EXTRACTION OF GAHARU ESSENTIAL OIL USING MICROWAVE ASSISTED EXTRACTION (MAE)

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DECLARATION

I declare that this thesis entitled “Extraction of Gaharu Essential Oil Using Microwave Assisted Extraction (MAE)” 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 : MOHD ROSLI BIN RAMLY

Date : 20 November 2006

Dedicated to my beloved father, mother, and family……..

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ABSTRACT

Gaharu or it scientific named *Aquilaria malaccensis* is one of the most precious trees in the world. It is one of the most expensive natural products existing today. Gaharu is also known as Agarwood, eaglewood, aloeswood, oud, chen-xiang, and jingkoh. Gaharu grade C from peninsular Malaysia was used in this study. Conventional extraction method such as ultrasonic extraction and Soxhlet extraction is not effective, lower yield and the reaction time to produce gaharu essential oil take longer extraction time. Microwave assisted extraction method was identified as one of the extracting methods account to the economical, high yield and ease of the operation. By doing microwave assisted extraction, the time and temperature is observed by analyzing the data. The pressure performed at atmosphere (1atm), and temperature range is from 60°C to 100°C. For overall result, the yield percentage obtained is 0%. This is because the parameters that used in this experiment are not suitable for gaharu using microwave assisted extraction method. Low yield of the gaharu essential oils can be improved in future study by carrying out the research in larger scale.

ABSTRAK

Gaharu atau nama sainstifiknya Aquilaria merupakan salah satu tumbuhan yang berharga di dunia. Pada masa kini gaharu merupakan hasil yang sangat berharga dalam bidang pengektrakan minyak.Gaharu juga dikenali sebagai Agarwood, eaglewood, aloeswood, oud, chen-xiang, dan jingkoh. Gaharu gred C dari semenanjung Malaysia akan digunakan dalam kajian ini.Teknik yang digunakan sebelum ini untuk pengekstrakan seperti ultrasonic dan Soxhlet adalah tidak sesuai. Ini kerana, hasil yang didapati adalah kurang dan masa yang panjang diperlukan untuk menghasilkan minyak pati gaharu. Kaedah pengektrakan mikrogelombang telah dikenalpasti sebagai satu kaedah yang menjimatkan, hasil yang tinggi, dan mudah untuk digunakan. Dalam kaedah ini, parameter yang terlibat adalah tekanan dan suhu. Tekanan yang digunakan adalah pada tekanan atmosfera (1 atm) dan suhu yang terlibat adalah diantara 60°C-100°C bagi mengenal pasti hubungan antara suhu dan masa untuk menghasilkan pati minyak gaharu. Untuk keseluruhan experimen, tiada peratusan penghasilan diperolehi (0%). Hal ini disebabkan, parameter yang ditetapkan adalah tidak sesuai dalam pengektrakan gaharu menggunakan kaedah mikrogelombang. Hasil minyak gaharu yang rendah ini dapat dipertingkatkan pada kajian akan datang menggunakan skala yang lebih besar.

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LIST OF ABBREVIATIONS

CITES	Convention on International Trade in Endangered Species
CO2	Carbon Dioxide
GC	Gas Chromatograph
MAE	Microwave Assisted Extraction
NTFP's	National Traffic Forest Papua New Guinea's
PGK	Papua New Guinea's Kina
PNG	Papua New Guinea's
SEM	Scanning Electron Microscopy
SME	Solvent Microwave Extraction

CHAPTER1

INTRODUCTION

1.1 Gaharu

Gaharu is one of the most precious trees in the world. It is one of the most expensive natural products existing today. Gaharu or it scientific named *Aquilaria malaccensis*. Gaharu is also known as Agarwood, eaglewood, aloeswood, oud, chen-xiang, and jingkoh - these are just a few of the names for the resinous, fragrant and highly valuable heartwood produced by *Aquilaria malaccensis* and other species of the Indomalesian tree genus Aquilaria [9]. Gaharu oil is used in perfume and toiletry products such as soap and shampoo.The grade of gaharu is divided by 5 types, which are Grade Super A, A, B, C, and D [3]. In this study, gaharu grade C used to produce essential oil and obtained from its stem by using microwave assisted extraction (MAE).

1.2 Microwave Assisted Extraction (MAE)

Microwave assisted extraction (MAE) is based on the direct application of electromagnetic radiation to a material (e.g. organic solvent, plant tissue) which has the ability to absorb electromagnetic energy (microwaves) and to transform it into heat. Unlike conventional heating by infrared energy or thermal conductivity, the increase in temperature occurs simultaneously in the whole volume of solvent. This process is

caused by the multiple collisions of the solvent molecules as they realign in the oscillating electromagnetic field, generating energy in the form of heat [8].

Compared with conventional methods, such as ultrasonic extraction and Soxhlet extraction, the advantages of MAE are reported to be a higher recovery of the analyte, shorter extraction times and the use of smaller quantities of solvent [2][13]. MAE can be performed in open or closed vessels [2]. In open systems, the extraction occurs at atmospheric pressure and with variable energy input. In closed systems, extraction takes place at controlled pressure (up to 5 atm) and a temperature that may exceed the boiling point of the solvent under atmospheric conditions, to increase extraction efficiency.

1.3 Problem Statement

Local companies still has not known the technology to produce the gaharu essential oil at an optimum output in terms of purity, production rate, energy consumption and process minimization. Previous extraction method such as ultrasonic extraction and Soxhlet extraction is not effective, the reaction time to produce gaharu essential oil take longer extraction time. Other than that, extraction using solvent will cause toxicity and reduce the quality of the product.

1.4 Objective

The objective of the study is to examine the feasibility of Microwave Assisted Extraction (MAE) as an improved method for gaharu oil extraction process.

1.5 Scope

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

i. Study the effect of temperature on gaharu essential oil extraction.

ii. Study the effect of microwave on micrograph structure of gaharu cell.

CHAPTER 2

LITERATURE REVIEW

2.1 Gaharu

2.1.1 Gaharu

Gaharu is one of the most precious trees in the world. It is one of the most expensive natural products existing today. Gaharu or it scientific named *Aquilaria malaccensis*. [9]. *Aquilaria malaccensis* is one of 15 tree species in the Indomalesian genus *Aquilaria*, family *Thymelaeaceae*. It is a large evergreen tree growing up to 40 m tall and 1.5-2.5 m in diameter, found typically in mixed forest habitat at altitudes between 0 and 1000 m above sea level [5].

The species has a wide distribution, being found in Bangladesh, Bhutan, India, Indonesia, Malaysia, Myanmar, the Philippines, Singapore and Thailand. *A. malaccensis* and other species in the genus Aquilaria sometimes produce resin-impregnated heartwood that is fragrant and highly valuable. There are many names for this resinous wood, including agar, agarwood, aloeswood, eaglewood, gaharu and kalamabak [3]. This wood is in high demand for medicine, incense and perfume across Asia and the Middle East. [6]

Figure 2.1 Gaharu [24]

2.1.2 Harvesting of Gaharu

Harvesting required the removed of the resin wood, gaharu, from the rest of the tree. Only a very small percentage (3-8%) of naturally occurring trees, produce the resin gaharu. Within those that do produce gaharu, production is often associated with tree injury such as broken branches or damaged roots which provide for openings into the tree stem or roots. Most pockets of resin wood comprise between 50-100g per tree [6].

The first step in harvesting is identifying trees containing gaharu. Villagers were taught to look for external signs of gaharu formation:

i. Ants around a wound in the trunk leaking resin;
ii. Insect damage or holes on the trunk;
iii. Broken branches in the canopy;
iv. Broken or damaged roots.

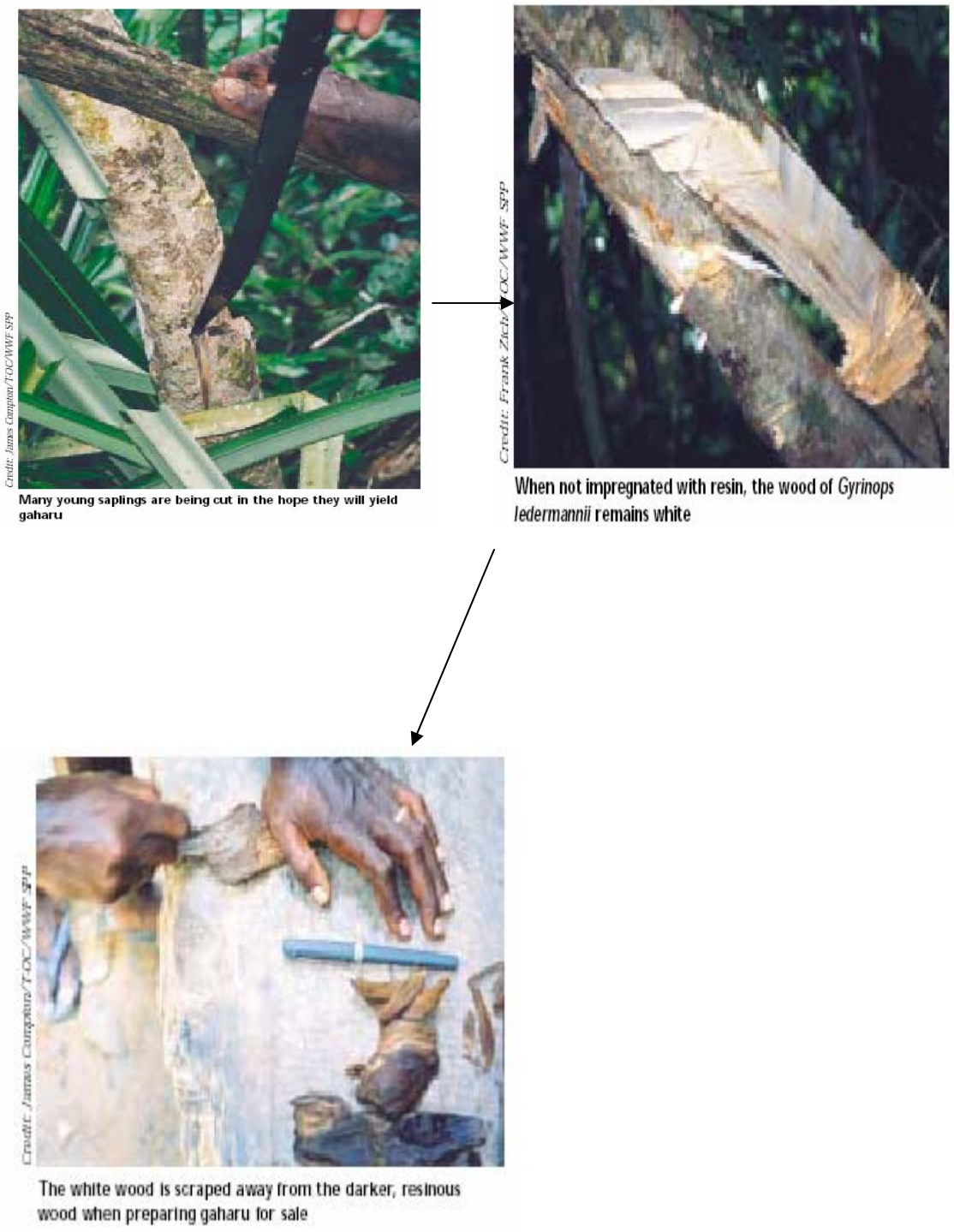


Figure 2.2 Harvesting of Gaharu [6]

2.1.3 Gaharu Trade

Grading gaharu is a complicated process [3]. This includes evaluating the size, colour, odour, weight (on scales and in water) and flammability of the wood but application of grade codes (Super A, A, B, C, D, E) varies between buyers in PNG. Chips are usually one to a few centimeters in length; larger pieces can be approximately 10 or more centimetres in length.

Resin content of gaharu is often tested by igniting the wood and smelling the smoke, while watching for bubbling 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 the water test, separating pieces that float (because of lower resin content) from those that sink (high resin content, better quality). After they are dried again, pieces are graded based on colour and size. Mixed chips of good quality (black, black and brown) are graded as C and often fetch a higher price per gram than large pieces of A and B grade.

According to Information collected from individual involved in the trade, prices in May 2001 and May 2002 averaged as follows:

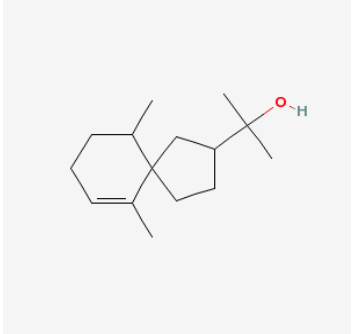
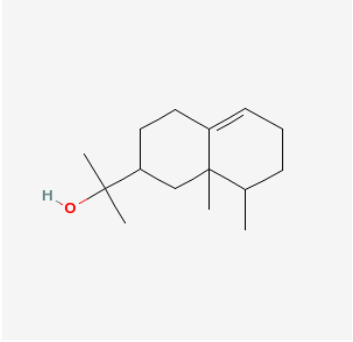
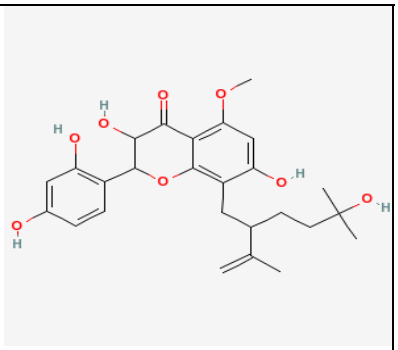
Table 2.1 Gaharu Grade [6]

Average (reported) prices paid by buyers to collector		Prices recommended by the PNG Forest Authority	
May 2001		May 2002	Super A-PGK 2000/kg
	Grade A-PGK 1140/kg		Grade A-PGK 1500/kg
	B grade- PGK 791/kg		B Grade -PGK 1000/kg
	C grade-PGK 575/kg		C Grade -PGK 500/kg
	D grade-PGK 372/kg		D Grade -PGK 50/kg

2.1.4 Structure and Chemical compound of gaharu

The best quality of gaharu essential oil is depend on the important chemical components and the number of components that contribute the characteristic of aroma gaharu, in all samples for every process of extraction[12]. In this study, there are three major components in gaharu - agarospirol, jinkoh-eremol and khusenol.

Table 2.2 Structure of agarospirol, Jinkoh-eremol and Khusenol [23]

		
• IUPAC name- 2-(6,10-dimethyl-2-spiro[4.5]dec-9-enyl)propan-2-ol.	• IUPAC name- 2-(8,8a-dimethyl-2,3,4,6,7,8-hexahydro-1H-naphthalen-2-yl)propan-2-ol.	• IUPAC name- 2-(2,4-dihydroxyphenyl)-3,7-dihydroxy-8-(5-hydroxy-5-methyl-2-prop-1-en-2-yl-hexyl)-5-methoxy-chroman-4-one.
• Formula molecule- C15H26O.	• Formula molecule- C15H26O	• Formula molecule C6H32O8
• Molecular weight- 222.366 g/mol.	• Molecular weight- 222.366 g/mol	• Molecular weight- 472.527 g/mol

2.2 Essential Oil

Essential oils are the volatile, aromatic oils obtained by steam or hydrodistillation of botanicals. Most essential oils are primarily composed of terpenes and their oxygenated derivatives. Different parts of the plants can be used to obtain essential oils, including the flowers, leaves, seeds, roots, stems, bark, wood, etc. Certain cold-pressed oils, such as the oils from various citrus peels, are also considered to be essential oils but these are not to be confused with cold-pressed fixed or carrier oils such as olive, grapeseed, coconut etc. which are non-volatile oils composed mainly of fatty acid triglycerides. Other aromatic, plant-derived oils, which technically aren't essential oils because they are solvent extracted, include Absolutes (hexane followed by ethanol extraction), CO2's (liquid carbon dioxide used as the solvent) and Phytols or Florosols (fluoro-hydrocarbon solvent) [22]

2.2.1 Gaharu Essential Oil

Agarwood oil is made from the wood of Agar. There are different grades of Agarwood oil. Agarwood comes in solid or liquid form. Solids are only solid at room temperature, and if warmed slightly, it turns to mobile liquid. It is an anti-asthmatic and can be applied directly to the skin as it is non-irritating. The oil is very tenacious and only the tiniest of drops is needed to fill the air with its soul evoking aroma. It is a complex aroma with many nuances, deep and ethereal. The aroma takes about 12 hours to unfold and it will last on the skin for more than a day, and if placed on any material, the scent can last for months. It can be used as a perfume, an aroma therapy and an essential oil or as an aid for the deepest meditation. This fragrance will unlock the subconscious and allow you to go deep into your memories.

2.2.2 Benefits of Essential Oil

Essential oils is the primary ingredients in aromatherapy which are safe and simple, natural products. They can be used just for pleasure, or to help individual heal physical and emotional ailments. It can be a complete which is holistic and natural form of therapy, taking into account the effect of the treatment on the body, the mind and the emotions of the person receiving it. The effectiveness of essential oils usage has been proven by scientific analysis, confirming the intuitive link, understood by our ancestors, between nature and general well-being. The dynamics of aromatherapy enable us to bring the essence of nature into our everyday lives.

2.2.3 Physical Properties of Essential Oil

The first and foremost physical properties of essential oil are the highly fragrant, concentrated, and potent substances. Essential oil has a liquid-soluble molecular structure which allows them to pass easily through the skin. They penetrate into the fat layers of the skin quickly, which is why massage is such an effective treatment. Other than that, it is volatile, so that it is easily evaporate into air. The storage of essential oils is usually in a dark bottle because of it is sensitive to light. Essential oils are not standard product and will vary from batch to batch. Chemical variations will occur based on the time of day, harvest time, growing location and part of the plant to be extracted. They are cytophylactic which regenerate new cells and enhance the function of our organs [24]

2.2.4 Chemical Properties of Essential Oil

Essential oils may have two major components which are terpene hydrocarbon, and oxygenated compounds [22] terpene hydrocarbon can be divided into two group; monoterpenes and sesquiterpenes. While oxygenated compounds are phenols, monoterpenes, and sesquiterpenes alcohols, aldehydes, ketons, esters, lactones, coumarins, ethers, and oxides.

Monoterpenes compounds are found in nearly all essential oil and have a structure of 10 carbons atoms and at least one double bond. The 10 carbons atoms are derived from two atoms isoprene units. Monoterpenes react readily to air and heat sources. These components have anti-inflammatory, antiseptic, antiviral, and antibacterial therapeutic properties. Sesquiterpenes consist of 15 carbons atoms and have complex pharmacological actions. It has anti-inflammatory and anti-allergy properties. Based on [1], there are three main aromatic groups which are phenols, terpenes alcohols, and aromatic aldehydes.

2.3 Extraction

Extraction is a separation process to separate the desired solute or removed an undesirable solute component from the solid where the solid is contacted with a liquid phase. The two phases are in intimate contact and the solute can diffuse from the solid to the liquid phases, which cause a separation of the components originally in the solid

In this process, there will be the advantages and disadvantages. One of the advantages is extraction can be performed at ambient temperature. Thus, it is relatively energy efficient and can be applied to separations involving thermally unstable molecules.