EXTRACTION OF PATCHOULI OIL USING HYDRO DISTILLATION

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I declare that this thesis entitled "*Extraction of Patchouli Oil using Hydro Distillation*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is concurrently submitted in candidature of any degree.

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Special dedication to my beloved father, mother, brother and sisters

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ABSTRACT

There are a lot of uses of patchouli. For example, it is used in pharmaceutical industry that is effective for fungal and bacterial infection and is of great help for insect bites. In cosmetic industry, with its excellent diuretic properties, it is effective in fighting water retention and to break up cellulite, easing constipation and helping to reduce weight. This research is conducted to study the effect of amount of patchouli leaves (raw material) on the extraction process and to find the optimum extraction time in the extraction of patchouli oil. In the first experiment, five different amounts of patchouli leaves, varying from 250gram to 450gram will be applied to identify the effect of amount of raw material in the extraction process. In the second experiment, different extraction times which are 3hour, 3.5hour, 4hour, 4.5 hour and 5 hour are used to find the optimum extraction time for extraction of patchouli. When the amount of patchouli leaves used is increase, the percentage of oil yield is also increase. 450gram patchouli leaves produced the highest yield which is 1.61%. When the extraction time is increased, the percentage of oil yield is also increased. Extraction time at 5hour is the optimum extraction time because it produced the highest oil yield that is 2.467%. For both experiment, the temperature is set up at 100° C and the pressure is at 1 atm.

ABSTRAK

Pada masa ini, terdapat banyak kegunaan minyak patchouli. Sebagai contoh, di dalam industri farmaseutikal, minyak patchouli berkesan dalam jangkitan kulat dan bakteria dan sangat membantu dalam gigitan serangga. Di dalam industri kosmetik, sifat diuretiknya menyebabkan minyak patchouli sangat penting kerana dapat memecahkan selulit, mencegah sembelit dan menolong mengurangkan berat badan. Eksperimen ini dijalankan untuk mengkaji kesan jumlah daun patchouli atau bahan mentah yang digunakan dalam proses ekstraksi minyak patchouli dan mencari masa ekstraksi yang terbaik yang akan menghasilkan peratusan minyak patchouli tertinggi di dalam proses ekstraksi minyak patchouli. Untuk eksperimen yang pertama, lima sampel disediakan dari 250gram hingga ke 450gram daun patchouli di mana eksperimen ini dijalankan untuk mengkaji kesan jumlah bahan mentah di dalam proses ekstraksi. Untuk eksperimen yang kedua, masa ekstraksi bermula pada 3jam, 3.5jam, 4jam, 4.5jam dan 5jam di mana eksperimen ini dijalankan untuk mencari masa ekstraksi yang terbaik. Setelah eksperimen dijalankan, didapati semakin banyak daun patchouli yang digunakan, semakin tinggi peratusan minyak yang dihasilkan. 450gram daun patchouli menghasilkn peratus minyak paling tinggi iaitu pada 1.61%. Begitu juga apabila semakin lama masa ekstraksi, semakin tinggi nilai peratusan minyak patchouli yang dihasilkan. Masa ekstraksi pada 5jam menghasilkan peratusan minyak tertinggi iaitu pada 2.467%. Untuk kedua-dua eksperimen, suhu ditetapkan pada 100⁰C dan pada 1atm.

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LIST OF SYMBOLS

SPE	Solid phase extraction
GC	Gas chromatography
GC/MS	Gas chromatograph mass spectrometer
Atm	Atmosphere
Q	Heat transfer rate
А	Area
Т	Temperature
T _s	Temperature of adjacent moving fluid stream
T_{f}	Temperature of fluid
Q	Convective heat flux
Н	Convective heat transfer coefficient
T_{w}	Wall temperature
T _{sat}	Saturation temperature of the liquid
\leq	Less than or same
q_{min}	Minimum heat flux
ΔT	Temperature difference
Х	Distance
Κ	Thermal conductivity
Ср	Heat capacity

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CHAPTER 1

INTRODUCTION

1.1 Background

Patchouli (also known as patchouly or pachouli) is both a plant and an essential oil (patchouli oil) obtained from the leaves of a plant of the same name. It is grown in the East and West Indies but primarily grown in Indonesia. The word derives from the Tamil *patchai* (green), *ellai* (leaf). Patchouli was first described by botanists in the Philippines in 1845. Today growing interest in its fragrance has led to patchouli's widespread cultivation throughout tropical Asia. Its scientific name is *Pogostemon cablin.* The taxonomic position of patchouli is given below (George Bentham, 1826):

Kingdom:	Plantae
Division:	Magnoliophyta
Class:	Magnoliopsida
Order:	Lamiales
Family:	Lamiaceae
Genus:	Pogostemon
Species:	P. cablin

Table 1.1: Taxonomic position of patchouli

True patchouli has hairy stems, flowers only reluctantly, and is usually propagated by cuttings. A second species, *Pogostemon heyneanus*, known as Java patchouli, has smooth stems and flowers. Java patchouli has an inferior aroma and may show up as an adulterant in the whole leaf form or the distilled oil. Plants available from herb nurseries labeled as *Pogostemon cablin* often turn out to be *Pogostemon heyneanus*.

The patchouli plant is a bushy herb reaching two or three feet in height. The plant grows well in southern climates. It enjoys hot weather but not direct sunlight. If the plant withers due to lack of watering it will recover well and quickly once it has been watered. The seed-bearing flowers are very fragrant and bloom in late fall. The tiny seeds may be harvested for planting, but they are very delicate and easily crushed. Cuttings from the mother plant can also be rooted in water to produce further plants.

Patchouli is most often used in aromatherapy. The dried leaves and stems are employed in traditional Chinese medicine to normalize the flow and balance of the life force known as qi (or chi). An aromatherapy blend inspired by the traditional Chinese use can evoke a feeling of gentle clarity and inspire the harmonious flow of emotional energy. It uses oils with balancing and mildly energizing aromas.

It is a component in about a third of modern, high-end perfumes, including more than half of perfumes for men. Patchouli is also an important ingredient in East Asian incense. It is also used as a scent in products like paper towels, laundry detergents, and air fresheners. The essential oil is obtained by steam distillation of the dried leaves of the plant, a process which provides a relatively high yield of the oil. An important component of the essential oil is patchoulol or patchouli alcohol that gives the smell to the patchouli oil (Albert, 1980) and it is also the largest component among the components in the patchouli oil (Trifilieff, 1980).

Hydro distillation is the most common method of essential oil production, where the plant material is boiled, with the resultant steam being captured and condensed. The oil and water are then separated; the water, referred to as a 'hydrosol', can be retained as it will have some of the plant essence.

A number of factors determine the final quality of a hydro distilled essential oil. Aside from the plant material itself, most important are time, temperature and pressure, and the quality of the distillation equipment. Essential oils are very complex products; each is made up of many, sometimes hundreds, of distinct molecules which come together to form the oil's aroma and therapeutic properties. Some of these molecules are fairly delicate structures which can be altered or destroyed by adverse environmental conditions. So, much like a fine meal is more flavorful when made with patience, most oils benefit from a long, slow 'cooking' process.

The temperature of the extraction chamber cannot be too high, lest some components of the oil be altered or destroyed. The same is true of the chamber's pressure. Higher temperatures and/or pressures result in a 'harsh' aroma, more chemical than floral and lessen the oil's therapeutic effects. Also, the extraction period must be allowed to continue for a certain period of time in order to flush all the oil's components from the plant, as some are released more quickly than others.

Despite the drawbacks of aggressive processing, high temperatures and pressures are often used to produces large quantities of oil in a short period of time. These oils are usually destined for use in cosmetic and processed food manufacturing, but are sometimes sold to final consumers as essential oils for use in aromatherapy. These oils will be less expensive, but are of limited therapeutic value, and the difference is apparent when the aromas are compared side by side.

1.2 Problem Statement

Extraction of patchouli oil is still new in chemical industries. There are no method that has been proven to be the most efficient to extract the oil from patchouli leaves. Hydro distillation is the simplest and cheapest method of extracting essential oils. Nowadays, the price of patchouli oil is increasing due to the increasing demand of patchouli oil from many industries such as pharmaceutical, aromatherapy and also cosmetic industry. So, in order, to lower the cost, and increase the profit, using hydro distillation process to extract the patchouli oil from the leaves is the best solution.

1.3 Objectives of the Research

The objectives for this research are to study the effect of quantity of raw material in the extraction process and to find an optimum extraction time for extraction of patchouli oil.

1.4 Scopes of the Research

This research consists of two components which are:

- i. The higher yield of patchouli essential oil is based on the higher quantity of patchouli leaves.
- ii. The effect of the different production or extraction time on the percentage of oil yield.

CHAPTER 2

LITERATURE REVIEW

2.1 Essential Oil

According to R. Deininger, (1995), essential oil is highly concentrated and potent oils extracted from plants, leaves, flowers, roots, buds, twigs, rhizomes, heartwoods, bark, resin, seeds, and fruits. Essential oil is found in special secretor glands or cells within plant life. The aromatic substances are formed and stored in certain organs of a plant as a by-product or because of its metabolism. Each essential oil has its very own blueprint that is absolutely unique. The combination of the plants blueprints, the energy of the sun, soil, air and water gives each of the oil its individual perfume and beneficial healing properties. The same species of plant can produce an essential oil with different properties depending on whether it was grown in dry or damp earth, at high or low altitude, or even in hot or cold climates. According to the International Organization for Standardization, essential oil is "a product made by distillation with either water or steam or by mechanical processing of citrus rinds or by dry distillation of natural materials. Following the distillation, the essential oil is physically separated from the water phase".

Besides, essential oil has many uses and maybe obtained from either wild or cultivated plants. An estimated 3000 essential oils are known of which approximately 300 are of commercial importance. The majority is obtained from agricultural plants but a number of oils are collected from wild sources including trees (Iqbal, 1993). One commercial source of essential and fragrance oils list over 50 different oils; 25 of which are used in cooking and over 20 are used in potpourri,

crafting, cosmetics, massage, aromatherapy and other uses. Still other essential oils are used to repel insects and other arthropods that are pests of humans, livestock and pets (mosquitoes, fleas, ticks) (Thomas and Schumann, 1992).

2.2 Patchouli

2.2.1 Introduction of Patchouli

Patchouli (the vernacular name over the greater part of the Madras Presiden, Sir G. Birdwood in Athenum 22 Oct. 1898), the elements of which are referred by some to Tamil pach, pachai(green) and ilai (leaf). Patchouli, Patch, Putch and Putcha-Leaf, are trade names of the dried leaves of a labiate plant allied to mint (Pogostemon patchouly, Pelletier). It is supposed to be a cultivated variety of *Pogostemon heyneanus*, Bentham, a native of the Deccan. It is grown in native gardens throughout India, Ceylon, and the Malay Islands, and the dried flowering spikes and leaves of the plant, which are used, are sold in every bazaar in Hindustan. The pacha-pat is used as an ingredient in tobacco for smoking, as hair-scent by women, and especially for stuffing mattresses and laying among clothes as we use lavender. In a fluid form, patchouli was introduced into England in 1844, and soon became very fashionable as a perfume (Holmes, Peter. 2001).

Weiss, 2004 said that the plant *Pogostemon cablin* is an "aromatic, herbaceous, perennial shrub, with erect stems, large green leaves and small whitepink flowers. Leaves are opposite, broadly ovate, epically acuminate, basally truncate to cuneate, with coarsely serrate to dentate margins; slightly fleshy, softly tormentors mainly on the underside, and dropping when mature. Mature leaves are 5-10 X 2.5-8cm, light to medium green in color, varying considerably with location or cultivar. The essential oil is contained mainly in glands on underside of leaves. Glandular hairs are small with a short single celled stalk and double-celled head". The fragrance of *Pogostemon cablin* or "Patchouli" became popular in the United States during the counter culture era, the 1960's, and for many people (who lived through that period) the smell has strong associations for better or worse. Unfortunately the aroma that was associated with that time was often a product of synthetic formulations or poor distillations and has sometimes given the actual aroma of a well-distilled oil a poor name. The fragrance may deserve "re-exploration" by those who have negative associations of the oil for one reason or another. Properly matured leaves that have been distilled in stainless steel vessels in particular display very soft, subtle, precious woods-herbaceous notes that few associate with this plant. Those oils are also very light in color as compared to the darker colored oils that come from crude iron distilling vessels.

The main source for patchouli oil is the leaves which have been properly prepared for distillation. The roots of mature plants as well as their stems also possess some essential oil but it is of an inferior order. According to Titin Handayani, (2006), the oil yield from the leaves is 1.8% to 2% and from the stems, the oil yield is only from 0.7% to 1%.

2.2.2 Environmental Considerations for Patchouli Plant

Patchouli is a plant which enjoys high relative humidity and temperature and an even rainfall distribution. It thrives best where the temperatures remain in the 25-30 centigrade range and the yearly rainfall amounts to 80-120 inches (even distribution). If natural rainfall falls below 60 inches then irrigation becomes necessary to produce a commercial crop. As important as sufficient water is a fertile, well drained soil which means that the crops grows best on undulating land. It can grow in flatter regions as well but no water logging should occur as this proves fatal to young plants. The plants are very demanding in terms of proper fertilization and the traditional pattern has been to establish the crop on newly cleared lands and after three years rotate a less demanding crop into its place. But for long term care, the soil has to be nourished with natural fertilizers in the form of compost and green manure crops. The application of these age old and simple technologies will accelerate as people on the consuming end become more interested in supporting the higher costs of organic production.

Plants are generally propagated from cuttings although in India and other countries tissue culture propagation is proving a good means of producing the plants in large quantities. These cuttings, selected from the mid-stem sections of semimature plants are then placed in shaded nurseries while they sprout new rootlets. As this is a crop which is generally handled by small land holders, the transplanting into field conditions is done by hand. Individual holes are dug for each rooted cutting and planted three nodes below the surface. They can be planted in open field conditions or as in intercrop among young or even mature plantations of coconut, rubber, citrus or coffee.

They often thrive under these situations where more intense cultivation practices are followed. It takes approximately 900 lbs of rooted cuttings to plant 1 acre of land devoted to patchouli. Planting is usually done so that within 6 months the plants will form a canopy to suppress weed growth. Weeding is generally done by hand during this time and after harvest as the tending of these crops is done by the farmers and their families on small plots of land.

The transplanting of the rooted cuttings usually occurs at the beginning of the rainy season as the cloud cover moderates the intense sun rays and helps prevent the tender young leaves from getting scorched. It also is the time when their is sufficient natural moisture for the plants to establish a strong root system so that they can survive periods of time later in their life cycle when water may not be as abundant (during the non-rainy season).

2.2.3 Types of Patchouli Plant

Although there are 3 types of patchouli plant with similar characteristics, only one is extensively cultivated for its oil. The other two are grown in backyards and used as natural perfume and remedies.



Figure 2.1: Pogostemon cablin benth

Pogostemon cablin benth is the most common type of patchouli and is also known as Pogostemon patchouli or Pogostemon mentha. Locally it is known as Aceh Patchouli.

It is believed to have originated from the Philliphines and later spreads to Indonesia, Malaysia, Madagaskar, Paraguay, and Brazil. The top leaf is shinny green and the bottom part of the leaf appears dull and hairy, containing most of the oil in the plant. This type seldom flowers and has high oil yield, from 2.5 to 3.5%.



Figure 2.2: Pogostemon heyneanus

Pogostemon heyneanus also known as Java Patchouli or "forest patchouli". Originally came from India, this plant grows wild in the Java forest. The leaves are thin, dark green, and more pointed. It flowers and has oil content 1.5 to 1.5%, relatively low for commercial use.



Figure 2.3: Pogostemon hortensis

Pogostemon hortensis also known as "soap patchouli", traditionally used in laundry. The primary area of production is West Java. The leaf is shiny, smooth and thin, similar to *Pogostemon heyneanus*; however it does not flower. The oil content is only 0.5 to 1.5 %, making it unattractive commercially.

2.2.4 Harvesting of Patchouli

The first crop is usually ready for harvest in the 4-6 month period when the plants are approximately 3 feet high. The foliage at that time is pale green to light brown. Subsequent harvests occur every 3-6 months during the productive life of the plant. One thing which could potentially improve the quality and quantity of oil is periodic foliar feed with kelp based products. In order to produce oil of the highest quality only the three to five uppermost pairs of mature leaves should be harvested as this is where the highest concentration of oil is found in its purest form (Nurhayatin Nufus, 2004). This practice is only possible among small landholders and is seldom followed.

From the level of the plant this method is ideal as it allows more rapid regrowth of the plant as there is a greater volume of remaining herbage to promote photosynthesis. It also is practical as the small local stills are often not equipped to handle large amounts of material as may happen when more vigorous harvesting techniques are followed. Generally speaking farmers tend to cut the plants 4-8 inches above the ground which means that included with leaves is a good deal of stem material. Ideally this should be separated out at the distillery but in fact is often included in the distillation process. Harvesting should not occur after rain or in the early morning when the leaves are wet with dew. The amount of material yielded by one acre is 2-4 tons in the first year under good conditions. A smallholder harvesting only the top sets of leaves obtains considerably less (although of higher quality). The yield is from 400-1100 kg of material.

The percentage of moisture in the fresh patchouli cuttings is 80-85% with an oil yield of 0.5-1.2%. It means that 150 kilos of air dried material is realized from 1000 kilos (1 metric ton) of fresh material and from these 1-2 kilos of oil is obtained (Nurhayatin Nufus, 2004). This is based upon Indonesian growing and harvests techniques. In Malaysia the yield is a bit higher with 200 kg of air dried leaves being produced from 1000 kg of fresh materials resulting in 2-3 kilos of oil. Indian grown patchouli has shown to be the richest in oil with 200 kilos of air dried leaves producing 3-4 kilos of oil. After the first harvest there is a significant decline in production of fresh leaves. It can fall from 20-50% of the initial crop.

2.2.5 Uses of Patchouli Oil

Patchouli oil has grounding and balancing effect on the emotions and banishes lethargy, while sharpening the wits fighting depression and anxiety. V., Amsterdam, 1988 said, "to create an amorous atmosphere".

It is effective for fungal and bacterial infection and is of great help for insect bites. It could also be used as an insect repellant and is also used as a support for dealing with any substance addiction. With its excellent diuretic properties, it is effective in fighting water retention and to break up cellulite, easing constipation and helping to reduce overweight. Furthermore, it has a great deodorizing action, and helps when feeling hot and bothered, while cooling down inflammations and assisting with wound healing.

On the skin, this oil is one of the most active and is a superb tissue regenerator, which helps to stimulate the growth of new skin cells. In wound