

COMPARISON OF AGARWOOD ESSENTIAL OIL & WATER SOLUBLE  
(HYDROSOL)

FAIRUL IDZWAN BIN MOHD

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I declare that this thesis entitled “*Comparison Of Agarwood Essential Oil & Water Soluble (Hydrosol)*” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : .....

Name of Candidate: FAIRUL IDZWAN BIN MOHD

Date : 28 MAY 2008

Special Dedication of This Grateful Feeling to My...

*Beloved father and mother;  
Mr. Mohd Abd Jamal and Mrs. Roslaini Rodzali*

*Loving brothers and sisters;  
Norhaslina, Fairul Nizam, Fairul Anwar*

*Supportive families;  
Uncles and Aunties*

For Their Love, Support and Best Wishes.

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## ABSTRACT

Gaharu which is also known as *Aquilaria* is the most valuable wood in the world with higher prices and demands nowadays. The widely uses of gaharu in meditation field, essential oil production and etc makes gaharu one of the precious things on earth. For last several years, many methods were founded to extract gaharu especially specifically in distillation. Methods such as steam distillation and spinning band distillation were discovered but the most common methods which more precise and saving cost was hydrodistillation. In this study, we use *Aquilaria Maleccensis* from Malaysia as the sample and it can be classified in grade C. This method requires temperature according to water boiling point which is 100 °C and gaharu sawdust size less than 1.0mm. After extraction, analysis was carrying out by using gas chromatography mass spectrometer to see the component in oil sample. To get the secondary oil, 2 different kind of solvent (ethyl acetate and hexane) were used to trap gaharu essential oil component by mix it with hydrosol which left in the flask. This step is carry out by going through several processes such as centrifuge, filtering and water termination. Then, primary oil and secondary oil is analysis to see compounds which contain in the sample. As the conclusion, we have founded several compounds in secondary oil which same as primary oil and this mean the water can be used to recycle in the next distillation process.

## ABSTRAK

Gaharu atau lebih dikenali sebagai Aquilira adalah antara kayu yang paling bernilai di dunia dengan permintaan dan harga yang tinggi. Penggunaan gaharu secara meluas dalam bidang perubatan, penghasilan minyak wangi dan sebagainya menjadikan gaharu sebagai benda yang paling berharga di dunia. Dalam beberapa tahun yang lepas, pelbagai cara telah ditemui untuk menghasilkan minyak kayu gaharu terutamanya dalam proses penyulingan. Kaedah seperti penyulingan berwap dan penyulingan menggunakan alatan *spinning band* telah ditemui tetapi kaedah yang biasa digunakan di mana lebih persis dan menjimatkan kos adalah kaedah penyulingan air. Dalam kajian ini, *Aquilaria Malaccensis* dari Malaysia digunakan sebagai sample dan diklasifikasikan dalam gred C. Kedua-dua kaedah ini menggunakan suhu takat didih air iaitu 100 °C dan serbuk gaharu yang bersaiz kurang dari 1.00mm. Selepas proses pengekstrakan, minyak tersebut dianalisis menggunakan *gas chromatography mass spectrometer* untuk melihat kandungan minyak tersebut. Untuk mendapatkan minyak kedua, 2 jenis pelarut iaitu heksana dan etil asetat digunakan untuk memerangkap komponen minyak gaharu dengan mencampurkan pelarut tersebut bersama-sama hidrosol yang digunakan dalam kelalang semasa proses penyulingan. Beberapa langkah perlu dilakukan untuk mendapatkan minyak ini contohnya proses pemisahan cecair, penapisan dan penyingkiran air. Kemudian, minyak pertama dan kedua akan dianalisis untuk melihat komponen yang terkandung dalam semua contoh minyak. Sebagai rumusan, beberapa struktur yang dijumpai dalam minyak pertama turut terkandung di dalam minyak kedua dan ini bermaksud air dalam kelalang yang digunakan dalam eksperimen ini boleh digunakan untuk proses penyulingan seterusnya.

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Introduction

Agarwood, also known as gaharu in Indonesia and Malaysia, jin-koh (Japan), Ch'en Hsiang or Ch'en Xiang (China), Chim-Hyuang (Korea) and oud (Middle East), is a resin produced inside a tropical rainforest tree. The dark resinous wood has a magnificent aroma when burned. It can grow on a wide range of soils, including poor sandy soil. Trees grow very fast and start producing flowers and seeds as early as four years old. At least fifteen species of *Aquilaria* trees are known to produce the much sought-after agarwood. In South Asia, *Aquilaria Achalloga* is found in India and *Aquilaria Malaccensis* is mostly known from Malaysia and Indonesia, and *Aquilaria Crassna* in Indochina. For *Aquilaria Malaccensis*, this species is distributed throughout Malaysia, except in Perlis and Kedah (Whitmore 1972) and is known to produce medium-quality grade gaharu (Burkill 1966)

There are several different uses for agarwood as example as antibiotic in medical field to reduce cool and digestion problem, fragrance which generated while burning, Muslims, Buddhists and Hindus use agarwood in religious ceremonies and as a perfume. The wood was also use to decorate boxes, belts, clothing articles, musical instruments, preservatives due to its strong antibiotics function and recently, the range of uses for gaharu has widened to include new products such as gaharu essence, soap and shampoo (Chakrabarty 1994). It also believed to have tonic and therapeutic properties (Burkill 1966, Okugawa 1993)

The value of first-grade agarwood is extremely high. A whole range of qualities and products is on the market varying with geographical location and cultural deposition. Prices range from a few ringgit for the lowest quality to over thirty thousand ringgit for top quality oil.

In Japan, agarwood describes with 5 basic odor classification which take a lead from the 5 basic taste which is sweet (resembling the smell of honey), sour (resembling the smell of plums), hot (resembling the smell of peppers on a fire), salty (resembling the smell of ocean water seaweed is dried on fire) and finally bitter (resembling the smell of bitter herbal medicines when mixed or boiled).

## **1.2 Objective**

To extract gaharu essential oil by using hydrodistillation method and the analysis of the primary and secondary oil.

## **1.3 Scope of study**

In order to achieve the objective, the following scopes have been identified and to be applied:

- I. To study the gaharu oil produce by using hydrodistillation method
- II. To analysis the essential oil or known as primary oil and it's secondary.

## **1.4 Problem statement**

In this project, the agarwood is being use as the substrate. *Aquilaria Malaccensis* or also known as gaharu in Malaysia and Indonesia is commonly extracted for its essential oils using water as the carrier. This method we called

hydrodistillation. In this experiment, we want to see whether this method is proven can produce the essential from gaharu sawdust and to be used as multifunctional oil. From the primary oil, we also have to analyze it to search for the main component which contain in the oil.

The second problem is to analyze whether there were gaharu oil left in the three neck flask that used in this experiment. This oil is usually called secondary oil and the oil which we extracted and collected in receiving tunnel is primary oil. So from this experiment we want to find the solution on how to collect the secondary oil which left in the flask. If there any, we can recycle back the soaking water or more known as hydrosol in next experiment and if not, we can say that hydrodistillation method can extract all gaharu oil in the experiment.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Essential Oil Distillation**

##### **2.1.1 Principles of Distillation**

Thermodynamics play an important role in the distillation of essential oils. In order to vaporize any liquid, energy in the form of heat must be applied. This energy is called latent heat. When a vapor is converted back to liquid, what is actually happening in this process is a reduction and release of that latent heat. In order for a liquid to be vaporized, latent heat must be applied to this liquid until its temperature attains the point where its vapor pressure becomes equal to the surrounding atmospheric pressure. When it reaches this point the temperature will rise no further. However, if heat continues to be applied, the liquid will take up this latent heat and then vaporize at the appropriate rate. At this point the liquid has said to have reached its “boiling point” under the established pressure.

When equally insoluble compounds such as water and essential oil are present, the total pressure exerted by the mixed vapor then becomes the sum of the partial pressures used by each component present. This liquid mixture will boil when its temperature is raised to the point where the combined vapor pressure of its components becomes equal to the surrounding pressure. This being said, it follows that the basic principle of distillation consists of bringing two mutually insoluble liquids (essential oil and water) into contact at a temperature very close to the boiling

point of the more volatile of the two, the water. With the addition of a small amount of vapor pressure from the essential oil the combined pressure is equal to the surrounding pressure. At this point the mixture will boil and the oil will vaporize into the passing steam as fast as its requirement of latent heat can be applied from an external source. When this vapor mixture enters the condenser, it is exposed to a cool surface which again results in a transfer of latent heat, this time causing a reduction in latent heat of the vapor mixture. The result of this reduction is the conversion of the vapor back to a liquid state.

We now have a liquid that is a mixture of the insoluble components found in the still, namely water and essential oil. These liquid components have different densities which allow their separation as a natural state of separation generally occurs. In most instances the essential oil will float to the top, however this is not always the case. Some oils such as Vetiver are more dense than water and thus will settle at the bottom while other oils such as Cinnamon leaf have differing densities, with part of the oil sinking and another floating to the top of the water. Heat from the distillation process can at times change the natural compositions found within the botanic and may even manufacture new components of immeasurable benefit.

## **2.2 Gaharu**

Gaharu, a highly commercial resinous wood used as incense (Ng 1997; Yamada, 1995; Chakrabarty 1994; Sidiyasa, 1986). The genus, which belongs to Thymelaeaceae, consists of 15 species, which occur in tropical forest in Asia. (CIFOR, 1996; Burkill, 1966; Ding Hou, 1960). A matured tree will grow until 40 meter in height with the diameter almost 60 meter. Very often, the quantity and quality of gaharu produced increase with age, with the best yield occurring in trees aged 50 and above (Sadgopal 1960). The 15 species around the world which exist in India, Burma, Myanmar, Indochina, China and Malaysia, have value to commercialize in gaharu production. Gaharu or also known as agarwood is one of

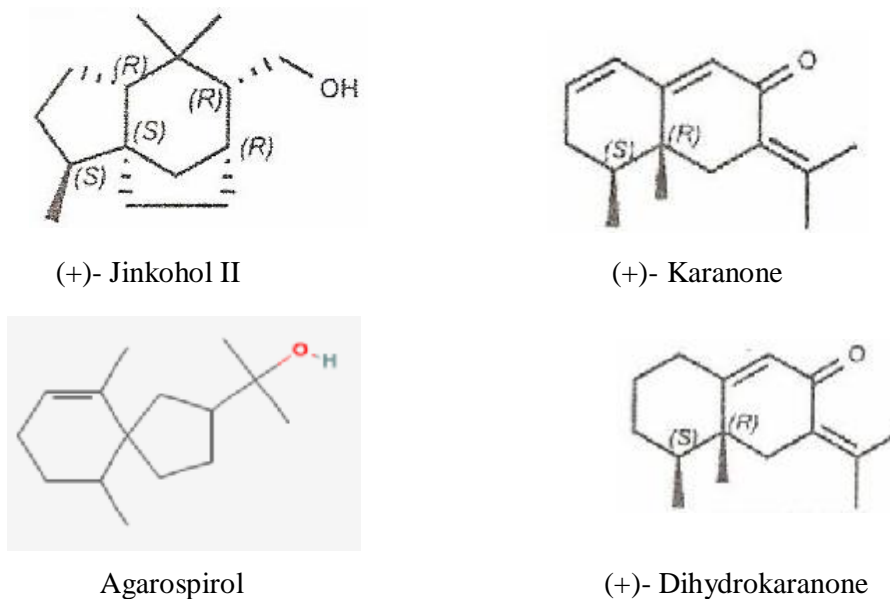


the most expensive woods in the world. The strong scent of gaharu which is unique and complex made it as a main component in production of essential oil. Nowadays gaharu have higher demand in market especially demand from country in Middle East which is use to replace an alcohol in perfume. The economic potential for gaharu products is substantial which consequently, many country are beginning to show increase interest in the commercial use of *Aquilaria* spp. (Qi 1990, Siripatanadilok 1990 cited in Nobuchi & Siripatanadilok 1991, Chakrabarty 1994, Ng 1995) It also uses as fragrant wood that has been traded since biblical times for its use in religious, medicinal and aromatic preparations (Zich, 2001).

In medical field, gaharu become an important substance because it variety of uses. The example of gaharu uses are for stimulant, as tonic, aphrodisiac, diuretic, relieve epilepsy, antimicrobial, carminative (gas), smallpox, rheumatism, illness during and after childbirth, relieves spasms in digestive and respiratory systems, lowers fever, digestive and bronchial complaints, abdominal pain, asthma, cancer, colic, diarrhea, nausea, nerves, regurgitation, weakness in the elderly, shortness of breath, chills, genral pains, cirrhosis of the liver and as a director or focuser for other medicines. It has also been used as a treatment for lung and stomach tumors.

### **2.3 Chemical Component & Structure in Gaharu Essential Oil**

The first investigation on the chemical components of agarwood was reported by Kafuku and Ichikawa (1935 cited in Shimada 1982) . Generally, different chemical component in gaharu oil will determine the characteristic or quality of the gaharu. Figures 2.1 will show some chemical component structure in gaharu essential oil.



**Figure 2.1 : Gaharu chemical components**

Four important naturally occurring aroma constituents of agarwood are agarospirol, (+)-Jinkohol II, (+)-Karanone, and (+)-Dihydrokaranone. They have been evaluated for their odor properties versus their enantiomer. The odor of enantiomeric forms is shown in table 2.1.

**Table 2.1 : Gaharu enantiomer and the odor descriptions.**

Enantiomer	Odor Description
(+)- Jinkohol II	The woody note characteristic of sesquiterpenes in combination with a somewhat camphoraceous odor and when heated or burnt, the characteristic odor of agarwood is strengthened
(-)- Jinkohol II	Very similar to (+)- Jinkohol II
(+)- Karanone	A strong bright woody-amber note
(-)- Karanone	A weak woody note with a citrus atmosphere
(+)- Dihydrokaranone	A remarkable and intense woody note

(-)- Dihydrokaranone	A citrus and mild woody not, it lacks in sharpness and brightness, is deficient in charm compare to its enantiomer but is pleasant in another aroma direction.
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## 2.4 Gaharu in Malaysia

Until now, only 5 species were found in Malaysia. The name of those species are *Aquilaria hirta*, *Aquilaria beccariana*, *Aquilaria rostrata*, *Aquilaria malacinsis* and *Aquilaria microcarpa* which is grow on ground soil until at 750 meter above level sea. For *Aquiliria malaccensis*, this species is one of the endangered species according to reduction of it population. The trade on *Aquiliria malaccensis* need permission permit from exporter country. In annually report, about 630,851 kg have been exported in 1998 and increasing until 887,600 kg in year 2000.

This entire species of aquiliria is very important in gaharu industry. Alternative name for gaharu are agarwood and eaglewood (English), Agor (Bangladesh), akyaw (Myanmar), adlerholz (German), Kanakoh (Vietnam) and calambour (French).

## 2.5 Grading and Prizing of Gaharu.

Grading and pricing of gaharu is a complicated process because to give a price to certain gaharu, we have to know several properties such as size, color, odour, weight, flammability, produce oil and sometimes place where the woodchip came from. This situation made gaharu have no standardizing on it price and grade. In South East Asia, Malaysia was leading the supplier, tie up 26.3% of the market with an import value of RM12.6 million (Anonymous 1994) followed by Singapore with RM11.7 million import value as Indonesia, Thailand and India trailed close behind.

In Malaysia, especially in East Malaysia like Kelantan & Terengganu, gaharu can be divided in several different grade and market price. Table 2.2 & 2.3 showed the grade and market price for gaharu in both states.

**Table 2.2 : Agarwood grade and market prices in Terengganu**

<b>Gaharu Grade</b>	<b>Market Price (per kilogram)</b>
A Grade	RM3,200 to RM4,000
B Grade	RM1,800 to RM2,500
C1 Grade	RM400 to RM800
C2 Grade	RM40 to RM80
D Grade	RM8 to RM30

**Table 2.3 : Agarwood grade with market prices in Kelantan**

<b>Agarwood Grade</b>	<b>Market Price (per kilogram)</b>
Super Double Gred	RM10,000 to RM12,000
Super Gred	RM8,000 to RM10,000
A Gred	RM4,000 to RM8,000
B Gred	RM3,000 to RM4,000
C Gred	RM1,000 to RM2,000
Mixed Gred	RM60 to RM250
Distillate Oil	RM19,000 to RM38,000

## **2.6 Gaharu Formation.**

Gaharu form from karas tree is hard to understand. Commonly, resin yielding because of several reasons which are tree wounding or immune response of wounding tree and fungal attack. The resin of a tree from a natural fungal attack and immune response is commonly known as agar #1. An inferior resin is created by the planned wounding of a karas tree, leaving it more open to a fungal attack by using a

forced method. This resin commonly called agar #2 and found in commercially grown agarwood. The fungus and decomposition process continue to generate a very rich and dark resin forming within the heartwood. That's the reason why agarwood develops very slowly over time-typically several hundred years or more to form. Hence, this is why it is so unusual and valued as the most sacred oil on the planet

According to gaharu principle, there were 2 method in gaharu production which is conventional method (involve sharp object like knife and non-conventional method (involve inoculate).

## **2.7 Protecting and Restoring**

Due to inefficient extraction methods and extensive exploitation of the species by unskilled person, certain species of *Aquilaria* are disappearing at an alarming rate. Furthermore natural population of *Aquilaria Malaccensis* is too low for regular harvest (LaFrankie 1994). This has contribution to the shortage of gaharu and its astronomical prices. To avoid this problem continue, several research institutions in Asia have taken up this challenges and have put serious research initiatives in place (Nobuchi&Siripatanadilok 1991, Chaiwong-Kiet 1994, Chakrabarty 1994, Ng 1995). As a result of this, prospects for discovering methods of including gaharu formation and non-destructive technologies for detecting gaharu in living trees are very promising.

But in Malaysia, a more seriously action was taken. Under National Forestry Act 1984, *Aquilaria* is classification as small forest product. According to section 28, National Forestry Act 1984, gaharu collection from forest need "Lesen Kecil" or small license and Transfer Pass which approved by Ministry of Forestry, gaharu collection need RM10,000 or USD 2632 as deposit, collector only can take 500 kg gaharu woodchip per month per license, 10% royalty is charge for per kilogram of gaharu and finally, collectors who disobey the rule will black listed and their license will cancel out.

Additional conditions for collector are to supply 3,000 young tree of karas tree every year for State Forestry Department, karas tree less than 20 cm in diameter is not allowed to take, responsible to submit shuttle form every month which contain total of gaharu produce.

## **2.8 Essential Oils**

An essential oil is any concentrated, hydrophobic liquid containing volatile aroma compounds from plants. They are also known as volatile or ethereal oils, or simply as the "oil of" the plant material from which they were extracted, such as oil of clove. The term essential indicates that the oil carries distinctive scent (essence) of the plant, not that it is an especially important or fundamental substance. Essential oils do not as groups need to have any specific chemical properties in common, beyond conveying characteristic fragrances. They are not to be confused with essential fatty acids.

## **2.9 Hydrosol**

Hydrosols, also known as floral waters, hydroflorates, flower waters or distillates are products from distilling plant materials. Hydrosols are like essential oils but in far less of a concentration. When a distiller boils plant material with water in a flask and, as it rises, it causes the glands of the plants to burst and release the oils and essence of the plant into the steam. The oil rises through a condenser and collects in a separate vessel. This is known as essential oil and the water that was steamed with the original plant material is our hydrosol, or floral water.

Hydrosols are usually the result of essential oil production as a by-product and contain all of the essence of the plant in every drop, just like essential oils but in a milder form, making them suitable for all manner of applications where essential

oils would be too strong. Sometimes the essential oil which dissolve in the hydrosol is extract back to get the pure essential oil which its price is more higher. This is called secondary oil recovery and can be carry out by using several type of solvents usually hexane

## **2.10 Extraction**

There are many ways to extract essential oils from plants. Essential oils are highly concentrated extracts and should not be confused with which oil that draws only the volatile components from the plant such as infused oils, tinctures and infusions. Essential oils are produced by several methods and each extraction method has strengths and weaknesses and the character and quality of the oil can be different depending upon the method used. Methods of extraction are often unsuitable for home use because of the large amounts of plant material needed to extract a small amount of essential oil.

New methods of essential oil extraction are entering the mainstream of aromatherapy, offering new choices in oils never before available. With the new labels of 'CO<sub>2</sub>' and 'SCO<sub>2</sub>', along with the traditional method, a little education for the aromatherapy lover can go a long way in essential oil selection. 3 main things that make people concern is which process produce better product, have nicer smell and greater value from each other. The value of the newer processing methods depends greatly on the experience of the distiller and the intended application of the final product. Each method is important, and has its place in the making of aromatherapy-grade essential oils.

The roots of distillation methods expand from the creativity of ancient Arabian chemists centuries back with Avicenna (AD 980-1037) complete the process of steam distillation. It was subjected that chemists with Avicenna himself writing over 100 books of science. In that century, he was known because inventing the cooled condenser which used to produce pure essential oils and aromatic water. The

first available and detailed description of distilling essential oils is by the Spanish physician, Anald de Villanova in the thirteenth century and become more advance the French during their perfumery revolution. In 1975 a perfectly distillation apparatus from about 3000 BC was found in Pakistan and along with this, distillation techniques were also developed in China between 300 and 600 AD.

## **2.10 Essential Oil Extraction Method**

There are many methods of essential oil extraction, the most popular being steam distillation. Essential oils are extracted from many different parts of their plants. For example, lavender is extracted from its flowers, orange from the rind of its fruit, frankincense from the resin of its tree, cinnamon from its bark and pine from its needles. Depending on the method of extraction and the quantity of the raw materials used, the price and quality of the oil are determined. There are many other factors to be taken into consideration when producing a good quality essential oil, which is important for its full benefit. Soil quality, climatic and geographic conditions were also contributed to the overall quality of the essential oil.

### **2.10.1 Distillation**

Distillation is the most widely used and the most economical method of extracting essential oils. This is because in the distillation process the precious essential oil is not to be lost or changed in its composition. Some plants are distilled immediately after harvesting, whereas others may be left for a few days or even dried prior to extraction.

In distillation, the plant material is heated, either by placing it in water which is brought to the boil or by passing steam through it. The heat and steam cause the cell structure of the plant material to burst and break down, thus freeing the essential