# EXTRACTION OF GAHARU ESSENTIAL OIL USING SPINNING BAND DISTILLATION

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# DECLARATION

I declare that this thesis entitled "Extraction of Gaharu Essential Oil Using Spinning Band Distillation." is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree."

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|---|---|
| Signature | :………………………………………. |
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| Date | : November 20th , 2006 |

To God, my beloved mother, father, brothers, sister and friends…

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# ABSTRACT

Gaharu is known as one of the most expensive wood in the world. Its essential oil is used in many industries such as perfume and also toiletries. The gaharu that was used in this study is grade C gaharu from peninsular of Malysia (*Aquiliria Malaccensis)* or known as ‘karas’ among the locals. Traditionally, gaharu oil is extracted by distilling the grinded gaharu sample in a copper still. However the process it is not effective and the yield of oil is relatively small and it acquire high temperature. The extraction of gaharu essential oil using spinning band distillation (batch distillation) and water as solvent at heating rate and temperature cut ranging from 20 – 40% and 25 - 100˚C respectively was studied. The size of gaharu particle that will be used is <1.00mm. Result obtained after the experiment is 0% of oil yield in parameter stated. This extraction technique is not suitable to extract the gaharu essential oil at specified parameter. Some changes need to be done to make such objective achievable.

# ABSTRAK

Kayu Gaharu terkenal sebagai antara kayu yang termahal di dunia. Pati minyak yang dihasilkan dari kayu gaharu digunakan dalam banyak industri dan sebagai juga dalam alat dandanan diri. Kayu Gaharu yang digunakan dalam kajian ini adalah daripada gred C dari semenanjung Malaysia (*Aquiliria Malaccensis*) atau lebih dikenali sebagai kayu karas dikalangan penduduk setempat. Secara tradisionalnya, pati minyak gaharu diekstrak dengan kayu gaharu yang telah dikisar itu di suling dengan menggunakan bekas yang diperbuat daripada kuprum. Walau bagaimanapun, cara ini tidak efektif dan pati minyak yang dihasilkan adalah terlalu sedikit dengan memerlukan suhu yang tinggi. Pengekstrakan pati minyak gaharu menggunakan Penyulingan Lingkaran Berpusing (Spinning Band Distillation) dan air sebagai pelarut pada kadar pemanasan dan potongan suhu pada 20 - 40% dan 25 - 100˚C telah dikaji. Saiz kayu gaharu yang dikisar adalah <1.00mm. Keputusan yang diperolehi daripada kajian ini mendapati peratusan pati minyak yang dihasilkan adalah 0% dengan menggunakan parameter yang telah dinyatakan. Ini menunjukkan kaedah pengekstrakan ini tidak sesuai untuk mengekstrak pati minyak gaharu menggunakan dengan parameter tersebut. Beberapa perubahan perlu dilakukan untuk membolehkan pengekstrakan menggunakan alat ini satu kenyataan.

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# CHAPTER 1

# INTRODUCTION

## 1.1 Introduction

Agarwood or eaglewood (Gaharu) is one of the most expensive wood in the world. It is the occasional product of two to four genera in the family *Thymelaeaceae,* with *Aquilaria agallocha* and *Aquilaria malaccensis* the best known species. The strong heavy scent of gaharu is unique and complex. Gaharu is a fragrant wood that has been traded since biblical times for its use in religious, medicinal and aromatic preparations (Zich *et al.*, 2001).

**Table 1.1:** Scientific Classification Of Gaharu/Agarwood

| | |
|---|---|
| Kingdom | Plantae |
| Division | Magnoliophyta |
| Class: | Magnoliopsida |
| Order: | Malvales |
| Family: | Thymelaeacea |
| Genus: | *Aquilaria* |

(Source : www.wikipedia.org)

Gaharu essential oil is highly prized for the scent produced and the oil is used in perfume and toiletry product such as soap, shampoo and etc . Generally, gaharu oils are mixture of sesquiterpenes, sesquiterpene alcohols, oxygenated compounds, chromone derivatives and resin (Chang *et al.*, 2002). Plant extracts as seen as a way of meeting the demanding requirement of the modern industry for the past two decades (Simandi *et al.*, 1996).

There are many method that had been done since 1943 to get the gaharu essential oil. M.A. Rahman in1980 have tried the artificial inoculation and wounding technique to the tree in order for the tree to produce aromatic base oleoresin (M.A. Rahman *et al.,* 1980). In Malaysia, the research on gaharu and also the extracting of gaharu essential oil have been done by Forest Research Institute of Malaysia (FRIM). Base on Chang *et al.,* gaharu essential oil usually obtained by distillation method. For small scale industries that concentrate on extracting the oil, they were using water distillation technique in a copper still. The problems of this technique are low efficiency and acquire high and continuous heating and required long extraction time.

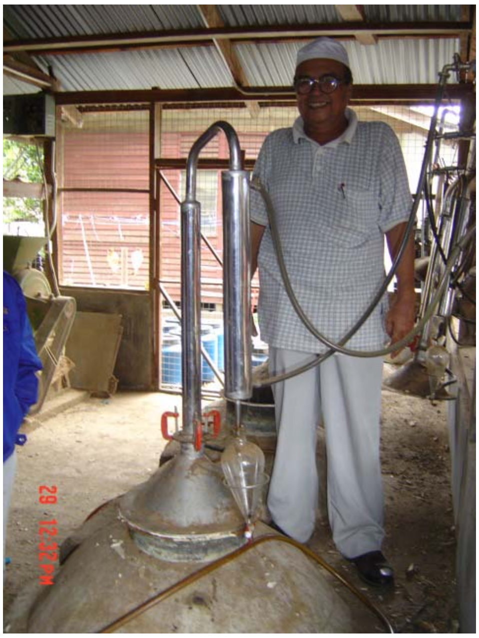


**Figure 1.1:** Traditional water distillation


**Figure 1.2:** Copper Still

## 1.2 Objective

To examine the feasibility of Spinning Band Distillation as an improved method for gaharu oil extraction process.

## 1.3 Scope Of Study

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

1. To study the effect of heating rate on gaharu essential oil extraction ranging from 20% to 40% of heating mantle power.
2. To study the effect of temperature cut on gaharu essential oil extraction.

## 1.4 Problem Statement

Current method of extracting gaharu essential oil is using traditional water distillation method (Chang *et al.,* 2002) or hydrodistillation. This extraction method acquires long extraction times that consume a lot of fuel for heating purposes. The extraction process didn't produce the maximum yield of oil from the wood because the efficiency of the method itself is relatively low. All this will result in higher operating cost especially for heating process.

Another problem is current method also including the extraction using solvent. Even though it take shorter time than the hydrodistillation, the oil produced by this method is not suitable for skin use (Wilson, 1995). Gaharu essential oil also being used in toiletries product. If this oil is to be in the toiletries market, it requires other extraction method.

.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Gaharu

Gaharu or agarwood is the resin-impregnated heartwood of Aquilaria species of the family Thymalaeaceae. It gives off a unique aromatic scent when the wood is burnt. The odor of agarwood is complex and pleasing, with few or no similar natural analogues. As a result, agarwood and its essential oil gained great cultural and religious significance in ancient civilizations around the world. Generally agreed to be the result of a pathological condition, gaharu aromatic resin is produced as the tree sap thickens in response to injury and fungal infection.

The degree to which the resin saturates the heartwood phloem fibers determines the market value of this product. In lesser quality specimens, the resin creates a mottled or speckled appearance in the naturally pale wood, but higher quality specimens are nearly solid in color—glossy and black (Donovan *et al.*, 2004). There are no less than twenty names associated with it, and this reflects its long story and widespread usage. Some other names for gaharu include agaru, aloes wood, eagle wood, oud, chen-xiang (in Chinese it means 'incense that sink'), jinkoh and so on (Chang *et al.*, 2002). Agarwood has been used for traditional medicine in Japan on account of its effectiveness as a sedative or tranquilizer, in detoxifying the body and in maintaining stomach health (Compton *et al.*, 2005).

## 2.2 Grading and Prizing of Gaharu

As noted by Barden *et al.,* grading gaharu is a complicated process. This includes evaluating the size, colour, odour, weight (on scale and in water) and flammability of the wood. The application of grade codes (super A, A, B, C, D and E) varies between buyers.

Resin content of gaharu is often tested by igniting the wood and smelling the smoke, while watching for bubling of resin as the wood burns. When there is a large amount of gaharu to be graded, buyers often make the first sort by using water test, separating pieces that float (because of lower resin content) from those that sink (high resin content) from those that sink (high resin content, better quality). After they are dried again, pieces are graded based on colour and size (Zich *et al.,* 2001).

The grading given an individual is different from others because they totally depend on their senses and sense between human are different. Different people give different grade values. This is always the problem because there is no standard on standardizing the grade of gaharu.

According to Zich *et al.,* 2001, price per kilogram in May 2001 averaged as follows:

| | | |
|---|---|---|
| A grade | = | USD 341 |
| B grade | = | USD 237 |
| C grade | = | USD 172 |
| D grade | = | USD 111 |

**Figure 2.1:** Large pieces of gaharu for sale

(Source : James Compton/T-OC/WWF SPP)

## 2.3 Malaysia Scenario on Gaharu

The majority of gaharu essential oil extraction in Malaysia happens is Kelantan where the villagers doing it as 'backyard' industry. They use water distillation method for that purpose. The process of extracting the oil takes until 96 hours of distillation process.

High quality gaharu can reach up to RM10,000 per kg and is burned like an incense stick. A 12g bottle of oil is sold at between RM50 and RM200. Because of the use of gaharu to get the fragrant oil is huge, high-grade *Aquilaria* resin had grown rare and demand was low for the lower grade chips that collectors were producing. A 21 extraction pot factory will use up to a tonne of wood per month. This shows how intense gaharu usage for one factory alone.

**Figure 2.2:** ‘Backyard’ industry in Kelantan

The federal Forestry Department has since then urged state governments to regulate the collection, trade and processing of agarwood through a licensing system. It also recommended issuing only one harvesting licence for each forestry district (different from administrative district).

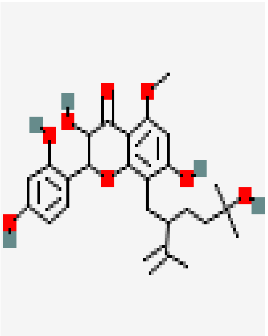
Gaharu collectors or buyers have to pay a royalty fee amounting to 10% of the raw material market price. An extraction permit is issued and this will facilitate the traders in obtaining export and Cites (Convention on International Trade in Endangered Species) permit (Hillary Chiew 2005).

## 2.4 Chemical Component of Gaharu Essential Oil

Generally, gaharu oils are mixture of sesquiterpenes, sesquiterpene alcohols, oxygenated compounds, chromone derivatives and resins. Some of the more important compounds are agarospirol, jinkohol-eremol, jinkohol and kusenol that may contribute to the characteristic aroma of gaharu (Nakanishi *et al*., 1984, Ishihara *et al*., 1993). Other compounds such as 2-(2-4’-methoxyphenylethyl) chromone produce a long lasting fragrance upon burning. Chemical profile for each grade such as grade A, B and C were different. In peninsular of Malaysia, the gaharu were mostly of grade C quality. Gas chromatograms showed similar gas chromatography profile suggesting a region of peaks with retention times ranging from 28.0 to 42.0 min to be indicative of gaharu presence (Chang *et al.*, 2002).

### 2.4.1 Chemical Structure of Gaharu Essential Oil Component

Different chemical component in gaharu oil will determine the characteristic or quality of the gaharu. Figures below will show some chemical component structure in gaharu essential oil.

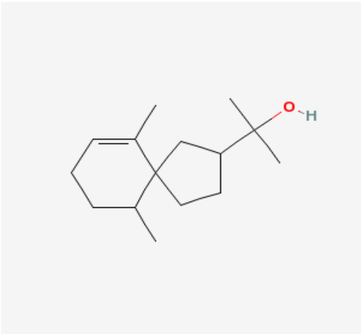


Khusenol

IUPAC: 2-(2,4-dihydroxyphenyl)-3,7-dihydroxy-8-(5--elicoid-5-methyl-2-prop-1-en-2-yl-hexyl)-5-methoxy-chroman-4-one

MW : 472.527 g/mol

**Figure 2.3:** Khusenol

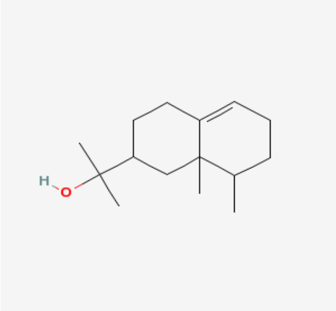



Agarospirol

IUPAC: 2-(6,10-dimethyl-2-spiro[4.5]dec-9-enyl)propan-2-ol

MW : 222.366 g/mol

**Figure 2.4:** Agarospirol

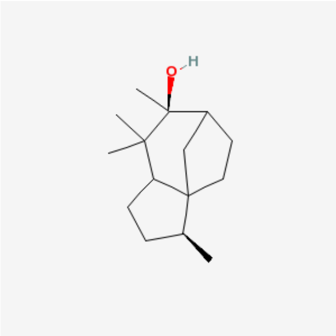



Jinkoheremol

IUPAC: 2-(8,8a-dimethyl-2,3,4,6,7,8-hexahydro-1H-
-elicoids-e-2-yl)propan-2-ol

MW : 222.366 g/mol

**Figure 2.5:** Jinkoheremol




Jinkohol

MF : $C_{15}H_{26}O$

MW : 222.366 g/mol

**Figure 2.6:** Jinkohol

(Source: http://pubchem.ncbi.nlm.nih.gov)

## 2.5 Types of Extraction Method

### 2.5.1 Spinning Band Distillation

One specific type of distillation apparatus which spins a band throughout a major portion of the length of the column is called the ‘spinning band distillation’. Generally, the spinning bands incorporated in these types of distilling columns take on a spirally wound shape, and are just wide enough to lightly scrape the surrounding walls of the column when they are rotated. The spiral shape, giving the band the appearance of elongated helicoids, produces an axial thrust as the band is spun at high rotation per minutes (r.p.m.). Thus, when rotated at high speeds and in the proper direction, these spinning bands force the reflux downward along the walls of the column in a quick and uniform manner. This latter feature is especially helpful in preventing these types of fractionating columns from flooding, even when operated at high boil-up rates (Roark *et al.,* 1997).

The major purpose of this equipment is to purify flavors, fragrances, natural product, and essential oils ranging from 1 to 50 liters capacity.

The key features of this equipment include:

a) High purity:

The 50 theoretical plate fractional distillation column can produce high purity distillates. For flavors, fragrance, natural products and essential oils, even small increase in purity can translate to large increase in value.

b) Low "hold up":

The spinning band distillation column has less than 1.5 milliliters remaining behind in the column after the distillation is complete. This means that the smallest amount of valuable material possible is lost in the purification process.

c) Low pressure drop:

The spinning band distillation column has virtually no pressure drop from the top to the bottom compared to a packed column. This can be a big advantage when distilling delicate samples that decompose easily when heated too high.

d) Automation:

The 9600 fractional distillation system can be fully automated. Once the desired parameters are programmed, the distillation proceeds without any operator intervention, freeing up valuable operator time to perform other important work.

### 2.5.1.1 Spinning Band

There are two main types of spinning bands, Teflon and metal. Teflon spinning bands are the most common an have a maximum of 50 theoretical plates at atmospheric pressure. They are suitable for distilling solvents that can be distilled up to 225 ºC. Teflon is not suitable above this temperature because it becomes soft and can come apart under the spinning force.

In situations where the boiler temperature will go above 225 ºC a metal spinning band can be used. The most common metal used to make spinning bands is Monel. This is a stainless steel with a high content of molybdenum to maximize corrosion resistance.

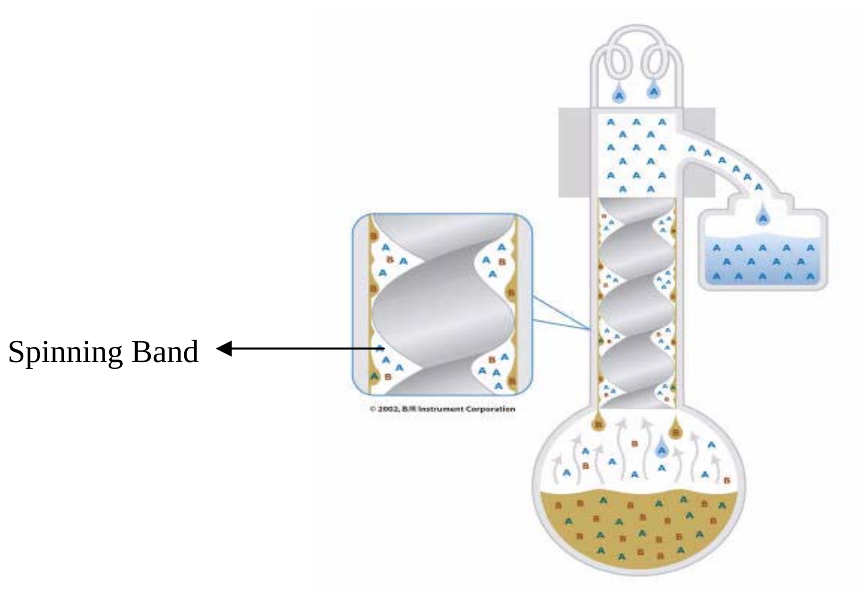



**Figure 2.7:** Spinning Band

(Source: www.brinstrument.com)

### 2.5.2 Supercritical Fluid Extraction

Supercritical Fluid Extraction (SFE) is the application of fluids in their supercritical state for extraction of components from solid materials. This is a relatively new process. This process gives a better quality extract but the capital costs are high. Carbon dioxide is usually used for solvent in this process. Its non-toxic and non-combustible properties make it environmentally friendly.

SFE is used for such application as the decaffeination of coffee, extraction of fragrances for perfumes and extraction of active compounds from natural products for medical purposes. SFE allows for waste separation and minimization, as well as solvent recycling. Other advantages of supercritical extraction include high efficiency, high extraction rates and more selectivity.

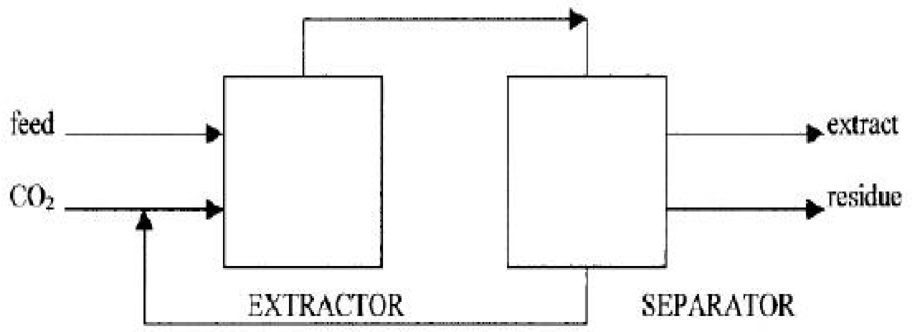



**Figure 2.8:** The Supercritical Fluid Extraction Process

**(**Source: McGaw D.R. *et al.*, 2000)

### 2.5.3 Steam Distillation

Steam distillation is the most common method of extracting essential oils. Steam distillation is done in a still. Fresh or sometimes dried, botanical material is placed in the plant chamber of the still, and pressurized steam is generated in a separate chamber and circulated through the plant material. The heat of the steam forces the tiny intercellular pockets that hold the essential oils to open and release them. The temperature of the steam must be high enough to open the pouches, yet not so high that it destroys the plants or fractures or burns the essential oils.

As they are released, the tiny droplets of essential oil evaporate and, together with the steam molecules, travel through a tube into the still's condensation chamber. As the steam cools, it condenses into water. The essential oil forms a film on the surface of the water. To separate the essential oil from the water, the film is then decanted or skimmed off the top.

The remaining water, a byproduct of distillation, is called floral water, distillate, or hydrosol. It retains many of the therapeutic properties of the plant, making it valuable in skin care for facial mists and toners. In certain situations, floral water may be preferable to pure essential oil, such as when treating a sensitive individual or a child, or when a more diluted treatment is required.

### 2.5.4 Cold Pressing

Another method of extracting essential oils is cold pressed expression, or scarification. It is used to obtain citrus fruit oils such as bergamot, grapefruit, lemon, lime, mandarin, orange, and tangerine oils. In this process, fruit rolls over a trough with sharp projections that penetrate the peel. This pierces the tiny pouches containing the essential oil. Then the whole fruit is pressed to squeeze the juice from the pulp and to release the essential oil from the pouches. The essential oil rises to the surface of the juice and is separated from the juice by centrifugation.

### 2.5.5 Enfleurage

Some flowers, such as jasmine or tuberose, have such low contents of essential oil or are so delicate that heating them would destroy the blossoms before releasing the essential oils. In such cases, an expensive and lengthy process called enfleurage is sometimes used to remove the essential oils. Flower petals are placed on trays of odorless vegetable or animal fat, which will absorb the flowers' essential oils. Every day or every few hours, after the vegetable or fat has absorbed as much of the essential oil as possible, the depleted petals are removed and replaced with fresh ones. This procedure continues until the fat or oil becomes saturated with the essential oil. Adding alcohol to this enfleurage mixture separates the essential oil from the fatty substance. Afterwards, the alcohol evaporates and only the essential oil remains.

### 2.5.6 Solvent Extraction

Another method of extraction used on delicate plants is solvent extraction, which yields a higher amount of essential oil at a lower cost. In this process, a chemical solvent such as hexane is used to saturate the plant material and pull out the aromatic compounds. This renders a substance called a concrete. The concrete can then be dissolved in alcohol to remove the solvent. When the alcohol evaporates, the absolute remains.

Although more cost-efficient than enfleurage, solvent extraction has disadvantages. Residue of the solvent may remain in the absolute and can cause side effects. While absolutes or concretes may be fine for fragrances or perfumes, they are not especially desirable for skin care application.

# CHAPTER 3

# METHODOLOGY

## 3.1 Introduction

In extracting gaharu essential oil, there are few processes that need to be done before extracting process. The extracting process will be run in different range of temperature cut and also different range heating rate to find the optimum condition to extract the oil.

## 3.2 Drying

Drying process need to be done so that the wood is completely dry from any moisture before goes to the next step of experiment. It is also to get rid of any substance that can distract the impurities of oil when it has been extracted (Norazlina 2005). The drying process in completed when the humidity inside the tray drier is longer falling. The air flow speed will be set at 1.44 meter per second (m/s) and the temperature will be set at 60˚C.

## 3.3 Grinding

This is to give the maximum surface area for extraction process and to maximize the contact time between the solvent and gaharu particle. In this experiment, the size of gaharu particle is prepared at 1.00mm. The large trunk of gaharu need to be choped to a smaller size and before it can be grind.

## 3.4 Soaking

Before the extraction process, grinded gaharu must be soaked in water. The ratio of gaharu to water is 1:7 (Dong-ping *et al.,* 1999) for period of three to seven days in order to break down the parenchymatous and oil glands (Chang *et al.,* 2002). For this experiment seven days was chosen in order to maximize the soaking effect. The amount of gaharu sawdust used is 437.5 gram and water equal to 3062.5 mL.

## 3.5 Spinning Band Distillation

The soaked gaharu then is put in the 5 liter heating pot and the temperature cut and heating rate will be set.

The range of temperature cut is around 25-100˚C. Heating rate that will be applied ranging from 20-40% from total 600W of heating mantle power rate.

Before the extraction time is recorded, the system will be leave to equilibrium state that is around 15 minutes so that the extraction of the essential oil will be stable. The heating rate at each cut is the same with early heating rate so that the heating process through the experiment is stable. Reflux ratio of each cut is set to zero so that it can maximize the end product that is the essential oil. The end pot temperature is set not to exceed more than 15°C because almost all of the equipment is made from glass to prevent from cracking. Every experiment with the different range of data will be run twice to duplicate the data.

Below are the summarize data to be set for the experiment:

**Table 3.1:** Summarize data set of experiment

| | |
|---|---|
| Heating rate | X % |
| Equilibrium | 15 minutes |
| Open cut 1 | $T_a$°C |
| Close cut | $T_b$°C |
| Heating rate 1 | X % |
| Reflux ratio | 1:0 |
| Cut 2 close temp. | $T_c$°C |
| Heating rate 2 | X % |
| Reflux ratio | 1:0 |
| Cut 3 close temp. | $T_d$°C |
| Heating rate 3 | X % |
| Reflux ratio | 1:0 |
| Cut 4 close temp | $T_e$°C |
| Heating rate | X % |
| Reflux ratio | 1:0 |
| Pot temp. end run | not more than 15°C from $T_e$°C |

**Table 3.2:** Planning of extraction

| **Experiment 1** | **Experiment 2** | **Experiment 3** | **Experiment 4** |
|---|---|---|---|
| Heating rate 20% | Heating rate 20% | Heating rate 30% | Heating rate 40% |
| Cut 1 (25.0°C-35.0°C) | Cut 1 (60.0°C-70.0°C) | Cut 1 (60.0°C-70.0°C) | Cut 1 (60.0°C-70.0°C) |
| Cut 2 (35.1°C-45.0°C) | Cut 2 (70.1°C-80.0°C) | Cut 2 (70.1°C-80.0°C) | Cut 2 (70.1°C-80.0°C) |
| Cut 3 (45.1°C-55.0°C) | Cut 3 (80.1°C-90.0°C) | Cut 3 (80.1°C-90.0°C) | Cut 3 (80.1°C-90.0°C) |
| Cut 4 (55.1°C -65.0°C) | Cut 4 (90.1°C -100.0°C) | Cut 4 (90.1°C -100.0°C) | Cut 4 (90.1°C -100.0°C) |

## 3.6 Yield

Yield oil extracted will be determined by:

$$\text{Yield} = \frac{\text{weight of product produce (gram)}}{\text{weight of sample fed (gram)}} \quad (1)$$

Drying process to ease the grinding process

↓

The 437.5 gram of grinded gaharu soaked with water (ratio 1:7) for seven days

↓

Soaked gaharu will be put in heating pot
Set the basic parameter.
R% heating rate will be set.
Temperature cut ($T_a$°C-$T_d$°C) will be set.

↓

The process will be leave for equilibrium for 15 minutes

↓

Extracted product from each cut will be collected in collection vessel.

**Figure 3.1:** Flow diagram of the extraction process

# CHAPTER 4

# RESULT & DISCUSSION

## 4.1 Introduction

The experiment of extracting gaharu essential oil using spinning band distillation technique with the Spinning Band Distillation System Model 9600 from B/R Instrument had been completed. The procedure of doing this experiment is carefully followed to ensure the optimum data obtained from each experiment run.

## 4.2 Preliminary Experiment

Before the experiment is started, preliminary experiment had been done to measure the equipment capability in doing this experiment. There are three preliminary experiments that had been done. From three, two was run without using vacuum condition while the other one with vacuum applied. Below are the parameter set and also the result from the studies:

**Preliminary experiment 1**

**Table 4.1:** Parameter for preliminary experiment 1

| | |
|---|---|
| Heating rate | 30 % |
| Equilibrium | 60 minutes |
| Open cut 1 | 40°C |
| Close cut | 100°C |
| Heating rate 1 | 15 % |
| Reflux ratio | 1:2 |
| Pot temp. end run | 110°C |

It is observed that the heating goes without any problem but when it reach the temperature cut range, the heating became slow and unable to boil the solution. The heating rate is more to maintain the heating pot temperature rather than heating the solution to increase the temperature. From the experiment, the heating rate need to be maintain at specific rate so that the heating can be proportional to time and desired temperature cut can be achieved.

**Preliminary experiment 2**

**Table 4.2:** Parameter for preliminary experiment 2

| | |
|---|---|
| Heating rate | 30 % |
| Equilibrium | 30 minutes |
| Open cut 1 | 25°C |
| Close cut | 100°C |
| Heating rate 1 | 30 % |
| Reflux ratio | 1:0 |
| Pot temp. end run | 110°C |

As expected, the heating process was stable and the solution manages to boil. It is observed that without reflux, the condensed vapor flow faster than previous experiment. From this, it is decided that for the experiment, it is better to used without reflux so that the end product will be maximize.

**Preliminary experiment 3**

**Table 4.3:** Parameter for preliminary experiment 3

| | |
|---|---|
| Heating rate | 30 % |
| Equilibrium | 15 minutes |
| Open cut 1 | 70°C |
| Close cut | 80°C |
| Heating rate 1 | 30 % |
| Reflux ratio | 1:0 |
| Cut 2 close temp. | 90°C |
| Heating rate 2 | 30 % |
| Reflux ratio | 1:0 |
| Cut 3 close temp. | 100°C |
| Heating rate 3 | 30 % |
| Reflux ratio | 1:0 |
| Pressure | 50% |
| Pot temp end run | 110°C |

In this experiment, vacuum condition is applied to lower the boiling point of the solution. But problem occurs in the middle of the experiment whereby the gaharu solid is being suck into the distillation column and into the spinning band rotation. The experiment is immediately terminated due to safety reason and to prevent the equipment failure that can affect the flow of the planned experiment.