLOGY

3.1 Ginger Extraction Using Rotary Evaporator (Lab Scale)

The processes involved in ginger extraction using a rotary evaporator were similar to the Ginger Extraction Pilot Plant. However, the experiment was done in a smaller scale which was considered as a lab study. The differences included the amount of raw material and solvents used, the equipment needed and the conditions of the extraction processes.

There were several safety precautions that had to be taken before operating a rotary evaporator. Among the safety measurements were:

Great care when raising the jacking mechanism. The jack had to be unlocked carefully while applying downward pressure to control the movement. This was to avoid damage to the mechanism since the jack rise rapidly without the weight of the glass.

All glassware had to be examined carefully for scratches, cracks and imperfections like chemical etching. Use of damaged glassware under vacuum could cause implosion. Therefore, the apparatus had to be operated behind a safety screen.

The equipment should not be used in hazardous atmosphere or for mixing of hazardous materials. Properties of the compounds had to be checked in order for the safety of the equipment and the experimenter.

A very large amount of organic solvent vapor was generated from the rotary evaporator. The solvent collection flask should always be cooled to minimize solvent loss and emptied regularly.

Personal protective equipment such as hand gloves, lab coat, goggle and respiratory unit should always be worn during experiments.

3.1.1 Extraction Process

Firstly, ginger rhizomes were sliced, weighed and dried using sunlight. The duration for drying was a day until moisture loss of 50% was achieved. 1 kg of dried ginger was then soaked in solvent (e.g. *n*-hexane, ethanol, diethylene glycol) in a beaker. Ratio of ginger to solvent was 1:3. The ginger was let to undergo extraction process for 24 hours.

3.1.2 Evaporation process

After 24 hours, the solvent was separated from the mixture solution of ginger and solvent using a rotary evaporator. Firstly, the cooling water in the condenser needed to be circulated. Then, the sample was put in the evaporation flask. The flask was filled to half of its capacity. Apart from that, liquid collected in the receiving flask should also be kept approximately half its capacity. Next, the bath temperature was set to the required degree and let to heat to the set point. The power was then turned on and the evaporation flask was moved gently into the water bath according to the appropriate operational position using the lift switch. After that, the control knob was set to rotate at the required speed. The vacuum was operated as well for evaporation of solvent. As soon as all the solvent had been evaporated, the feed cock was opened to release vacuum. Lastly, the evaporation flask containing ginger oil and the receiving flask containing solvent was removed. These steps were repeated for different kind of solvents (e.g. *n*-hexane, ethanol,

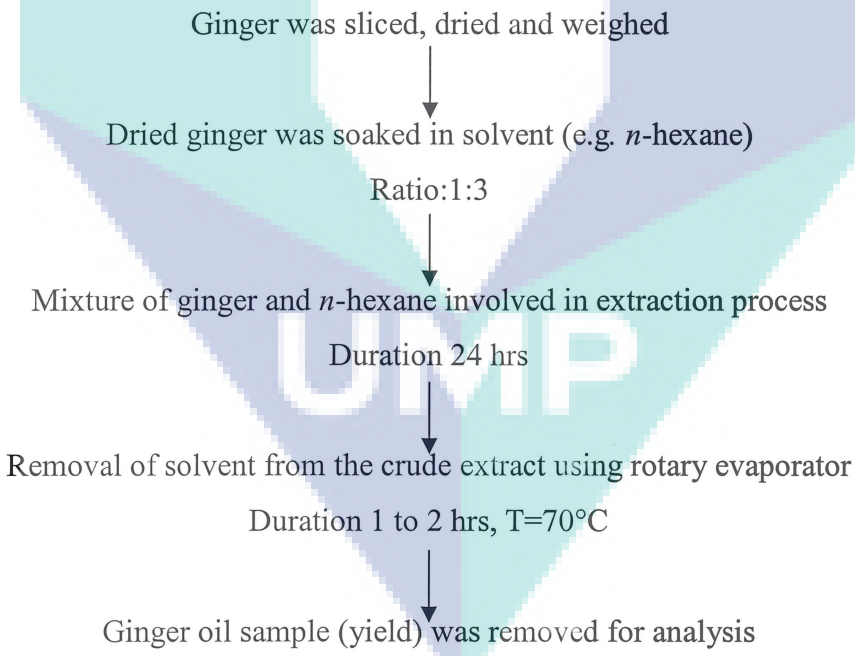
diethylene glycol) to study the effects of different solvent on yield. The ginger oil was later sent for analysis using Gas Chromatography.

3.1.3 Operating Conditions

Table 3.1: Operating conditions for each component of a rotary evaporator

Equipment	Condition
Rotary evaporator	Speed: 190 rpm max. Vacuum: <1 mmHg
Water bath	Temperature: 95°C Capacity: 3.5 L Heater power: 1.3kW

3.1.4 Flow Diagram for Ginger Extraction Procedure



3.2 Ginger Extraction Using Ginger Extraction Pilot Plant

3.2.1 Initial Safety Steps

There were several steps needed before the extraction process; initially valve positions and line tracing were checked. The line tracing included:

i Process/ product line
ii Vacuum line
iii Cooling water line
iv Sampling point

Next, all equipment involved were ensured to be in a safe condition which included the covers for extraction vessel and evaporation vessel were closely tightened. Then, all valves were closed. The control panel main switch was turned ON.

The most important precaution that had to be taken in operating the Ginger Extraction Pilot Plant (GEPP) was the use of Personnel Protective Equipment (PPE) such as:

i Hand gloves
ii Goggle
iii Safety helmet
iv Lab coat/Jacket
v Respiratory Unit

3.2.2 Extraction Process

Ginger and solvent (*n*-hexane) were fed in the extraction vessel C-01 for extraction process (See Figure 3.1). Ginger was dried and sliced. The ratio for ginger to solvent is 1:3.

There was a stainless steel bucket placed in the middle of the vessel which had smaller pores than ginger particles. The vessel utilized a tangential air blower to mix the solution and heat energy using a clamped heater. The vessel upper cover had a relief valve and pressure gauge system to avoid overpressure.

The extraction process was then carried out at atmospheric pressure. The immersion was done for a specified temperature such as 60°C and within a limit of time. Time was varied (e.g. 2, 4, 6, 8 hours) to study the effect of extraction time on yield.

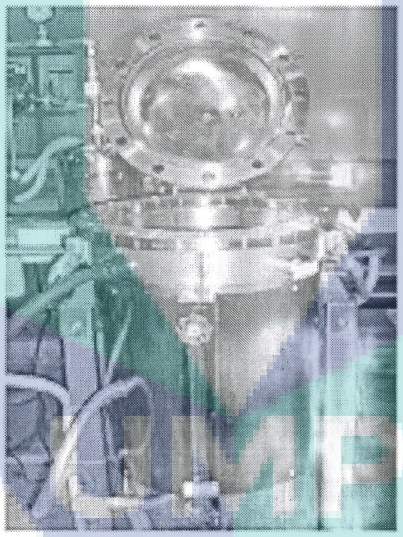


Figure 3.1 Extraction vessel

3.2.3 Evaporation /Separation Process

The crude extract in the extraction tank was pumped into the evaporator (See Figure 3.2). This was where the solvent was converted to vapor phase which passed through a packing above the evaporation vessel. Meanwhile, the solute was condensed into distillate and returned to evaporation vessel. Then, the vapor phase was condensed in a condenser to form condensate. This condensate was later returned to the solvent recovery tank to be reused in extracting solute from the solids (See Figure 3.3).

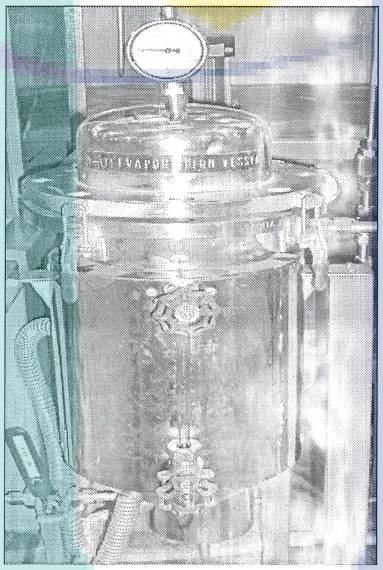


Figure 3.2 Evaporator

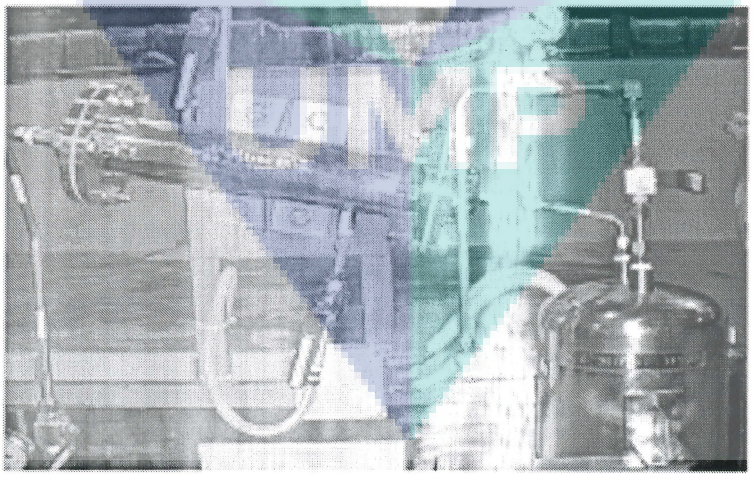


Figure 3.3 Condenser and solvent recovery vessel

3.2.4 Final Separation Process

After evaporation, the concentrated crude was pumped into the thin film evaporator for short path distillation (See Figure 3.4). This process was done to purify the crude from the last percent of remaining solvent. The yield (oleoresin) was then collected to be analyzed for its purity.

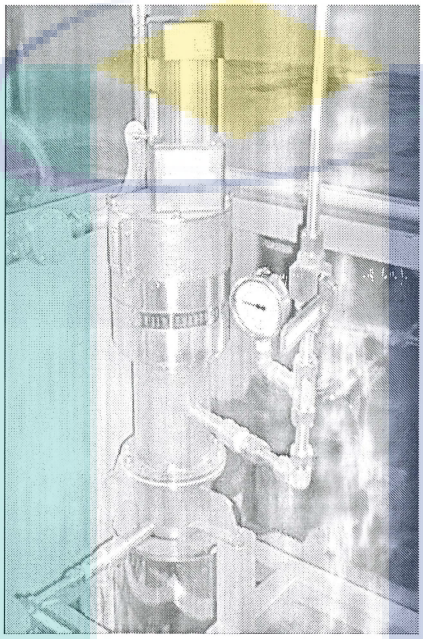


Figure 3.4 Thin film evaporator

3.2.5 Operating Conditions

Table 3.2: Operating conditions for each equipment of the Ginger Extraction Pilot Plant

Equipment	Condition
Extraction Vessel	Capacity: 25L max. Electrical capacity for clamp heater: 8 kW Setting temperature: 65°C max
Evaporation Vessel	Capacity: 5L

	Electrical capacity for clamp heater: 3.5 kW Setting temperature: 150°C max
Thin Film Evaporator	Electrical capacity for clamp heater: 4 kW Setting temperature: 100°C max Rotor: Graphite
Condenser	Cooling medium: cooling water Temperature: 32°C

3.2.6 Flow Diagram for Ginger Extraction Procedure

Ginger sample was weighed and placed into the extraction vessel

↓

n-hexane was filled into the vessel

↓

Mixture of ginger and n-hexane involved in extraction process

Duration 2 hrs, T=70°C

↓

Removal of solvent from the crude extract under vacuum

↓

The extract was transferred to a short path distillation

Duration 30 min, heating T=115 °C, cooling T= -5°C

↓

Oleoresin sample (yield) was removed for analysis

3.3 Gas Chromatography

The stationary phase was a high-boiling liquid which was usually a viscous oil or waxy substance. This high-boiling liquid was then packed into a long, narrow glass or metal column. Next, the mixture to be analyzed was loaded by syringe into the beginning of this column. Meanwhile, the mobile phase was an inert gas which continuously flowed through the column. The components of the mixture distributed between the stationary high-boiling liquid (these components were either condensed or absorbed on the high-boiling liquid) and mobile gas (vapor) phase moving through the column. The gaseous mixture flowed through a detector at the end of the column and finally, if it had been successfully separated, the components showed as different peaks on a recorder.

The ginger extracts were analyzed with the following instruments and conditions:

i Gas Chromatograph HP5890 (Hewlett Packard Co.)

ii Column: Fused silica capillary column, coated with PEG 20M(PC-WAX, 0.25mm i.d., 25 m length

iii Film thickness:0.25μm

iv Detector: FID

v Oven temperature: Programmed from 60°C (8 min) to 240°C at 3°Cmin^{-1}

vi Detector temperature: 250°C

vii Injection temperature: 250°C

viii Flow rate of carrier gas (He): 1 mL min^{-1}

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Process Optimization

This study mainly concerned of the design and functions of the multi-purpose extractor (Ginger Extraction Pilot Plant). Every part of the equipment contributed towards the quality and quantity of the products produced. Therefore, before operating the GEPP, several experiments were done to discover the appropriate conditions needed for ginger extraction and to know the pattern of the yield versus time curve. These experiments were executed using the same method of extraction which was solvent extraction, only in lab scale. The ratio of ginger to solvent was 1:3 while the temperature was fixed at 70°C. The following are result and discussion pertaining to the study:

4.2 Results

4.2.1 Ginger Extraction Using Rotary Evaporator (Lab Scale)

Figure 4.1 concerned with the change of yield percentage along with time when ginger was extracted using *n*- hexane as solvent. Based on the line graph, it clearly showed that the yield constantly increased with time. The lowest yield was at the starting time while the highest yield was at the ending time of extraction. Besides that, the highest increment occurred within the range of 46 to 48 minutes. The difference was 0.4% extract

in 2 minutes. Apart from that, there was only a little change that happened on the yield in the other ranges of time.

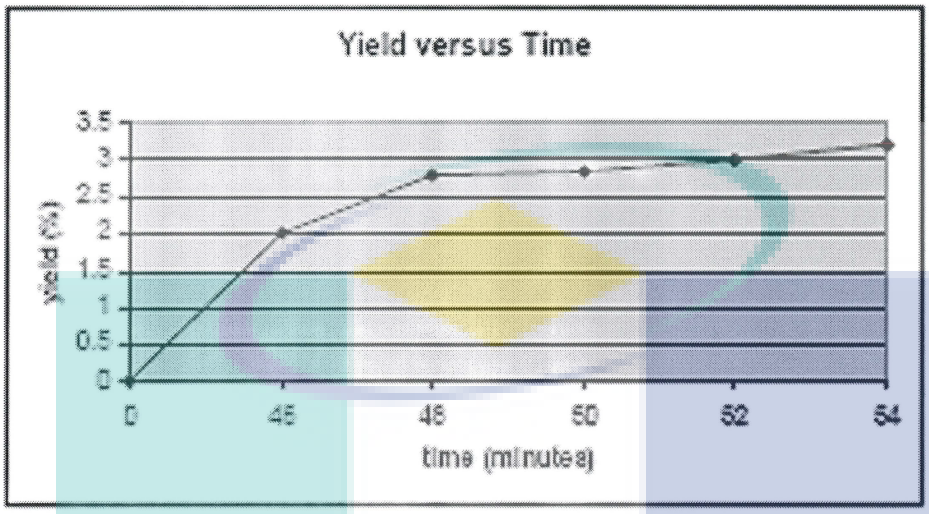


Figure 4.1 Graph yield versus time for ginger extraction using *n*-hexane

Figure 4.2 demonstrated the change of yield percentage along with time when ginger was extracted using a different solvent which was ethanol. It indicated that the yield initially increased as the time lengthened. However, after a period of time, the graph began to decrease and the decrement occurred at 160 minutes. The largest amount of yield was achieved at 150 minutes which was 2.24% extract.

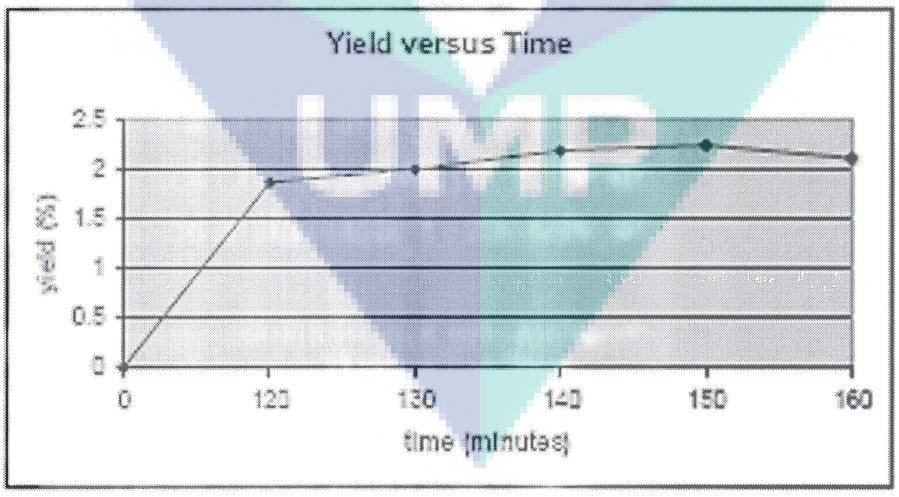


Figure 4.2 Graph yield versus time for ginger extraction using ethanol

Table 4.1 indicated the change of volume for solvent and ginger oil mixture after 160 minutes extraction time using the rotary evaporator. The solvent involved was diethylene glycol. Based on the readings, the amount of mixture only changed in the first range of time (0 to 40 minutes) and became constant after 40 minutes.

Table 4.1: Change of volume along with time for ginger extraction using diethylene glycol

SOLVENT	EXTRACTION TIME (MINUTES)	VOLUME OF MIXTURE (ML)
Diethylene Glycol	0	1000
	40	970
	80	970
	120	970
	160	970

4.2.2 Ginger Extraction Using Multi-purpose Extractor (Ginger Extraction Pilot Plant)

Figure 4.3 illustrated the change of yield percentage along with time when ginger was extracted using *n*-hexane in Ginger Extraction Pilot Plant. The diagram showed that the graph initially increased as the time increased but after achieving maximum limit, it immediately decreased. This occurred after 6 hours of extraction where the readings dropped 1.24% extract from 2.54% extract. Aside from that, the graph became constant beginning from 8 hours of extraction.

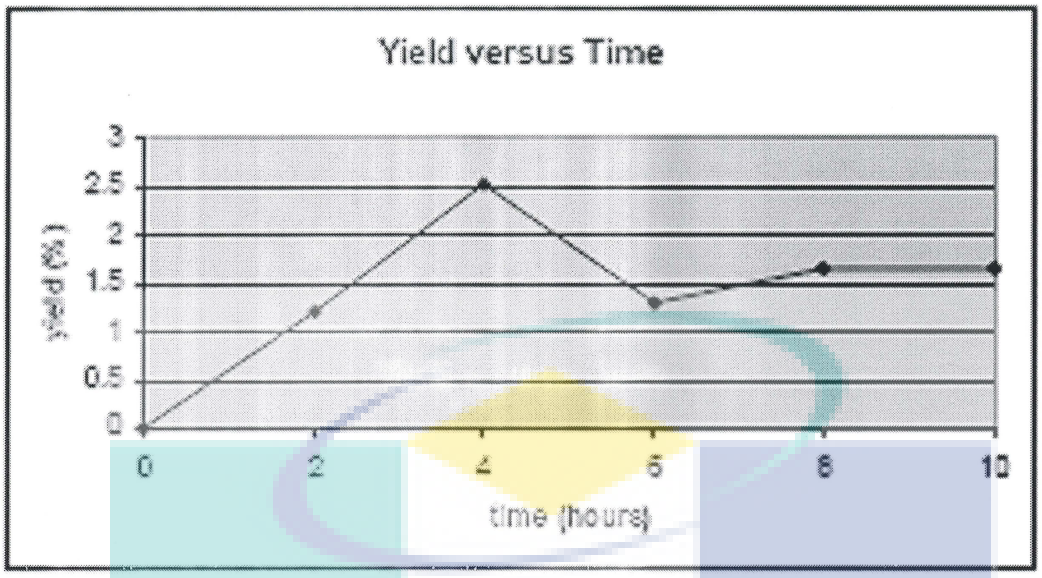


Figure 4.3 Graph yield versus time for ginger extraction using *n*-hexane

4.2.3 Gas Chromatography Analysis

Gas Chromatography analysis was done to detect the traces of *n*-hexane left in the ginger oil. Analysis was done for approximately 70 minutes. The chromatogram (See Figure 4.4), indicated peaks for *n*-hexane which functioned as a standard in this study. There are five evident peaks at the range of 10 to 14 minutes. The highest peak was at 10.701 minutes of retention time and the area was 9.99243e4 pA. Meanwhile, the lowest peak was at 61.613 minutes and the area was 1.57307 pA. Peaks of *n*-hexane appeared at almost every range of retention time except none at the range of 40 to 50 minutes (Refer Appendix A).

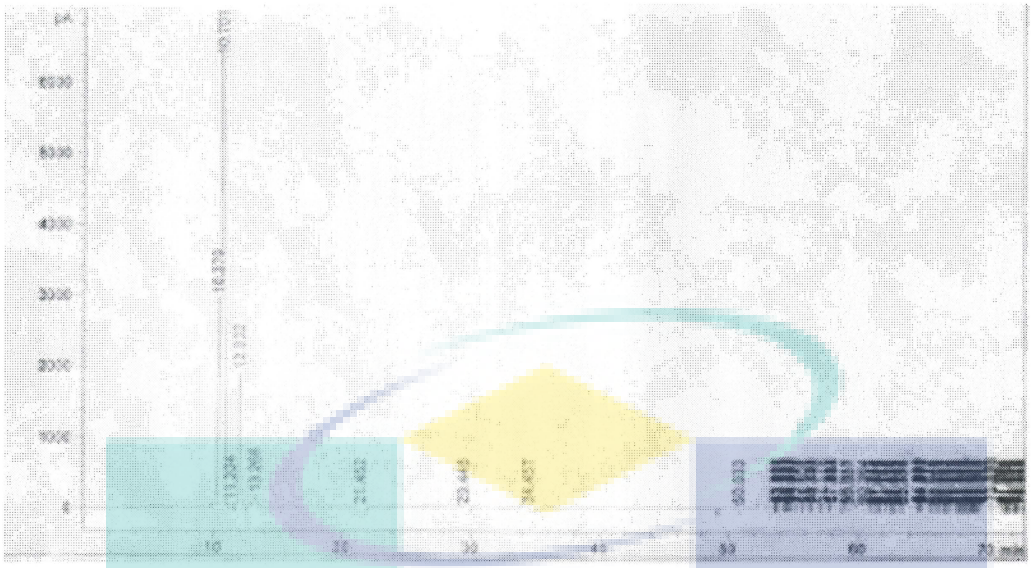


Figure.4.4 GC analysis of *n*-hexane

Another chromatogram (See Figure 4.5) demonstrated peaks for ginger oil obtained. There are five evident peaks also in the beginning of analysis. As the retention time lengthened, the peaks became shorter and less evident (Refer Appendix B). The highest peak was at 10.781 minutes and the area was 7.42053e4pA. Different readings exist at almost every retention time but the least area of 1.03014pA was found at 63.097 minutes.

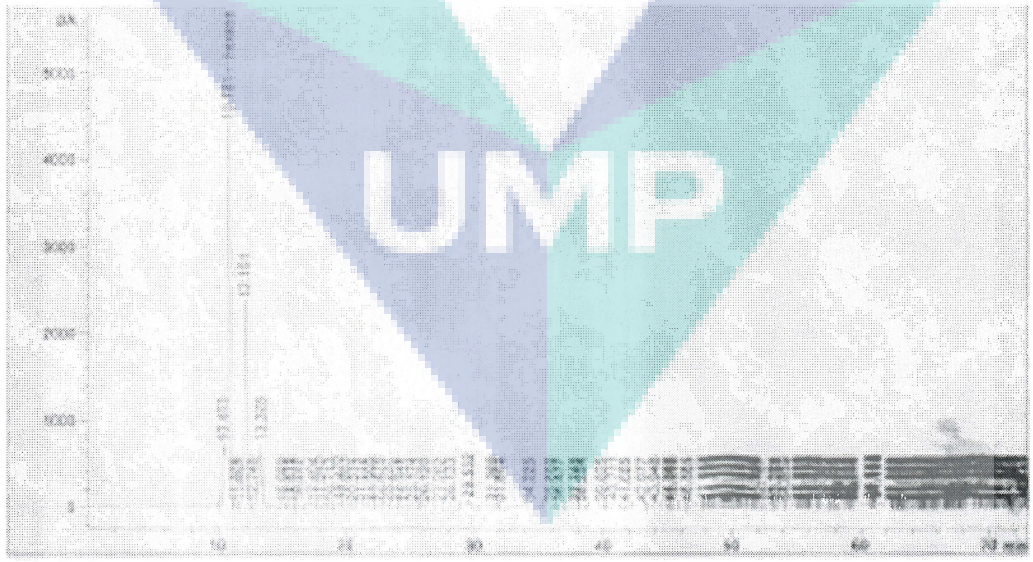


Figure.4.5 GC analysis of ginger oil

4.3 Discussion

4.3.1 Ginger Extraction Using Rotary Evaporator (Lab Scale)

Ginger oil was highly achieved using *n*-hexane as solvent. The extraction time was the shortest compared to other solvents such as ethanol and diethylene glycol. It took less than an hour to obtain the product. In the beginning, there was little yield as reaction between ginger and *n*-hexane had just started. Due to the *n*-hexane's low boiling point which was 69°C and high volatility, the solvent quickly vapourised when sufficient heat was applied. The amount of yield increased gradually as more solvent were being separated from the oil from time to time. After 46 minutes, even more oil was obtained. This was when the heat applied fully contacted with the ginger oil and hexane mixture, enabling the solvent to be vapourised in a short limit of time. Apart from that, the physical of the ginger oil obtained was the cleanest compared to those obtained from other types of solvents. It was clear and yellow in colour as well as having high viscousity. The ginger aroma was also present indicating that the yield consisted pungent and aromatic components such as shogaols and zingiberenes. These descriptions were justified by literature review as aroma/essential ginger oil was earlier described as being a clear light yellow oil, less peppery, with very strong fragrance. It is very suitable for food additive, cosmetic, aroma therapy, massage oil and pharmaceutical (Kingsing, 2005).

In ginger extraction using ethanol, the time taken for extraction was longer than *n*-hexane but shorter than diethylene glycol. This was proven as *n*-hexane produced 2%extract on the 46th minutes while ethanol took 130 minutes to produce as much yield. Ethanol was chosen to be used in this experiment as it was one of the most common solvent and also a good alcohol for extracting most lipids (Cyberlipid, 2005). Therefore, ethanol was considered a wise selection to be used in making a comparative study to discover the most suitable solvent for extracting ginger in the Ginger Extraction Pilot Plant. Initially, the mixture of ginger oil and ethanol reacted to the heat applied, causing the solvent to be vapourised. However, this process took a while as the boiling point of

ethanol (79°C) was higher than the temperature set (70°C). As the heat was insufficient, the separation of solvent from oil was difficult to be done. Therefore the yield was lesser than those using *n*-hexane as solvent. Besides that, the ginger oil produced was not clear and dark yellow to brown in colour. It also had low viscousity indicating that ethanol was not completely removed from the ginger oil.

Based on the table, diethylene glycol was proven not suitable to be used in ginger extraction. The ginger oil did not mix well with the solvent and it was more difficult to separate the two substances. Both ginger oil and diethylene glycol had high boiling points, 254°C and 244.8°C and the slight difference between them caused failure in separating the oil from the ginger and solvent mixture. In the beginning, there was a slight decrement in the readings. The temperature of the rotary evaporator was fluctuating (in the range of 65°C to 75°C), causing a certain portion of the mixture to be heated and thus vapourised. After 40 minutes, there was no change at all indicating that the reaction had completed. No product was obtained.

According to the experiments done on these three different solvents, the results showed that *n*-hexane was the best solvent to be used in the Ginger Extraction Pilot Plant. Apart from having the least extraction time due to low boiling point and high volatility, it possessed several interesting characteristics such as cheap (7 cents per pound) and abundant: every petroleum refinery on earth produces it, as it is an essential high-vapor pressure easily-ignited component of gasoline. Hexane was also considered "too efficient" as it extracts virtually every oil soluble fraction. The combination of extremely large availability, very low cost, and simple effectiveness had caused hexane to be commonly used in the extraction of edible oils (Miller, 2005).

Besides that, the results also showed that the rotary evaporator was not the most effective equipment to be utilized in ginger extraction. The operating temperature was limited to temperatures lower than 200°C. Therefore, certain substances, specifically solvents with high boiling points could not be separated from the oil mixture. In addition, the design of the rotary evaporator only allowed small amount of volume mixture to be

extracted at a time. Fluctuating temperature of the equipment (in the range of 65°C-75°C) also caused inaccurate readings as the heat supplied was insufficient to vapourise all of the solvent content.

4.3.2 Ginger Extraction Using Ginger Extraction Pilot Plant

Ginger Extraction Pilot Plant was designed to extract value added products from *Zingiber Officinale Roscoe* which are ginger oil and oleoresin. This equipment consisted of three unit operations and was operated batch wisely. Processes included extraction, evaporation and condensation. In this study, the pilot plant was used for the first time as soon as being completely fabricated. Thus, a few preliminary experiments were done to check whether the equipment was fully functional. Formerly, water was used as solvent to ensure every component including the pumps and valves were working well. Besides that, processes of extraction were done batch by batch to test the effectiveness of every equipment involved in producing yield. After being convinced that the pilot plant was safe and ready to be used, extraction of ginger using n-hexane was done. Firstly, the dried and sliced ginger was soaked in solvent in the extraction vessel for a limit of time. This was when the solvent interacted with the ginger surfaces and thus extracted the oil content in the ginger slices. 5 readings were taken at 5 different times (See Figure 4.3). As the time lengthened, more ginger surfaces were exposed to n-hexane and therefore enabling more oil to be pulled out from within. Then, the mixture of ginger and solvent was pumped into the evaporator where the solvent was vaporized. Next, the remaining mixture was further purified in the thin wall evaporator using short path distillation. Lastly, the oil was obtained and later analyzed while the vaporized solvent was condensed and recovered in the solvent recovery vessel.

Based on the result, the most optimum time for ginger extraction was at 4 hours as the yield was the highest at that time (See Figure 4.3). The yield produced by Ginger Extraction Pilot Plant was a bit different compared to those obtained from rotary evaporator. It was clear, dark amber to orange in colour and had a pungent smell. It was

presumed to be a mixture of ginger oil and oleoresin. As described in literature review, ginger oleoresin is reddish orange in color and possesses flavour and taste that is just as fresh ginger style, but much more strong than fresh ginger. Besides that, it is peppery and fragrant. There is more than 15% gingerols in this kind of oil which made it more valuable in the food additive and pharmaceutical industry (Kingsing, 2005). After 4 hours of extraction, the yield began to decrease. Volatile components in the mixture, including the ginger oil were vaporized along the extraction process as there was heat accumulated in the equipment as the time lengthened. All of the heat could not be released to atmosphere as there was no cooling system such as a heat exchanger attached to the equipment.

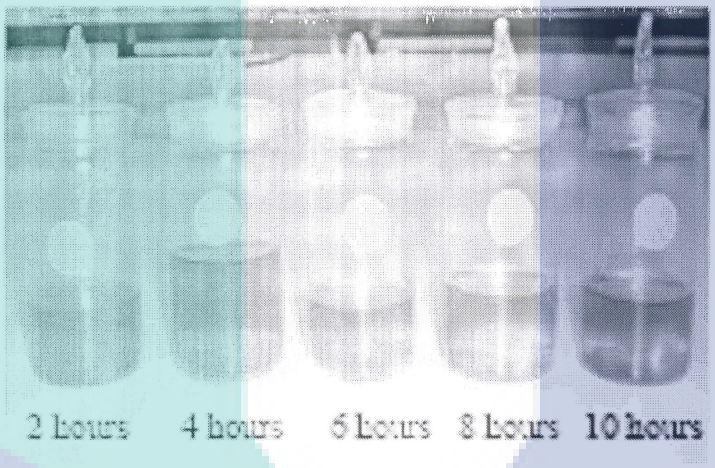


Figure 4.6 Yield (ginger oil and oleoresin) at different extraction times

Figure 4.7 Maximum yield (ginger oil and oleoresin)

4.3.3 Gas Chromatography Analysis

The two chromatograms (See Figure 4.4 and Figure 4.5) were being compared to know whether *n*-hexane does exist in the ginger oil sample after the purification process. The highest peak was at 10.701 minutes indicating the existence of *n*-hexane. During this time, the sample was initially injected in the GC column and *n*-hexane was used for washing the syringe. The other evident peaks at 10.379, 11.304, 12.302 and 13.266 minutes (Refer Figure 4.4 and Appendix A) were considered impurities in the sample. Impurities also existed at 10.463, 11.339, 12.748 and 13.328 minutes for the ginger oil sample (Refer Figure 4.5 and Appendix B). There are a number of components in the extract but could not clearly be established in Figure 4.5. The sample might contain ginger oil constituents but could not be identified as the peaks were not clear. According to literature review (Yonei and Ohinata, 1995), the principal constituent of volatile oil which was zingiberene, a kind of sesquiterpene hydrocarbon was discovered highest at the range of 40 to 50 minutes of retention time. The result obtained in this study (Figure 4.5) showed readings along 40 to 50 minutes (Refer Appendix B) but the peak did not appear clearly. Therefore, not much pure ginger oil was considered collected. The differences occurred as this was a preliminary study to test the effectiveness of Ginger Extraction Pilot Plant in producing yield. Appropriate operating conditions such as suitable temperature, pressure, volume and type of GC columns needed were yet to be investigated. These parameters were important in determining the quality and quantity of the yield.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The multi-purpose extractor is effective to be used. Based on the study done, it was discovered that this technology enables shorter extraction time for the extraction of ginger. The optimum time for producing ginger oil and oleoresin using this equipment is 4 hours which is shorter than the one in the conventional method. The conventional solvent extraction takes 8 hours to produce yield. Apart from that, a large amount of ginger could be extracted at a time as the processes are conducted in pilot scale. Therefore, the use of multi-purpose extractor is economical as it could save time and operational cost.

Besides that, the best solvent for ginger extraction is *n*-hexane. When being compared to other solvents such as ethanol and diethylene glycol, it was proven that *n*-hexane could extract more ginger oil and oleoresin than the others. It also takes shorter time for extraction process. The yield appeared to be clear yellow to orange in colour, similar to the descriptions in the literature review. The aroma is present in the yield too, indicating the existence of volatile constituents such as zingiberenes. These constituents are especially valuable in the medicinal field as they could be the antidote for several diseases.

Lastly, the use of multi-purpose extractor enhances the purification process in ginger extraction. The equipment is quite practical to be utilized to achieve least residual solvent in the final product. This technology could be a new alternative in separating

traces of solvent from the extract after all the appropriate operating conditions were successfully identified. However, in this preliminary study, not much pure ginger oil and oleoresin were obtained. The ginger oil and oleoresin are considered pure and of better quality when there is no traces of remaining solvent.

5.2 Recommendations

There are a few recommendations provided to improve the system of the multi-purpose extractor. Firstly are the scales; proper scales need to be done on each vessel to ensure accurate measurements of the fluid involved. Level indicators should be placed on the front of the vessel to make it easier to take readings during experiments. Besides that, the equipment should be designed with an effective cooling system to avoid overheating of the vessels, especially in the extraction vessel. A heat exchanger could be attached to the equipment to reduce the heat accumulated along the extraction process. Next is the condition of the pressure and temperature indicators. These indicators should be checked from time to time to ensure they are fully functional. Error on the indicators could cause unwanted mishaps such as overpressure which could lead to explosions. Automatic relief valves need to be constructed to release pressure when there is pressure build up in the vessels such as at the extraction vessel and at the evaporator. Every vessel, pipe line, pump and valve should be checked before the pilot plant is operated. Any damages such as leaks have to be corrected as soon as possible to avoid chemical and product loss. Sufficient Personal Protective Equipment (PPE) should be provided and used by the experimenter at all times during operating the pilot plant. This is to avoid from being overexposed to toxic chemicals, specifically solvents like *n*-hexane. Alternative solvents such as acetone could be used to replace *n*-hexane. More experiments and researches involving various parameters such as pressure, temperature and concentration need to be conducted in order to discover the optimum conditions for producing a high quality and quantity yield.

LIST OF REFERENCES

Badalyan A.G., Wilkinson G.T.and Byung-Soo Chun (1998). Extraction Of Australian Ginger Root With Carbon Dioxide And Ethanol Entrainer. *Journal of Supercritical Fluids*. Vol. (13): 319-324.

Bramer S.E.V. (1996). *Overview Of Chromatography*.www.sciencewidener.edu

Bone, M.E.(1990). Ginger Root - A New Antiemetic. *Anesthesia* 45(8): 669

Cyberlipid (2005). *Lipid Extraction*.www.cyberlipid.org

Farlex (2004). *Zingiber*. www.encyclopedia.thefreedictionary.com

Feist (2000). *Organic Laboratory Techniques, 3rd Ed.* Fessenden, Fessenden, Feist, Brooks/Cole

Fischer W. (1995).*Short Path Vacuum Distillation From Laboratory To Production.*www.UIC-GmbH.com

Floridata (2003). *Zingiber Officinale*. www.floridata.com

Foster S. (2000). *Ginger Your Food Is Your Medicine.* www.stevenfoster.com

Isbell T. A. and Cermak S.C. (2004). Purification of Meadowfoam Monoestelide From Polyestolide. *Industrial Crops and Products*. Vol. (19.): 113-118.

Kingsing (2005).*Ginger oil, Oleoresin and Powder Extract.* www.ecplaza.net

McCabe W.L., Smith J.C. and Harriot P. (2001).*Unit Operations of Chemical Engineering.* MacGraw Hill. 739-756.

McMahon C. (2005). *Methods Of Extracting Essential Oils.* www.naturegift.com

Miller R. (2005). *Edible Oil Extraction: Severe Limitations Of Hexane (Solvent Extraction).* www.pnpi.com

Mowrey and Clayson (1982).Motion Sickness, Ginger And Psychophysics. *Lancet.* (20): 655-667.

Naik S.N., Lentz H. and Maheshwari R.C. (1989). Extraction Of Perfumes And Flavours From Plant Materials With Liquid Carbon Dioxide Under Liquid –Vapor Equilibrium Conditions. *Fluid Phase Equilibria*. Vol. (49.): 115-126.

Noble R.D. and Terry P.A (2004). *Principles Of Chemical Separations With Environmental Applications.* Cambridge University Press.1-3, 14-18 & 120-127.

Nurul Azlina Muhamad (2004). *Process Design Of Hydro-Distillation In Ginger Oil Production.* The 4th Annual Seminar of National Science Fellowship 2004.90-94.

Toreki R. (2005). *Rotary Evaporators*. www.ilpi .com

Undurraga D., Markovitz A. and Erazo S. (2001). Cocoa Butter Equivalent Through Enzymic Interesterification Of Palm Oil Midfraction. *Process Biochemistry*. Vol. (36.): 933-939.

Yonei Y. and Ohinata H. (1995). Extraction Of Ginger Flavor With Liquid Or Supercritical Carbon Dioxide. *Journal of Supercritical Fluids*.Vol. (8.): 156-161.

APPENDIX A

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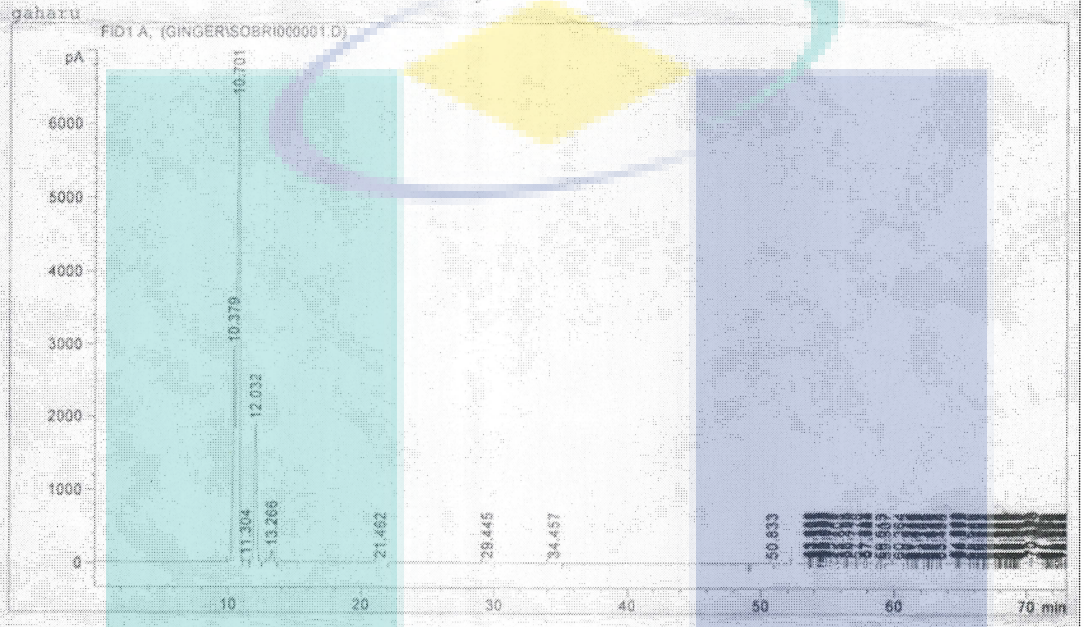
gaharu



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53.881	VV	135.94693	0.00000	0.00000		
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Instrument 1 28/10/2005 10:14:05 Hasbi Page 1 of 3

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54.445	VV	54.64986	0.00000	0.00000		
54.469	VV	45.12196	0.00000	0.00000		
54.520	VV	64.90832	0.00000	0.00000		
54.550	VV	115.99841	0.00000	0.00000		
54.647	VV	82.00054	0.00000	0.00000		
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54.768	VV	120.77190	0.00000	0.00000		
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55.240	VV	180.42570	0.00000	0.00000		
55.808	VV	37.77260	0.00000	0.00000		
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APPENDIX B

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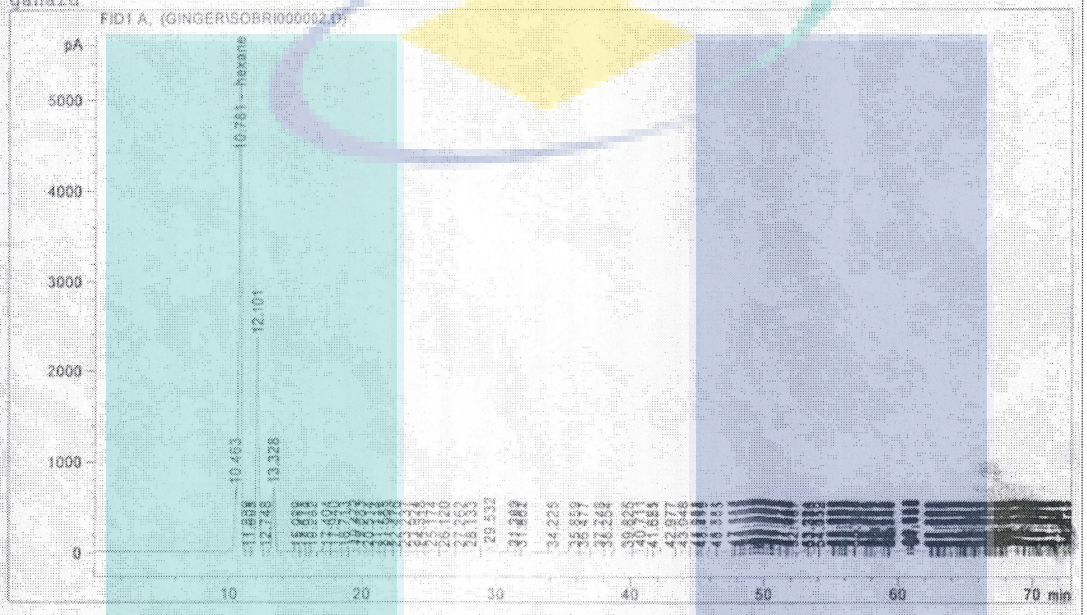
gaharu



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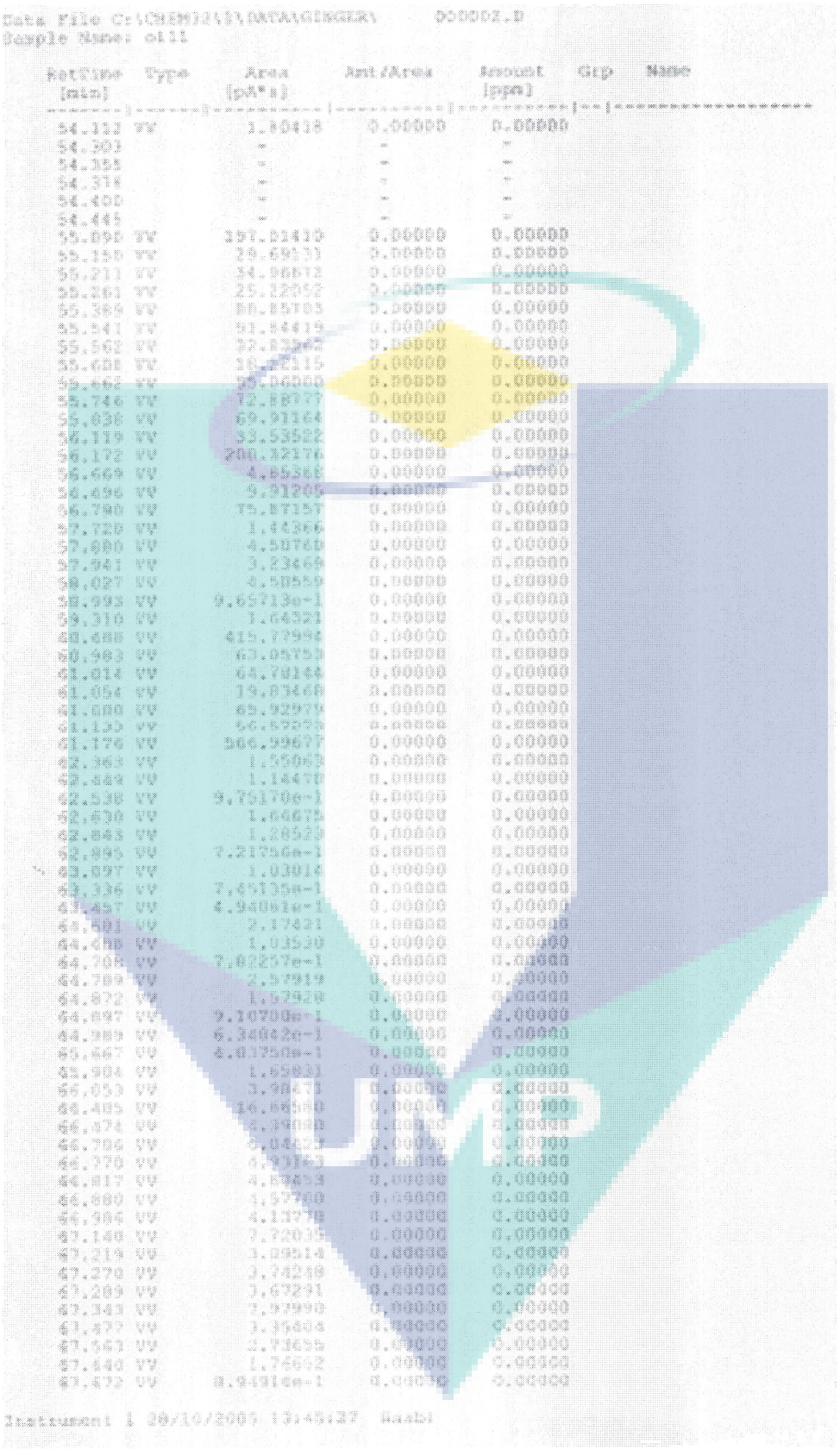
Instrument 1 28/10/2005 13:45:27 Hasbi Page 1 of 3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APPENDIX C

Gantt Chart

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Receive chemicals																
Collect raw materials																
Sample preparations																
Experimentation and data analysis																
Thesis writing																
Complete project writing																

PUBLICATIONS

1) **Ahmad Ziad Sulaiman, Rosli M.Y.** *"Effect of Sonication for In-Situ Stainless Steel Leaf Filtration,* Journal of The Institution of Engineers, Malaysia Vol. 67, No. 1, March 2006

2) **Sulaiman A.Z, Yunus, R.M, Azilah Ajit.** *"Effect of Sonication for In-Situ Stainless Steel Leaf Filtration,* Advanced Membrane Technology III: Membrane Engineering for Process Intensification, Cetraro Calabria Italy (11-15 June 2006)

3) **Sulaiman A.Z**, Yunus,R.M, A.A.Badhrulhisham,M.Y.Abu Azam. *"The Effect of Ultrasonic Wave Fields for In-Situ Stainless Steel Leaf Filtration,* 10th Asia Pacific of Chemical Engineering Conference (APPChE) 2004, Kitakyushu, Japan.

4) **Sulaiman A.Z**, Yunus,R.M, R.Junin. *"The Study of the Effectivess of High Intensity Ultrasonic (HIU) to Increase the Rate of Filtration in palm Oil Industries,* 4th Annually Seminar National Science Fellowship (NSF) 2004, Ministry of Science, Technology & Innovation, Malaysia, Vistana Hotel Penang.

PATENT

Sulaiman A.Z, Rosli M.Y. In-Situ High Intensity Ultrasonic (HIU) Asissted for Stainless Steel Leaf Filtration. **Malaysian Patent Identification Number : PI 20032885**

AWARDS

1. Gold Medal Award in the 34th International Exhibition Inventions New Techniques and Products Geneva,Switzerland from 5 – 9 April 2006 for project "Mobile Ultrasonic Ginger Extraction System"

2. Gold Medal Award "Mobile Ultrasonic Ginger Extraction System" IPTA R&D EXPO 2005, World Trade Centre (October 2005)

3. Silver Medal Award "Mobile Ultrasonic Ginger Extraction System" 16th International Invention Innovation Industrial Design & Technology Exhibition 2005 (ITEX 2005) (May 2005)

4. Gold Medal Award "Pilot scale of the production of value-added product from Zingiber Officinale Roscoe" KUKTEM Research Exhibition Fair 2004. (October 2004)