

STUDY ON IMPORTANT PARAMETERS AFFECTING THE  
HYDRO-DISTILLATION FOR GINGER OIL PRODUCTION

NURUL AZLINA BINTI MOHAMED

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*Dedicated to my beloved father, Dato' Hj Mohamed Omar for his constant encouragement and motivation. To my beloved mother, Datin Hj Semah Kasim for her inspiration and to my forever supportive family members.*

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## ABSTRACT

Ginger or its scientific name *Zingiber officinale Roscoe* are extensively used globally for food flavourings, condiments, fragrances, aromatherapy and pharmaceuticals. There are two main products from ginger; (i) ginger oleoresin and (ii) ginger oil. Currently, there are a few conventional and modern methods of extracting essential oils such as by hydro-distillation, supercritical fluid extraction and microwave extraction. Hydro-distillation is the oldest and most common method of extracting essential oil since it is economically viable and safe. In this research, studies were done to identify the ideal operational conditions involved in the extraction of ginger oil by hydro-distillation; steam and water distillation. Work done in this research identified that the ideal operating conditions involved were the temperature ( $100^{\circ}\text{C}$ ) and pressure (1 atm) of the operating vessel, sample type (ground and 90% dryness), extraction method (steam distillation), extraction time (4 hrs), ratio of water : ginger (7L:400g), packing height (1cm) and tray height (4.5cm) based on the yield of the ginger oil. From the vapour pressure study, the boiling point of ginger oil is  $141.0^{\circ}\text{C}$  and the boiling point of the mixture is  $97.5^{\circ}\text{C}$ . Through analysis of ginger oil using the Differential Scanning Calorimeter, the  $C_p$  value starts to decrease at  $85.8^{\circ}\text{C}$  and this means that the ginger oil starts to decompose at this point. At the end of this research, some recommendations were given in improving the current equipment used in Malaysia for the extraction of ginger oil and help boost the Malaysian herbal industry.

## ABSTRAK

Halia atau nama saintifiknya *Zingiber officinale Roscoe* selalunya digunakan secara global dalam perasa makanan, minyak wangi, aromaterapi, farmasi and sumber bahan kimia. Terdapat dua produk utama daripada halia; (i) oleoresin halia dan (ii) minyak pati halia. Pada masa ini, ada beberapa kaedah tradisional dan moden bagi mengekstrak minyak pati halia seperti penyulingan air, pengekstrakan bendalir genting lampau dan pengekstrakan ketuhar gelombang. Penyulingan hidro adalah kaedah yang paling lama dan selalu digunakan kerana ianya murah dan mudah dikendalikan. Dalam penyelidikan ini, kajian dijalankan bagi mengenalpasti keadaan operasi yang unggul terlibat dalam mengekstrak minyak pati halia melalui proses penyulingan hidro; penyulingan berstim dan air. Kerja-kerja yang dilakukan dalam penyelidikan ini telah mengenalpasti parameter operasi unggul iaitu suhu ( $100^{\circ}\text{C}$ ) dan tekanan (1 atm) alat operasi, jenis sample (dicanai dan 90 peratus kekeringan), kaedah pengekstrakan (penyulingan berstim), masa pengekstrakan (4 jam), nisbah air : halia (7L:400g), tinggi padatan (1cm) and tinggi dulang (4.5cm) bergantung kepada hasil minyak pati halia. Daripada kajian terhadap tekanan wap, takat didih minyak pati halia ialah  $141.0^{\circ}\text{C}$  dan takat didih campuran ialah  $97.5^{\circ}\text{C}$ . Melalui analisis minyak pati halia menggunakan Differential Scanning Calorimeter, nilai  $C_p$  mula merosot pada suhu  $85.8^{\circ}\text{C}$  dan ini bermakna minyak pati halia mula terurai pada suhu tersebut. Di akhir penyelidikan ini, beberapa cadangan telah dikemukakan bagi pembaikan ke atas alat terkini yang digunakan di Malaysia bagi mengekstrak minyak pati halia dan membantu dalam meningkatkan industri herba Malaysia.

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## LIST OF SYMBOLS AND ABBREVIATIONS

Bp	-	Boiling point
C	-	Carbon
FRIM	-	Forest Research Institute Malaysia
LM	-	Light Microscopy
MARDI	-	Malaysian Agricultural Research and Development Institute
ML	-	Moisture loss
P	-	Pressure
SFE	-	Supercritical fluid extraction
T	-	Temperature
UV	-	Ultraviolet
VLE	-	Vapour Liquid Equilibrium
$\alpha$	-	Alfa
$\beta$	-	Beta
$d$	-	Delta
$\varepsilon$	-	Open void fraction
$D_{AB}$	-	Diffusivity of water in ginger oil
$\tau$	-	Tortuosity
$c_{A1}$	-	Concentration of solute in solvent at point 1
$c_{A2}$	-	Concentration of solute in solvent at point 2
$z_2$	-	Diffusion path at point 2
$z_1$	-	Diffusion path at point 1

$p_T$	-	Total vapour pressure
$p_A$	-	Partial vapour pressure of A
$p_B$	-	Partial vapour pressure of B
$y_A$	-	Mole fraction of component A
$y_B$	-	Mole fraction of component B
$n_A$	-	Number of moles A
$n_B$	-	Number of moles B
$n_T$	-	Total number of moles in the distillate
$m_A$	-	Mass of A
$MM_A$	-	Molecular mass of A
$m_B$	-	Mass of B
$MM_B$	-	Molecular mass of B
$m_{water}$	-	Weight of water
$m_{EO}$	-	Weight of ginger oil
$y_A$	-	Composition of the vapour
$x_A$	-	Composition of the liquid
$\alpha$	-	Volatility
$q$	-	Heat removed per unit time
$U$	-	A constant depending on operating conditions
$A$	-	Area available for removal of heat
$\Delta t$	-	Temperature difference between the hot vapour and the cooling medium
$C_{ps}$	-	Specific heat capacity of the sample
$C_{pr}$	-	Specific heat capacity of the reference
$Y_s$	-	Difference between the DSC curves of the empty container and sample
$Y_r$	-	Difference between the DSC curves of the empty container and reference

$M_r$	-	Weight of the reference
$M_s$	-	Weight of the sample
$\rho$	-	Density of water
$A$	-	Cross-sectional area of the vessel
$h$	-	Height of the water inside the vessel
$\dot{m}$	-	Flowrate of water
$t$	-	Extraction time
$x$	-	Amount of water distilled
$Q_A$	-	Heat absorbed by the system
$m$	-	Mass
$C_p$	-	Heat capacity
$\Delta T$	-	Temperature difference
$Q_R$	-	Heat released from the system
$n$	-	Number of moles of water
$Q_S$	-	Energy used by the system in this process
$Q_A$	-	Total energy supplied by the heater
$t$	-	Optimum operating time
$\xi$	-	Equipment efficiency

## GLOSSARY

<b>Adaptogenic</b>	Plants with properties that exert a normalizing influence on the body, neither over-stimulating nor inhibiting normal body function, but rather exerting a generalized tonifying effect.
<b>Antioxidant</b>	Substance that prevents or slows the breakdown of another substance by oxygen.
<b>Aromatic</b>	Any chemical that has aroma or flavour properties.
<b>Boiling point</b>	The temperature at which the vapour pressure of a liquid is one atmosphere.
<b>Bottom Note</b>	The characteristic left when top and middle notes disappear; the residue when a flavouring evaporates.
<b>Error</b>	In a statistical interpretation the word 'error' is used to denote the difference between an observed value and its 'expected' value as predicted or explained by a model. In addition, errors occur in data collection, sometimes resulting in outlying observations.
<b>Essence</b>	Concentrated fragrance or flavourant.

<b>Essential oil</b>	An oily substance obtained from plant material through various methods. The essential oil normally has the characteristic taste and odour of the plant from which it was derived. An essential oil is still called volatile oil as differentiated from a fixed oil. The hydraulically pressed sesame seed yields a fixed oil (sesame oil) that has low odour and is not volatile oil-oil of anise. Essential oils may have received their name because at one time they were thought to be essential to the life processes of the plant or that they were the essence of the plant.
<b>Experiment</b>	A set of measurements carried out under specific and controlled conditions to discover, verify, or illustrate a theory, hypothesis, or relationship. Experiments are the cornerstone of statistical theory, and are the only method for suggesting causal relations between variables. Experimental hypotheses cannot be proved using statistics; however, they can be disproved. Elements of an experiment generally include a control group, randomization, and repeat observations.
<b>Experimental Error</b>	Any error in an experiment whether due to stochastic variation or bias (not including mistakes in design or avoidable imperfections in technique).
<b>Extract</b>	A solution obtained by passing alcohol, or an alcohol-water mixture, through a substance. An example would be vanilla extract. Extracts such as orange, almond and lemon are essential oils dissolved in an alcohol-water mixture.
<b>Flash point</b>	The lowest temperature at which a flame will propagate through the vapour of a combustible material to the liquid surface. It is determined by the vapour pressure of the liquid, since only when a sufficiently high vapour concentration is reached, can it support



combustion. It should be noted that the source of ignition need not be an open flame, but could equally be, for example, the surface of a hot plate, or a steam pipe.

**Homeostasis** From the Greek words for "same" and "steady", homeostasis refers to any process that living things use to maintain stable conditions necessary for survival. In animals, this means maintaining a fine balance of the content of such vital ingredients as salt, water, sugar, temperature, blood pressure and oxygen. By studying how the brain maintains this homeostasis, scientists can determine why - in some cases - such a balance is upset as when the kidneys fail - and how to assist the brain in restoring the balance needed for health.

**Melting point** The temperature at which the vapour pressure of the solid and the liquid are the same and the pressure totals one atmosphere.

**Middle Note or Main Note** The substance of flavour; the main characteristic.

**Note** A distinct flavour or odour characteristic. For example, many raspberry flavour have a seedy note.

**Oleoresin** A resinous-viscous product obtained when a substance is extracted with a non-aqueous solvent such as hydrocarbon. The solvent is later removed. Spices as a class from most of the oleoresins that the flavorist encounters: an example would be oleoresin pepper.

**Parenchyma** These are cells in a tissue or tissues in an organ that are concerned with function.

**Refractive index** Of a medium, the ratio of the velocity of propagation of an

( $\eta$ , n)

electromagnetic wave in vacuum to its velocity in the medium.

*Synonym* index of refraction. *Note:* When the Greek character eta is unavailable, the letter n is used to represent the refractive index.

**Specific gravity**

The ratio of the density of a solid or liquid to the density of water at 4 degrees Celsius. The term can also refer to the ratio of the density of a gas to the density of dry air at standard temperature and pressure. It is a dimensionless quantity; it is not expressed in units.

**Top Note**

The first note normally perceived when a flavour is smelled or tasted. Usually a top note is relatively volatile and suggests identity.

**Vapour pressure**

The pressure exerted by a vapour in equilibrium with the solid or liquid phase of the same substance.

## CHAPTER 1

### INTRODUCTION

#### 1.1 Research Background

Nowadays, people worldwide are looking towards natural base products since there are no side effects when taken accordingly. Furthermore, there is also an interest in the production of functional, high value, natural products without chemical modification and residues of solvents or additives. This trend in consumer preference increases the demand tremendously with variety products range from essential oils. Essential oils, which are natural volatile extracts of plant materials, hold high export potential. Plant essences and extracts that have developed into our modern essential oils were in regular use in Rome, Greece, and Egypt and used throughout the Middle and Far East, for some centuries (Knowlton and Pearce, 1993). As a common feature, they possessed the essence of a plant, the identifiable aroma, flavour, or other characteristic that was of some practical use. They were used as perfumes, food flavours, deodorants, pharmaceuticals, and embalming antiseptics (Lawrence and Reynolds, 1984). Usually, plant material was steeped in a fatty oil or wine that acted as a solvent for the desired flavour or aroma (Chrissie, 1996). The extracts (usually impure and dilute) were used as oils or creams.

In Spain and France from the early 1300s, distillation was developed to produce more concentrated essences of rosemary and sage (Knowlton and Pearce, 1993). The demands of medieval pharmacy improved the distillation process. By 1550 (Knowlton and Pearce, 1993), different trends had become obvious. Spike lavender oil was being

produced in France for export as a trading commodity. Flavours and aromas were being distilled (Lawrence and Reynolds, 1984) or expressed (Chrissie, 1996) from an increasing number of new plant sources and pharmacists, chemists, and physicians were studying the physical, chemical, and medicinal properties of the oils.

Supercritical fluid extraction (SFE) is the latest technology in essential oil extraction. This process was hailed as nothing less than revolutionary when it was first introduced in the beginning of the 1980s (Chrissie, 1996). Although a potentially excellent method of extraction, producing oils whose aromas are closer to those of the living plant, the apparatus required for this operation is massive and extremely costly. It will also take years for the equipment to pay for itself; until such time, the cost of carbon dioxide extracted oils will remain very high.

Today, due to the increase in demand for essential oil base products, another method of extraction is being developed but currently under study. The new method is by microwave extraction (Soud, et. al, 2002). Eucalyptus leaves were used in this study to investigate the applicability of microwave irradiation for essential oil extraction.

In 1952, reports done by Guenther suggested that ginger oil could be extracted by hydro-distillation of dried ginger rhizome with an oil recovery of 1.5 to 3 percent. Recent reports done by Lawrence and Reynolds (1984) also mentioned that the volatile oil of ginger or ginger oil, which is an important raw material of the food, cosmetic and pharmaceutical industries is generally prepared by the steam distillation of dried comminuted rhizomes.

In Malaysia, the techniques currently practised in the industry for the extraction of essential oils are by hydro-distillation; steam, water and water / steam distillation and solvent extraction (Nor Azah Mohd. Ali, 2002). Locally, the Forest Research Institute Malaysia (FRIM), Malaysian Agricultural Research and Development Institute (MARDI), Universiti Kebangsaan Malaysia (UKM) and Universiti Teknologi Malaysia (UTM) are among the local institutes that do research on essential oil extraction on the local fruits, spices and medicinal plants. Among the research that has been done at FRIM is the extraction of essential oils from the lemongrass, patchouli, *Melaleuca cajuputi*, *Eurycoma apiculata* and *Zingiber* (Nor Azah Mohd. Ali, 2002). The essential

oils were extracted by hydro-distillation and solvent extraction. Figure 1.1 shows the equipment used for the purpose of extracting essential oil at FRIM. The equipment set up consists of a still proper, condenser and an oil separator. This equipment is able to function as a steam and water-distiller.

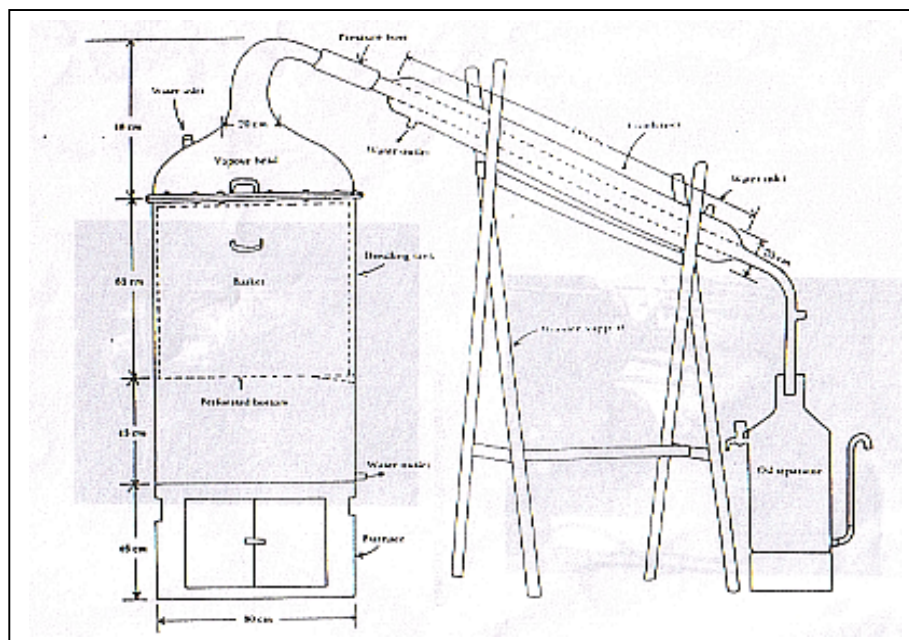


Figure 1.1: The steam distillation and water-distillation unit used at FRIM.  
(Courtesy of the Forest Research Institute Malaysia (FRIM))

MARDI on the other hand, has done research on the extraction of the tea tree oil. The tea tree oil is extracted by hydro-distillation at atmospheric pressure. Figure 1.2 shows the equipment used for the purpose of extracting essential oil at MARDI. It also consists of a still proper, condenser and a separator. All the equipment parts are made of stainless steel.

Moreover, Universiti Kebangsaan Malaysia has started their research on essential oils on various Malaysian plants since 1978. The essential oils of *Kaemferia galanga*, *Cinnamomum porrectum*, *Hyptis suaveolens* and *Zingiberaceae* were steam distilled (Laily Din *et al.*, 1988).



Figure 1.2: The hydro-distillation unit at MARDI.

(Courtesy of Malaysian Agricultural Research and Development Institute (MARDI))

All the institutes used hydro-distillation as the method of essential oil extraction. These methods were chosen since it is much more suitable for a developing country like Malaysia. The advantages of these techniques are that they are economically viable and safe to operate.

## 1.2 Objective and Scope

The objective of this research is to study the operational conditions involved in the extraction of ginger oil based on the yield of ginger oil using the hydro-distillation process so that recommendations on the improvements on the present design used in Malaysia can be made.

There are some important tasks to be carried out in order to achieve the objective of this study. Two important scopes have been identified for this research in achieving the objective:

1. Identification of the ideal conditions to produce maximum yield of ginger oil through experimental works. In this study, two types of samples are being used; (i) sliced and (ii) ground ginger. The ginger oil would be extracted by the hydro-distillation processes. Initially, a study will be carried out to determine which type of sample gives the maximum yield of ginger oil. After deciding on the best sample type, experiments on determining the ideal conditions in the ginger oil extraction would be done. At the same time, light microscopy micrographs of the ginger rhizome at each processing stage showed changes in the oil cell, justifying the operational conditions involved in the extraction process.
2. Study the vapour pressures of ginger oil and mixture of the ginger oil and water at different proportions. In this research, experiments would be done to determine the vapour pressure of the ginger oil and mixture of the ginger oil and water. By doing so, some important parameters on the hydro-distillation theory can be unveiled.

### **1.3 Problem Statement**

The increasing importance of essential oils as pharmaceutical and aromatherapy aid besides their traditional role in cosmetics not only as potent ingredient but also as a fragrance donor has opened up wide opportunities for global marketing. The worldwide market for essential oils has been estimated at US\$2.6 billion, with an annual growth rate of 7.5 percent (Noor Azian, 2001). It is projected that the value of the global market for herbal products would reach US\$200 billion by the year 2008 (The Sun, 2001). In Malaysia, the herbal market was estimated to be worth RM2.5 billion annually, with the local herbal industry capturing only 5 percent to 10 percent of the market. Moreover, the herbal industry is expected to be the main contributor to the country's income in the future (Berita Harian, 2001).

Around US\$ 185 million worth of ginger (excluding ginger oil and oleoresin) were traded world over. India's share in this export is mere 6.3% only. China with 36.5% market share is leading the world market for ginger. However, in ginger oil and oleoresin trade, more than half the quantity is from India. Ginger oil prices vary according to strength and purity. Chinese oil sells at around US\$ 22 to US\$ 30/kg, Indian US\$ 40 to US\$ 50/kg and Sri Lankan at around US\$ 65 to US\$ 70/kg and have rising recently (David Cookson & Co., 2005).

Most of the ginger oil base products are being imported from other countries due to lack of interest and technology in the third world countries and in the developing countries. Malaysia should be more actively involve in the production of essential oil. Spices are produced in the local region but the production of its oleoresins and essential oils is not done in this region. The production is done overseas for example in France and that is why it is very expensive. In order to fulfil the demand of ginger oil in this region and to make it consumer friendly, the production should be done in this region. Only recently, the Malaysian government had shown an interest in the herbal industry, hence the exploitation of these resources through cultivation and commercialisation. Due to lack of technology and knowledge of ginger oil and oleoresin extraction processes in our part, research is now being carried out to fulfil the demand of herbal base products in this region.

In Malaysia, ginger oil extractions are mostly done by hydro-distillation. Local institutions like the Forest Research Institute of Malaysia (FRIM) and Malaysian Agricultural Research and Development Institute (MARDI) play a major role in the essential oil technology transfer. Even though research were carried out at these institution, lack of documentation and research publication on their part, contributed to this study. Most of the research published done on ginger oil locally and worldwide are on the chemistry part and lack in the processing part. This research will fill in the gaps and give detail information on what not to do and do in extracting ginger oil.

*Zingiber officinale Roscoe* (ginger) has been used for a very long time (Lawrence and Reynolds, 1984), in terms of its uses, it is well established. Nevertheless, the processing part has not been well researched. Ginger consists of two main constituents, which are ginger oleoresin and ginger oil. This combination makes ginger an excellent



remedy for digestive problems, such as flatulence, nausea, indigestion, intestinal infections and certain types of food poisoning. The combination of sweat and circulatory stimulation allows ginger to move blood to the periphery. This makes it a good remedy for high blood pressure and fever. Ginger inhibits platelet aggregation, therefore, should be the ideal condiment for people predisposed to clotting which may lead to either heart-attack or stroke (Srivastava *et al.*, 1964). Ginger is also highly effective for motion and morning sickness. Besides having medicinal properties, the ginger oil is used as an ingredient in aromatherapy candles, oils, lotion and in perfume. With ginger's endless versatility and impressive medicinal properties, it truly is the spice of life. This is why ginger becomes our main interest in this research in order to accomplish the objective mentioned earlier.

#### **1.4 Research Contribution**

Although distilling equipment has gradually improved through the years, the method for extracting ginger oil from the plant has changed very little especially in this region. Therefore, based on the knowledge of the best sample type and optimal operating parameters for the production of ginger oil gained through this research will enable for the development and technology transfer to the local producers. It is likely that once the knowledge has been established, it will help the local entrepreneurs boost the herbal industry in this region.