EXTRACTION OF PATCHOULI OIL USING STEAM DISTILLATION

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EXTRACTION OF PATCHOULI OIL USING STEAM DISTILLATION

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A thesis submitted in fulfillment of the
requirement for the award of the degree of
Bachelor Degree of Chemical Engineering

Faculty of Chemical and Natural Resources Engineering
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MAY 2008

DECLARATION

I declare that this thesis entitled "*Extraction of Patchouli Oil Using Steam 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 not concurrently submitted in candidature of any other degree.

Signature	:	………………………………..
Name	:	Dewi Haryani binti Sulaiman
Date	:	14 May 2008

To my beloved parents, siblings, and fiancé

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ABSTRACT

This study is about Extraction of Patchouli Oil Using Steam Distillation. The objective of this research is to study the feasibility of the optimum condition of steam distillation in patchouli oil extraction process. Patchouli oil extraction is still new but has gained large market demand for the benefit on therapeutic and healing properties of this essential oil. However, cost-effective route have yet to be develop. This research has identified two scope of study to achieve the objective which is to vary the effect of different extraction time and sample mass on the yields. In this extraction, part of the plant used is the leaves and stick. Firstly, the raw material is exposed 3 hours under direct sunlight and 3 days in room temperature. Dried patchouli plants are then cut to 2 cm in size. Then, the leaves are stacked in the extraction vessel. High pressured steam passed through the plant material from the bottom of the vessel. Hot steam will force open the pocket in which the essential oil of the patchouli was kept. Next, the steam which contains the essential oil passed through cooling system to condense the steam which would separate the essential oil from water. Pure oil is extracted with this method. For this equipment with the range of 7 hours extraction time and 2 kg to 4 kg sample masses, the optimum extraction time is at 7 hours with 3 kg sample mass.

ABSTRAK

Projek ini bertajuk Pengekstrakan Minyak Nilam Menggunakan Penyulingan Berwap. Objektif projek ini ialah untuk mengetahui keadaan yang paling optimum untuk penyulingan berwap dalam melakukan proses pengekstrakan ini. Pengektrakan minyak nilam adalah masih baru di pasaran tetapi permintaan terhadap kebaikan minyak ini dari segi perubatan terapi telah mendapat sambutan meluas. Namun begitu, proses yang efektif dari segi kos masih lagi dalam kajian. Dalam projek ini, dua parameter telah ditetapkan untuk mencapai objektif projek ini iaitu kesan perubahan masa pengekstrakan dan berat nilam terhadap hasil penyulingan. Dalam proses ini bahagian yang digunakan ialah daun dan batang pokok nilam. Pertamanya, nilam perlu dikeringkan di bawah sinaran matahari selama 3 jam dan suhu bilik selama 3 hari. Nilam yang sudah dikeringkan itu di potong dalam anggaran saiz 2 cm. Kemudiannya, nilam itu akan dimasukkan ke dalam ‘vessel’ penyulingan dan wap air bertekanan tinggi akan di alirkan dari bawah ‘vessel’. Wap air yang panas ini akan menyebabkan minyak yang berada di dalam poket tumbuhan akan terbuka. Campuran wap air dan minyak nilam akan terus mengalir ke sistem penyejukan dan terkondensasi di mana air dan minyak nilam ini akan terpisah. Minyak yang asli kebayakannya mengunakan cara ini bagi proses pengeksrakan. Bagi alat ini untuk julat masa 7 jam masa pengekstrakan dan bagi berat jisim sampel di antara 2 kg hingga 4 kg, keadaan optimumnya ialah pada 7 jam masa pengekstrakan dan pada 3 kg berat sampel.

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LIST OF SYMBOLS

USD $	-	United State Dollar
kg	-	Kilograms
RT	-	Retention Time
%	-	Percentage
>	-	more than
±	-	Estimation (plus minus)
^{o}C	-	Degree celcius
T	-	Temperature
cm	-	Centimeter
atm	-	Atmospheric pressure

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Patchouli is an essential oil obtained from dried leaves or of a plant of the same name. Its botanical name is *Pogostemon Cablin*. Patchouli oil is widely used in perfumes as one of the important natural essential oils used to give a base, lasting character and fixative ability to a fragrance. It is originated from East and West Indies and their name derives from the Tamil *patchai* (green), *ellai* (leaf). Patchouli is also known as patchouly, *tamala pattra* in Sanskrit and *guang huo xiang* in Chinese. Indonesia is the major producer of patchouli oil in the world with and estimated 550 tons per year, which is more than 80% of the total (Robbins, 1983; Tao, 1983). The taxonomic position of patchouli is given below in Table 1.1 (Wikipedia, 2007. *Patchouli*).

Table 1.1: Taxonomic position of patchouli

TAXONOMIC POSITION	
Kingdom	Plantae
Division	Magnoliophyta
Class	Magnoliopsida
Order	Lamiales
Family	Lamiaceae (Labiatae)
Genus	Pogostemon
Species	P.cablin

Before it became popular in Europe, the unique patchouli odor was being impregnate in the Indian shawls and Indian ink. Despite being used as alternative lifestyle in modern industry in fine products like cosmetic product as well as 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 in paper towels, laundry detergents, and air fresheners as a scent in products.

Patchouli is a very fragrant, bushy herb with soft oval leaves and square stems. It grows from two to three feet in height and provides an unusual odor that is nonetheless characteristic of patchouli when the leaves are rubbed. 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 a tropical member of the mint family. Leaves are harvested several times a year, dried, and exported for distillation of the oil, although the highest quality oil is usually produce from fresh leaves, distilled close to the plantation (Wikipedia, 2007. *Patchouli*).

An important component in a patchouli oils is patchouli alcohol ($C_{15}H_{26}O$) or patchoulol known as terpene. Generally, patchouli oil consists of over 24 different sesquiterpen es. One of the organic compounds responsible for the typical patchouli scent is the optical isomer. All the chemical compositions in the patchouli essential oil are analyzed by using the Gas Chromatography Mass Spectrometry technology.

1.2 Problem Statement

At present, the essential oil industry is not only focusing to the production and distribution of essential oils alone, but has focus more on the improvement of methods and maintenance of standard quality. This is because of the large market demand for the benefit in therapeutic and healing properties of the essential oils. Patchouli oil is an important ingredient in many fine fragrance products but the patchouli oil extraction is still new compared to other essential oil extraction. However, cost-effective route to produce the oil has yet to be developed. Furthermore, the price of patchouli oil increasing by years (Lerner and Ivan, 2003).This study focuses specifically on extraction of patchouli oil, as it is widely appreciated for its pleasant characteristic and long lasting odor, and to find the best method for extraction. The extraction method used in this study is steam distillation as patchouli is commonly extracted using this method.

1.3 Objective of the Study

The objective of this research is to study the feasibility of the optimum condition of steam distillation in patchouli oil extraction process.

1.4 Scope of Study

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

1. Effect of different extraction time on oil yield
2. Effect of different sample mass on oil yield.

For the first parameter, extraction time, the sample mass is fixed for 6 hours duration where the oil yield will be record for every one hour starting from 3 hours. The same experiment will be run for three times to get the average reading for each extraction time. For the second parameter the yield will be recorded at different sample mass ranging from 2 kg to 4 kg and the yield will be record at certain period. The experiment also will be run for 7 hours.

CHAPTER 2

LITERATURE REVIEW

2.1 Essential Oil

Early in history, the term “essential oil” or ”ethereal oil” defined as the volatile oil obtained by the steam distillation of plants. Essential oils which are also referred to as “essences”, not only originated from flowers, but from herbs, shrubs, trees and various other plant materials.

Gradually with the advance knowledge in science arise improvement in the methods of preparing the oils, parallel with the development of a better knowledge understanding of the constituent of the oils. It was found that the oil contains chiefly liquid and more or less volatile compound of many classes of organic substances usually dependent upon the oxygenated compounds. Four main groups, which are characteristic of the majority of the essential oils, i.e.:

1. Terpenes, related to isoprene or isopentene;
2. Straight-chain compounds, not containing any side branches;
3. Benzene derivatives;
4. Miscellaneous

(Haagen-Smit, 1949)

Large volumes of oils are usually distilled from leafy material such as lemongrass, citronella and cinnamon leaves. Meanwhile, the small volumes of oils are usually distilled from fruits, seed, buds and flowers (e.g cloves, nutmeg and coriander). The percentages of characteristic differ at different part of the plant.

2.1.1 Benefit of Essential Oil

The plant derives specific benefit from its own essential oils. The two major advantages they gain from their essential oil are as protection and reserve food. The irritating effect of many oils affords a degree of protection against the depredations of animals and plant parasites. In individual cases a contribution is made toward more effective pollination through insect visits. The action of some essential oils is similar in certain respect to that of anaesthetics on animal cells. The inhibiting and damaging effect of the oils on many life processes has been turned to our advantage in the use of these compounds as bactericidal and fungicidal agents. In other cases of protection, plants which emanate a considerable amount of oils are prevented from becoming too warm since heat is absorbed in the vaporization of the oils. In this way the oil function as a water-sparing mechanism.

As for human being, the essential oils were regularly used in ancient Rome, Greece, and Egypt and throughout the Middle and Far East. Their common feature, the essence of a plant; an identifiable aroma, flavour, or other characteristic was of some practical use. They were used as perfumes, food flavours, deodorants, pharmaceuticals, and embalming antiseptics. For example, the essential oil is the primary ingredient in aromatherapy treatments which are safe and simple natural product.

2.1.2 Physical Properties

Most plants contain essential oils but only the aromatic plants produce essential oil in sufficient quantities. They can be more or less fluid of which some are viscous; others are fairly solid and most are watery. They are sometimes resinous and often have a coloring, which ranges from yellow to emerald green and blue to dark brownish red. Essential oils are diffuse and penetrating. Their high degree of vibratory capacity gives them this quality. Therefore, they should be kept in well stopper with a sound cork. Other than that, as essential oils are very sensitive to heat and light, they should be stored in a cool place and dark bottles. In order to minimize oxygen exposure, a small amount of oil in a large bottle should be transferred to a small bottle instead. Another important physiochemical criterion of the quality and purity of an essential oil is the specific gravity which the values vary between the limits of 0.696 and 1.188 at 15°C which in general, the gravity is less than 1.000 (Gildemeister *et al.*,1956). Essential oil at 15°C/15°C of specific gravity defined as the ratio of the weight of a given volume of oil at 15°C to the weight of an equal volume water at 15°C. Essential oil boil generally between 150^{o}C-300^{o}C (consist of many compound) however the compound are steam volatile and can be distillate at around at 100^{o}C.

2.1.3 Chemical Properties

Essential oil chemical properties are usually analyze with a chromatography and the primary components are terpene hydrocarbons (monoterpene hydrocarbons and sesquiterpenes), oxygenated compounds consists of phenols and alcohols (monoterpene alcohols and sesquiterpene alcohols), aldehydes, ketones, esters, lactones, ethers and oxides.

Most essential oils consist of mixtures of hydrocarbons (terpenes, sesquiterpenes, etc.), oxygenated compounds (alcohols, esters, ethers, aldehydes, ketones, lactones, phenols, phenol ethers, etc.), and a small percentage of viscid or solid nonvolatile residuen (paraffin, waxes, etc.). Of these the oxygenated compound are the principal odor carriers, although the terpenes and sesquiterpenes, too, contribute in some degree to the total odor and flavor value of the oil. The oxygenated substances posses the added advantage of better solubility in dilute alcohol and, with the exception of some aldehydes, of greater stability against oxidizing and resinifying influences (Guenther, 1949).

Essential oils that are rich in monoterpenoid constituent were those obtained from the leaves and fruit peels of *C. hystrix*, the fruit peel of *C. aurantifolia,* and the leaves of *O. citriodorum*. Limonene, α-pinene, β-pinene, linalool, geraniol, citral, terpinen-4-ol and alpha-terpineol were the major representatives of monoterpenoids present. Essential oils that are rich with sesquiterpene constituent and phenyl propanoids were *O. tenuiflorum* and *P.cablin* (Nor Azah *et al*., 2007).

The first primary component found is the terpenes hydrocarbons. One of it is the monoterpene compounds that are found in nearly all essential oils and have a structure of 10 carbon atoms and at least one double bond. The other one is the sesquiterpenes consists of 15 carbon atoms and has complex pharmacological actions.

Another primary component is the oxygenated compound consists of phenols and alcohols. Phenols found normally have a carbon side chain. Due to the nature of phenols, essential oils that are high in them should be used in low concentrations and for short periods of time, since they can lead to toxicity if used over long periods of time, as the liver will be required to work harder to excrete them. It can also cause skin and mucus membrane irritants and although they have great antiseptic qualities, like cinnamon and clove oil, they can cause severe skin reactions. Alcohols found in the essential oil like monoterpene alcohol on the other hand have good antiseptic, anti-viral and anti-fungal properties with very few side effects such as skin irritation or toxicity and have an uplifting energizing effect. As for sesquiterpene alcohols, they are not commonly found in essential oils but when found they have great properties, which include liver and glandular stimulant, anti-allergen and anti-inflammatory. For the rest of the other components, aldehydes, ketones, esters, ethers, and oxides are found in a small quantity.

2.1.4 Types of Essential Oil

Today, there is a lot of essential oil from different kind of aromatic plant. Table 2.1 show some of the plants plant used in the extraction of essential oils.

Table 2.1: Types of essential oil

Plant	Part of Plant Used	Botanical Name	Country of Origin	Important Constituent	Properties
Chamomile	Flower	*Matricaria recutita*	England, Germany, France, Morocco	Bisabolol	Sedating, nurturing, soothing, calming, reassuring
Cinnamon	Leaf	*Cinnamomum zeylanicum*	Sri Lanka, India	Eugenol	Condiment and flavouring material, anti-oxidant, antimicrobial
Lavender	Flower	*Lavendula intermedia*	England, France, Yugoslavia, Bulgaria	Linalol	Anti-depressant, appeasing, balancing, , purifying, relaxing, sedative, soothing
Lemongrass	Leaf	*Cymbopogon spp*	Tanzania	Citral, Citronella, Terpenes	Analgesic, anti-depressant, antimicrobial. antipyretic, antiseptic, astringent, bactericidal
Sandalwood	Wood	*Santalum Album*	Nepal, Sri Lanka, Hawaii	Santalol	Antimicrobial, antiseptics
Clove	Bud	*Eugenia Caryophyllus*	Indonesia	Eugenol	Carminative, anthelmintic, anodyne
Turpentine	Resin	*Pinus spp*	Mediterranean country	Terpenes	Solvent for paints, antiseptic, Diuretic

2.1.5 Effect of Extraction Process to Essential Oil

A number of factors determine the final quality of a steam 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. Lavender essential oil, for example, should not be processed at over 245^{o}F and pressure at 3 psi. Higher temperatures 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. (The Ananda Apothecary, 2007. *Making Essential Oil-Steam Distillation CO_2 and Absolutes*)

2.2 Patchouli

2.2.1 Introduction

Patchouli or scientifically known as *Pogestoman cablin (P.cabin)* is a member of the mint family. Patchouli is perennial, bushy plant that grows up to three feet high, with a sturdy, hairy stem and large, fragrant, furry leaves, about four inches long and five inches across as shown in Figure 2.1 (Aroma-pure,2007.*Patchouli*). It has whitish flowers tinged with purple as shown in Figure 2.2 (Anniesremedy,2007.*Herb*). It grows in tropical climates. The plant originated from India and Indonesia. However become popular in the west beginning around 1844 when the first dried leaves, Figure 2.3 (Scents-of-earth,2007.*Patchouli*), arrived in London. Before that, it was a well-known fragrance in Indian textiles throughout Europe. It is used as an insect repellant and perfume. It is a base note in several famous perfume ingredients for both men and women. It was grown in China almost two thousand year ago and was used as a perfume for ink. Today, it is commonly used in cigarettes to compensate for a lack of taste due to reduced tar content. Good quality patchouli will retain its sweeter notes on a perfume blotter for months. The leaves must be fermented, Figure 2.4 (Alchemy-works,2007.*Herb*), during the process before they could produce the full blast of their scent.

Figure 2.1: Bushy of patchouli leaves

Figure 2.2: Patchouli flower

Figure 2.3: Patchouli leaves (cut)

Figure 2.4: Fermented patchouli leave

2.2.2 Uses of Patchouli Oil

There are many uses of patchouli oil recorded. One of the major uses is in the aromatherapy treatment. The oil is used as a topical remedy for skin problems such as acne, eczema, inflamed, cracked, chapped and irritated skin. It is known as a cell rejuvenator and helpful in healing wounds and scars. As an antifungal, patchouli oil has been used to treat athlete's foot. For the hair, patchouli oil has been used for dandruff and to aid oily hair. For the nervous system, patchouli essential oil helps to reduce tension, insomnia and anxiety. It is also known as uplifting fragrance that helps to soothe away everyday cares, and to bring about a sense of nourishment. In this way, and due to its wine-like intoxicating aroma, patchouli oil is also known an aphrodisiac.

In the perfume industry, Patchouli acts as a base note and fixative par excellence. It is used in many famous perfumes such as Tabu and Shocking. A little patchouli oil, used as a fixative can be used in many natural perfume formulations. Patchouli oil mixes well with many essential oils including vetiver, sandalwood, frankincense, bergamot, cedarwood, myrrh, jasmine, rose and the citrus oils. The traditional use of the patchouli is as incense in temples. It is said to assist in grounding and centering the mind prior to meditation. It is also produces a strong connection to the earth as it is an aid to connection with the natural beauty of the planet.

2.2.3 Physical Properties of Patchouli Oil

Patchouli oil has a rich musky-sweet, strong spicy and herbaceous smell with a nearly hidden fruity note. It is light yellow to dark golden brown in color and is a viscous liquid. The boiling point of patchouli oil is 287.00 °C at 1 atm.

2.2.4 Chemical Components of Patchouli Extractives

The composition of the patchouli oil is complex like many essential oil, but distinct because of the large of sesquiterpenes. The chemical compound reported found in the patchouli plant is in Table 2.2 (Oller,2007.*Patchouly*).

Table 2.2: Chemical compound found in patchouli plant

Chemical Compound	Part of the Plant
1,10-epoxy-alphabulnesene	Plant
1-alpha,5-alpha-epoxy-alpha-guaiene	Plant
1-beta,5-beta-epoxy-alpha-guaiene	Plant
2-methyl-butyric-acid	Leaf
2-methylhexanoic-acid	Leaf
4-methyl-pentanoic-acid	Leaf
alpha-bulnesene	Plant
alpha- bulnesene-oxide	Plant
alpha-bulnesone	Plant
alpha-guaiene	Plant
alpha-guaiene-oxide	Plant
alpha-patchoulene	Plant
alpha-pinene	Plant
Apigenin	Plant
apigenin-7-o-beta-d-(6”-p-coumaroyl)-glucoside	Plant
apigenin-7-o-beta-glucoside	Plant
Azulene	Plant
Benzaldehyde	Plant
beta-elemene	Plant
beta-patchoulene	Plant
beta-pinene	Plant
Bulnesol	Plant
Cadinene	Plant
Camphene	Not-stated
Cinnamaldehyde	Plant
cis-2-pentylcyclopropylcarboxylic-acid	Leaf
Cycloseychellene	Plant
dehydracetic-acid	Leaf
Dimethylphenol	Leaf
Epiguaipyridine	Plant
Epoxycaryophyllene	Plant
Eugenol	Plant
Gamma-patchoulene	Plant
guaiacol	Leaf
Guaipyridine	Plant
heptanoic-acid	Leaf
Humulene	Plant
Limonene	Plant
nonanoic-acid	Leaf
Nordehydropatchoulol	Plant
Norpatchoulenol	Plant
o-cresol	Leaf

octanoic-acid	Leaf
Ombuine	Plant
p-vinyl-phenol	Leaf
Pachypodol	Plant
patchouli-alcohol	Plant
Patchoulipyridine	Plant
pentanoic-acid	Leaf
Phenol	Leaf
Pogostol	Plant
Rhamnetin	Plant
Seychellene	Plant
Tannin	Plant
trans-2-pentylcyclopropylcarboxylic-acid	Leaf/Plant
Caryophyllene	Plant
caryophyllene-oxide	Plant

There are different compositions of compound contributed depending on where the sample is collected. Usually part of the plant used in extraction of patchouli essential oil is the leave or the flower. Therefore, the main chemical components of patchouli oil are β-Pinene, β-Elemene, β-Caryophyllene, α-Guaiene, β-Cubebene, β-Patchoulene, α-Patchoulene, α-Selinene, α-Bulnesene, Patchouli alcohol as shown in Table 2.3 (Tsai *et al.*, 2006).

Table 2.3: Main compounds of the *P.cablin* essential oil

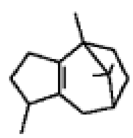
Compound	**Retention Time (°) (HP-5MS column)**	**Area (%)**
β-Pinene	14.38	1.00
β-Patchoulene	34.21	12.12
β-Elemene	34.58	1.23
β-Caryophyllene	35.74	3.89
α-Guaiene	36.78	20.62
α-Patchoulene	37.41	10.52
β-Cubebene	38.69	1.34
α-Selinene	39.07	4.17
α-Bulnesene	39.24	16.18
Patchouli alcohol	45.25	11.12

2.2.5 Chemical Structure in Patchouli Oil

In 2005, Hu *et al.* obtained fourteen batches of *P.cablin* samples from various areas of China and had separated and purified to analyze them. The structures were confirmed by comparing UV, MS and NMR data with literature. One year later, Tsai *et al.* (2006) analyzed steam distillate essential oil from the aerial parts of *P.cablin* from commercial plant in China by gas chromatography-mass spectrometry (GC-MS). Both of them summarize the same five major compounds found in the essential oil as follow:

1. β-Patchoulene
2. α-Guaiene
3. α-Patchoulene
4. α-Bulnesene
5. Patchouli alcohol

The structures of the above listed compound are shown in Figure 2.5 (Deguerry *et al.*, 2006):



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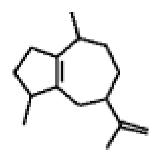

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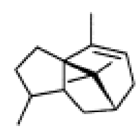

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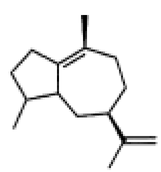

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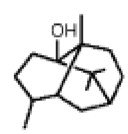

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Figure 2.5: Structure of major constituents from *P.cablin*

2.2.6 Patchouli Oil Industry

Patchouli oil industry is still dealing to have a cost-effective and efficient extraction route. The demand keeps on increasing by years. Patchouli oil reached highest exportation during year 1993 when the exporter gained US$ 20.691.000 per year profit. Past 10 years, gradual demands reach almost 6% per year. For year 2004, the profit reached US$ 27.137.000 (Feri, 2007)

Therefore, many researches have been conduct to determine the best method. Some of the factor that can affect patchouli oil quality are extraction time, pressure, temperature, and sample mass. In this study, the focus is on the extraction time and sample mass. From past study longer extraction time have higher yield (Nufus, 2004). Same goes to the sample mass factor as higher sample mass is proportional to higher oil yield (Nufus, 2004).

2.3 Extraction of Essential Oil

There are various ways to extract essential oil from aromatic plant. The type of botanical plant material that distinct the method of extraction. For example, the available methods are hydrodistillation, steam distillation and turbo ultrasonic method. There is also CO_2 extracts, also known as supercritical extracts or supercritical fluid CO_2 extracts which are extremely pure plant extracts produced from a relatively new and highly efficient extraction process. However this method is very costly.

Although the extraction of essential oils may sound only to be of technical interest, it is one of the key points which determine the quality of the oil that is used, since a wrong or wrongly executed extraction, can damage the oil, and alter the chemical signature of the essential oil. (Essential Oils, 2007. *Extraction Method)*

2.3.1 Hydrodistillation

Hydrodistillation or better known as water distillation is a method where the material to be distilled comes in direct contact with boiling water. It may float on the water or completely immersed, depending upon its specific gravity and the quantity of material handled per charge. The water is boiled by application of heat by any of the usual method i.e., direct fire, steam jacket, closed steam coiled, or, in a few cases, open or perforated steam coil. The characteristic feature of this method lies in the direct contact it affords between the boiling water and plant material. Some plant materials (e.g., powdered almonds, rose petals, and orange blossoms) must be distilled while fully immersed and moving freely in boiling water, because on distillation with injected live steam (direct steam distillation) these material agglutinate and form large compact lumps, through which the steam cannot penetrate (Guenther,1949).

2.3.2 Turbo Ultrasonic

Ultrasound can be successfully employed to enhance extraction when low boiling point solvents are used, and the temperature of the extraction mixture is kept below its boiling point. The extraction mechanism involves two types of physical phenomena: diffusion through the cell walls and washing out (rinsing) the cell contents once the walls are broken. Both phenomena are significantly effected by ultrasonic irradiation. Ultrasound can facilitate swelling and hydration and so cause an enlargement in the pores of the cell wall. This will improve the diffusion process and therefore enhancing mass transfer. Ultrasound increases the swelling index i.e. the water uptake by the plant material during sonication. The extractive value is much greater under sonication, compared with mechanical stirring. An increase in the swelling of plant tissue can, in some cases, break the cell walls which favors the washing out process.

2.3.3 Superficial Fluid CO_2

The CO_2 extraction process consists of pumping pressurized carbon dioxide into a chamber filled with plant matter. When carbon dioxide is subjected to pressure it becomes "supercritical" and has liquid properties while remaining in a gaseous state. Because of the liquid properties of the gas, the CO_2 functions as a solvent, pulling the oils and other substances such as pigment and resin from the plant matter. Thus, the difference between CO_2, or supercritical, extraction and traditional distillation is that CO_2 is used as a solvent instead of heated water or steam. The temperature involved in the supercritical extraction process is around 95 to 100^{o}F as opposed to 140 to 212^{o}F in steam distillation.

There are many positive aspects of the supercritical CO_2 extraction process and the resultant supercritical CO_2 essential oils. The CO_2 supercritical extraction process eliminates the need for potentially harmful solvents like hexane, avoiding unnecessary environmental pollution and potential human bodily harm. Another very important consideration is that the supercritical CO_2 extraction process avoids heat degradation to the plant matter, producing an essential oil that is a more authentic version of the original plant matter. Many medicinal properties of the plant are thus kept intact in the oil, exemplified by the aforementioned German chamomile extract. Another positive aspect to the CO_2 distillation process is the aroma of the essential oil. The CO_2 supercritical extract offers a more genuine aroma of the actual herb, spice or plant. The aroma of the CO_2 extracts of ginger, cardamom and other spices are more active, spirited and warm in nature than the rather flat and lifeless aroma of the same plants that have been steam distilled.

2.3.4 Steam Distillation Extraction

Steam distillation is also known as direct steam distillation. Live steam, saturated or superheated, and frequently at pressure higher than atmospheric pressure steam is injected through open or perforated steam coils below the charge, and proceeds upward through the charge above the supporting grid.

In this experiment, the extraction method used is steam distillation. When steam distillation is used in the manufacture and extraction of essential oils, the botanical material is placed in a still and steam is forced over the material. The hot steam helps to release the aromatic molecules from the plant material since the steam forces open the pockets in which the oils are kept in the plant material. The molecules of these volatile oils then escape from the plant material and evaporate into the steam. The temperature of the steam needs to be carefully controlled - just enough to force the plant material to let go of the essential oil, yet not too hot as to burn the plant material or the essential oil. The steam which then contains the essential oil is passed through a cooling system to condense the steam, which form a liquid from which the essential oil and water is then separated. The steam is produced at greater pressure than the atmosphere and therefore boils at above 100°C which facilitates the removal of the essential oil from the plant material at a faster rate and in so doing prevents damage to the oil. Some oils, like Lavender is heat sensitive (thermolabile) and with this extraction method, the oil is not damaged and ingredients like linalyl acetate will not decompose to linalool and acetic acid (Essential Oils, 2007. *Steam Distillation*).

2.4 Chromatography

Chromatography is an equipment to analyze a mixture. Used in a lab scale, it is a laboratory technique for the separation of mixture. A mixture dissolved in a "mobile phase" pass through a "stationary phase", which separates the analyte to be measured from other molecules in the mixture and allows it to be isolated. There are two common divided group of chromatography, preparative or analytical. Preparative chromatography seeks to separate the components of a mixture for further use while analytical chromatography normally operates with smaller amounts of material and seeks to measure the relative proportions of analytes in a mixture. Preparative chromatography is a form of purification. The two are not mutually exclusive. Retention times were utilized as primary criterion for the peaks identification. Retention time measure a speed at which a substance moves in a chromatographic system or can be measure as

$$R_f = \frac{\text{distance moved by compound}}{\text{distance moved by solvent}}$$

CHAPTER 3

METHODOLOGY

3.1 Introduction

Guenther (1949) stated that distillation may be defined as "the separation of the components of a mixture of two or more liquids by virtue of the difference in their vapor pressure" (as cited in Miall, 1940). In this study, steam distillation is used to extract patchouli essential oil from patchouli leaves. The process flow in steam distillation is shown in Figure 3.1 (Erowid,2007).

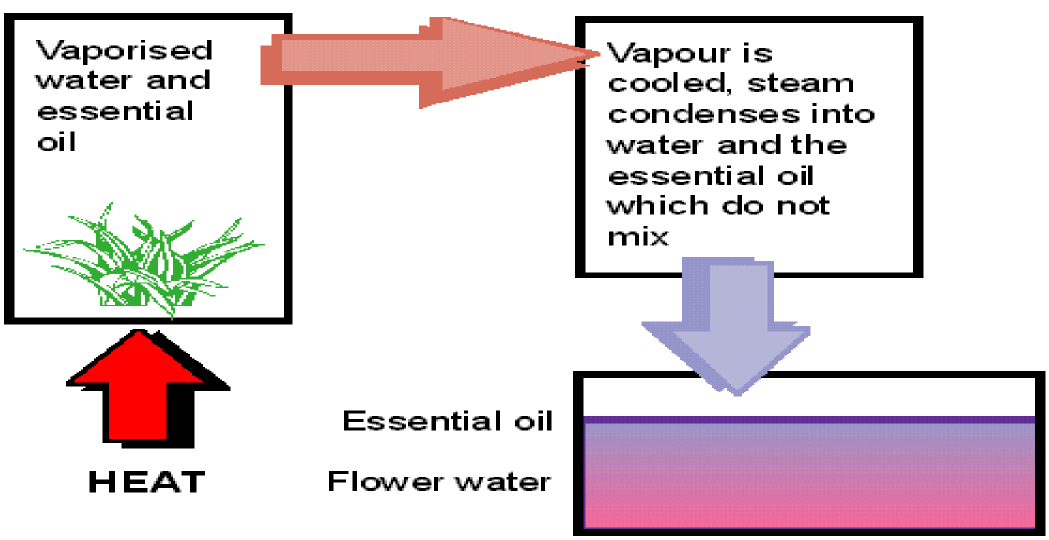

Figure 3.1: Process in steam distillation

3.2 Pretreatment

The preliminary step is important to let the plants completely dry from any moisture before the next step in the experiment. This will ensure that water will not distract the extraction process. The estimation of moisture content is around 15%.

Firstly, weight the plants and record the reading. Next step requires the plants to be dry under direct sunlight for 3 hours and expose to room temperature for 3 days. The leaves then are cut into 2cm in size pieces. This is to ensure that sufficient space provided for the steam to penetrate all part of the plant.

The volatile patchouli oil is enclosed within the plant tissues and cut off from direct contact with the steam by several layers of membrane, often very tough. For this reason most plant must be comminuted prior to distillation. Where steam distillation is practiced, this process should not be carried too far because the interspaces within the plant charge will then become too small. The rising steam must have sufficient space to penetrate all parts of the charge uniformly. Very small interspaces necessitate a slow, ineffective distillation, because any increase in pressure will cause the steam to break channels through the plant charge. (Guenther, 1949)

3.3 Extraction of Patchouli Oil

This process is where the steam passes over the plant material. The steam forces open the pockets in which the oils are kept in the plants and release the aromatic molecules from the leaves. The steam then condenses again to liquid form, and the essential oil separated from the liquid. The most pure essential oils are collected in this way.

In this study, the patchouli oil from the plant which majority of it is a patchoulol, is being extracted at $100^{o}C$ and pressure at slightly above ambient temperature. The essential oil will be collected in a beaker in a liquid phase with portion of water. The diagram of the process is shown in Figure 3.2 (Ccnphawaii,2007.*Distillation*). Figure 3.3 show the pilot plant steam distillation equipment.

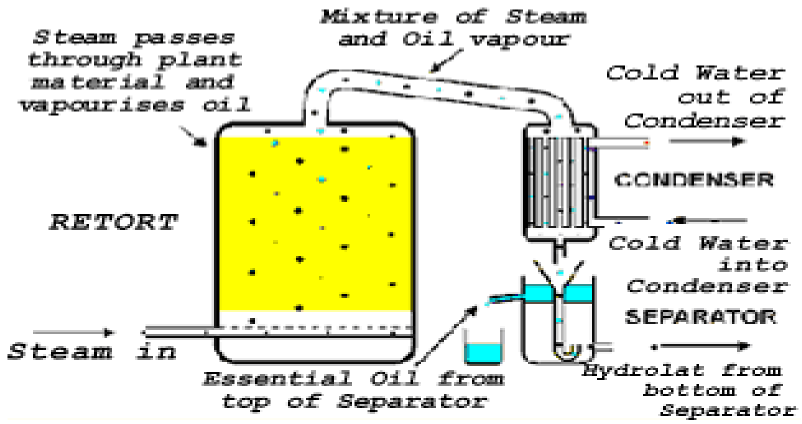

Figure 3.2: Schematic diagram of steam distillation equipment

Figure 3.3: Steam distillation

3.3.1 Start-Up Procedure

Firstly, the steam is passed through the steam distillation column without installing the top of the column (condenser and column cover). This step is to ensure that the water has fully turned to steam. At the same time make sure to fully open the drain valve at the bottom of the column and at the drain valve at the main pipeline. After the steam is fully in steam phase, close the main steam valve. Then, half-close the drain valve at the main pipeline and this valve is fixed at this position throughout the experiment.

3.3.2 Oil Yield Collection for Extraction Time Parameter

After the start up procedure, stack the patchouli plant tray by tray. There are three trays in this study. Therefore, each tray is stack with 1 kg plants. The column cover and the condenser are then installed and half-close the drain valve at the bottom of the column. After this process, open the main steam valve and let the steam at pressure slightly above ambient temperature passed through the plant. The temperature will rise to 100^{o}C after 30 minutes. The oil vaporizes with the rising steam and condenses to liquid phase by condenser. The first yield will be collect at 3 hours because this is the minimum time for oil collection based on literature review. The yield will be collected at one hour interval until 7 hours (3, 4, 5, 6 and 7 hours). This experiment will be conduct three times to get the average reading for each hour and for each run, it will be at constant mass (3kg).

3.3.3 Oil Yield Collection for Sample Mass Parameter

For this experiment, the same start up procedure need to be done. 2 kg patchouli plant is then scattered for each tray. The temperature will increase gradually to 100^{o}C and the oil will also vaporize and then condense to liquid phase by the condenser. The yield will be collected at the same interval, which is 3, 4, 5, 6 and 7 hours. This experiment will be run twice for each different sample mass.

3.4 Separation of Patchouli Oil and Water

The solution which contain a mixture of patchouli oil and water is collected and separate by using rotary evaporator at temperature above the boiling point of water, T=120^{o}C but below boiling point of patchouli oil and at speed of 90 rpm. The essential oil will separate from water after 30 minutes depending on ratio of water in the mixture. The separation takes longer time if the solution contains more water. Figure 3.4 show the equipment for separation process.

Figure 3.4: Rotary evaporator

3.5 Gas Chromatography Mass Spectrometer Analysis

The GC-MS is composed of two major building blocks: the gas chromatograph and the mass spectrometer. The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties.The difference in the chemical properties between different molecules in a mixture will separate the molecules as the sample travels the length of the column. The molecules take different amounts of time (called the retention time) to come out of (elute from) the gas chromatograph, and this allows the mass spectrometer downstream to capture, ionize, accelerate, deflect, and detect the ionized molecules separately. The mass spectrometer does this by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio (Wikipedia. 2007, *Gas Chromatography Mass Spectrometer*)

When the oil has already separated from water, the oil is then analyzing using gas chromatography mass spectrometer to determine the components of the oil. This analysis is to prove that the oil contain patchouli oil components. Only one sample will be analyzed using this gas chromatography because this analysis is not in the scope of study.

Figure 3.5: Gas chromatography mass spectrometer

3.6 Overall Process of Patchouli Oil Extraction

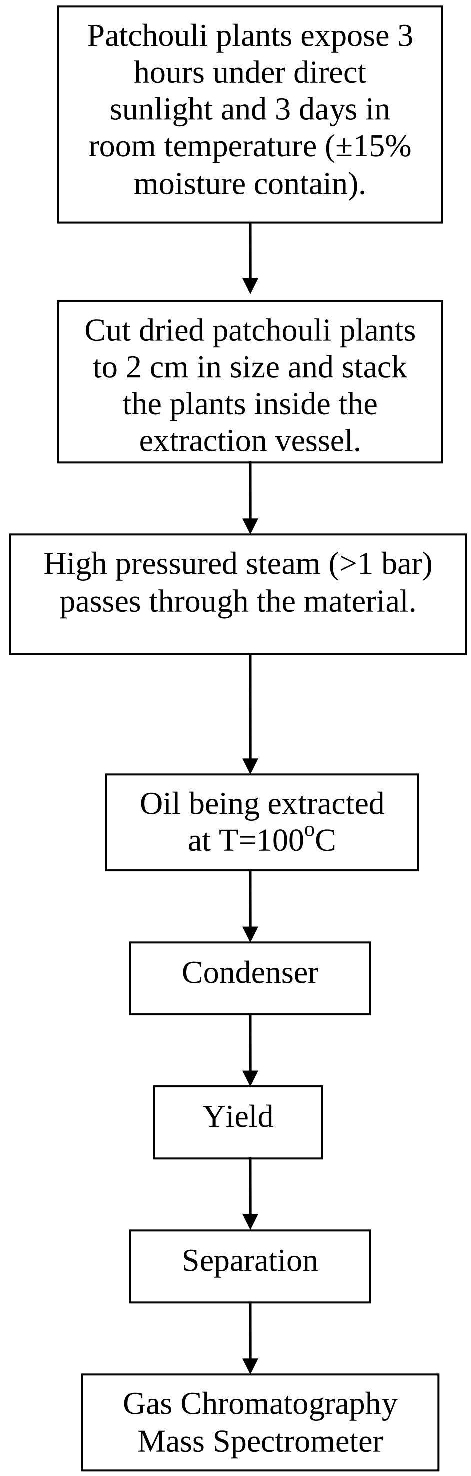

Figure 3.6: Process flow chart

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

"The Examination and Analysis of Essential Oil, Synthetics, and Isolates" describes the commercial methods of testing and evaluating the raw materials of the essential oil industry. Most of these methods have been used in New York and Clifton laboratories of Fritzche Brothers, Inc., during the course of the years. They frequently represent standard official procedures or modifications of such procedures (Edward, 1949). However in this study, the focus is on quantitative study and qualitative study of the yield of patchouli oil. The effect of stated parameter, extraction time and mass, will be discuss further in this section.

4.2 Observation

4.2.1 Quantitative Study on Effect of Different Extraction Time on Oil Yield

1. For this experiment the amount of raw material used is constant at 3 kg for all 3 runs.
2. The temperature of steam distillation took 30 minutes to increase to 100^oC, where at this temperature the first droplet of oil can be seen at the end of the condenser which connected to the separator.

3. The yield will be at the top of separator which contains water. The oil then flowed to a beaker from the top separator.
4. Oil with a small amount of water was collected from the beaker.
5. The first collection was done after 3 hours run.
6. The yield for first collection, 3 hours, was a light yellow. The temperature of the steam distillation is constant at 100^{o}C.
7. As for 4 hours, the colour became yellowish and gradually darker proportional to longer extraction time as shown in Figure 4.1.

Figure 4.1: Oil yield proportional to longer extraction time

8. The quantity of the oil increase as longer extraction time.
9. Proportional to longer extraction time, the odor of the oil also became stronger.

4.2.2 Quantitative Study on Effect of Different Sample Mass on Oil Yield

4.2.2.1 Sample Mass at 2 kg Patchouli Plant

1. The steam distillation took 15 minutes to reach to 100^{o}C.
2. At temperature 100^{o}C, the first droplet of oil can be seen at end of condenser tube which connected to the separator.
3. The oil with small amount of water was collected from the beaker.
4. For the first collection at 3 hours, shows certain amount of oil.
5. The oil was a light yellow in colour.

6. Gradually as longer extraction time, the oil became darker in colour.
7. Proportional to longer extraction time, the odor of the oil also become stronger.

4.2.2.2 Sample Mass at 4 kg Patchouli Plant

1. Longer time needed for the steam distillation to increase temperature to 100^oC, about more than 45 minutes.
2. The oil with small amount of water was collected from the beaker.
3. For the first yield collection at 3 hours, very small amount of oil can be collected. Therefore, the amount was negligible.
4. The practical amount of oil was collected at 4 hours.
5. The observation of colour and odor are the same as 2 kg patchouli plant run.

4.3 Result/Data Collection

4.3.1 Effect of Different Extraction Time on Oil Yield

Refer to Table 4.1. The table shows the data collection for volume of oil yield at different extraction time. The oil yield for the first collection, at 3 hours, is 13.3 mL. The reading increases gradually for the next hour. For 4 hours extraction time the reading is 16.7 mL and for 5 hours extraction time is 18.1 mL. At 6 hours extraction time the oil yield is 20.6 mL and for the last collection, at 7 hours extraction time, the yield is 23 mL.

Table 4.2 is the mass of oil yield from the same data collection of Table 4.1. The oil yield mass in gram is a value of mass of bottle with patchouli oil minus mass of empty bottle. The mass of oil yield also show increment from 12.0387 g for 3 hours extraction time, 14.7213 g for 4 hours extraction time, 15.699 for 5 hours extraction time, 17.9269 g for 6 hours extraction time to 20.2067 g for the last collection.

The oil yield percentage for this data collection is shown in Table 4.3. The volume-weight percent and the weight-weight percent also increase for longer extraction time. The highest value for volume-weight percent is 0.77% and for the weight-weight percent is 0.67%.

Table 4.3: Oil yield percentage for the effect of different extraction time

Oil Yield		**Time (hr)**				
		3	4	5	6	7
Oil Yield (mL)		13.3	16.7	18.1	20.6	23
Oil Yield (g)		12.0387	14.7213	15.699	17.9269	20.2067
Oil Yield (%)	v/w	0.44	0.56	0.6	0.69	0.77
	w/w	0.4	0.49	0.52	0.6	0.67

$$(v/w)\ \% = \frac{\text{volume of oil yield in mL}}{\text{mass of dried patchouli plant in gram}}$$

$$(w/w)\ \% = \frac{\text{mass of oil yield in grams}}{\text{mass of dried patchouli plant in grams}}$$

- For this parameter, the mass of dried patchouli plant is fixed at 3000g

4.3.2 Effect of Different Sample Mass on Oil Yield

Refer to Table 4.4 and Table 4.5. Both tables are data collection for the second parameter, different sample effect on oil yield. The oil yield for 2 kg, 3 kg and 4 kg sample mass all show increment value. At 2 kg mass sample, the yield show that less oil can be collected for every hour of extraction times comparing to 3 kg and 4 kg sample mass. At 7 hours extraction time, there is no more oil can be collect.

Table 4.4: Volume and mass of oil yield for the effect of different sample mass

Mass of Sample (kg)	**Oil Yield**	**Time (hr)**				
		3	4	5	6	7
2 kg	volume (mL)	2	3.8	6.4	7.4	7.4
	mass (g)	1.657	2.642	4.164	5.876	5.876
3 kg	volume (mL)	13.3	16.7	18.1	20.6	23
	mass (g)	12.039	14.721	15.699	17.927	20.207
4 kg	volume (mL)	0	2.5	14.5	19.3	23.7
	mass (g)	0	1.833	12.229	16.559	19.645

Table 4.5: Oil yield percentage for the effect of different sample mass

Mass of Sample (kg)	**Oil Yield**	**Time (hr)**				
		3	4	5	6	7
2 kg	Volume	2	3.8	6.4	7.4	7.4
	v/w	0.1	0.19	0.32	0.37	0.37
	Mass	1.667	2.642	4.164	5.876	5.876
	w/w	0.082	0.132	0.208	0.294	0.294
3 kg	Volume	13.3	16.7	18.1	20.6	23
	v/w	0.44	0.557	0.603	0.687	0.767
	Mass	12.039	14.721	15.699	17.927	20.207
	w/w	0.401	0.491	0.523	0.598	0.674
4 kg	Volume	0	2.5	14.5	19.3	23.7
	v/w	0	0.063	0.363	0.483	0.593
	Mass	0	1.833	12.229	16.559	19.645
	w/w	0	0.046	0.306	0.414	0.491

Whereas, for 4 kg sample mass, the yield takes longer time to have a sufficient collection of oil yield. For the 3 hours collection, there is no oil yield make it 0% of oil yield collection. However for the next hour of extraction time, the value increases from 0.046 w/w% oil yield to 0.491 w/w% at 7 hours extraction time. Based from the collection of oil yield at 3 kg, at the end of the last collection for this 4 kg sample mass, it is assume that there is still left substantial amount of essential oil that still can be extract.

4.4 DISCUSSION

4.4.1 Effect of Different Extraction Time on Oil Yield

Figure 4.2 shows the volume of oil yield for the first parameter, effect of extraction time to oil yield. From the graph it shows that the volumes gradually increase either for the first run or second and third run.

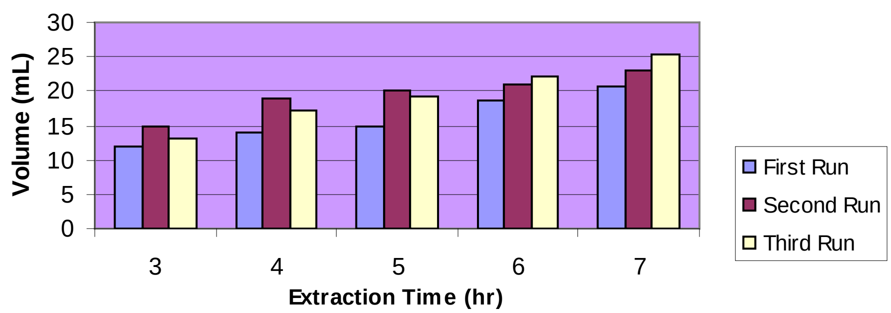

Figure 4.2: Volume of oil yield for the effect of different extraction time

Figure 4.3 prove this statement where the average readings show increment for every extraction hour. The longer extraction hour, in this case up until 7 hours, have higher volume yield. This result tally with literature review, saying that at longer extraction time will yield a higher quantity of oil (Nufus, 2004). The steam have longer time to penetrate and forces open the pocket in which the oil are kept hence able to vaporize more oil to condense with the steam at the collector.

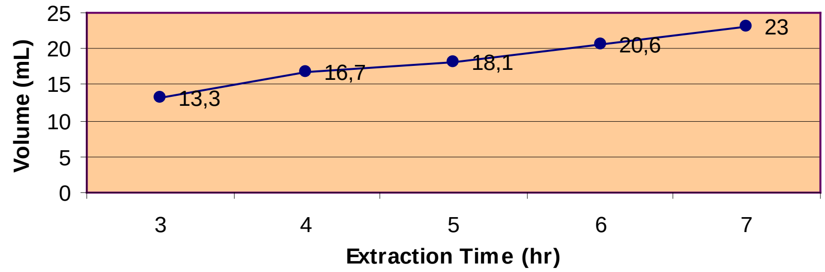

Figure 4.3: Average volume of oil yield for the effect of different extraction time

Figure 4.4 show the mass of oil yield for this data collection. This data indicate that the longer extraction time have higher mass yield of oil. The average of mass for every extraction hour, at Figure 4.5 also proved that the mass also increased with the longer extraction time. This is because at longer time, more compounds of the patchouli oil can be extracted from the plant material especially compound that have higher boiling point. At the last collection, many higher boiling point compounds can be found comparing to at early collection especially at the first collection, 3 hours extraction time.

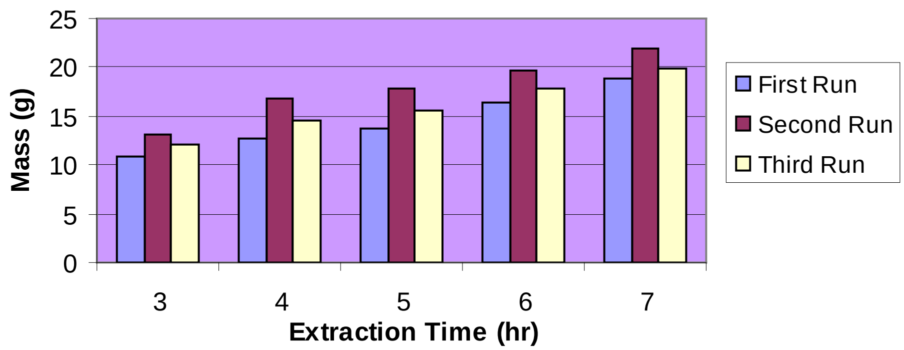

Figure 4.4: Mass of oil yield for the effect of different extraction time

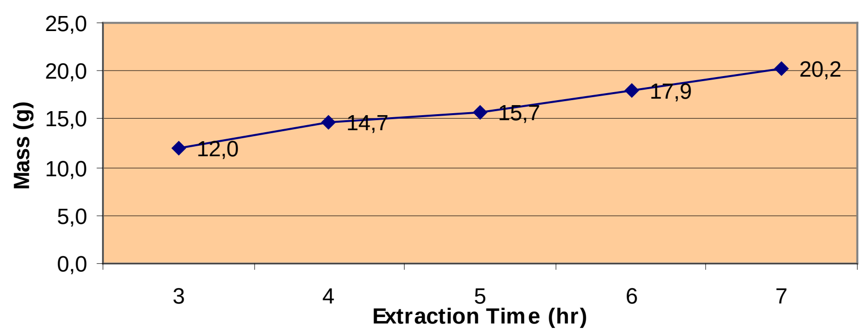

Figure 4.5: Average mass of oil yield for the effect of different extraction time

Oil yield percentage in volume-weight percent (v/w%) or weight-weight percent (w/w%) are shown in Figure 4.6. The figure can be the conclusion for the effect of extraction time. At longer extraction time more volume and mass of oil yield can be collect. Distillation must be long in order to obtain a maximum amount of different molecules in the essential oils because some molecules appear only at the end of distillation.

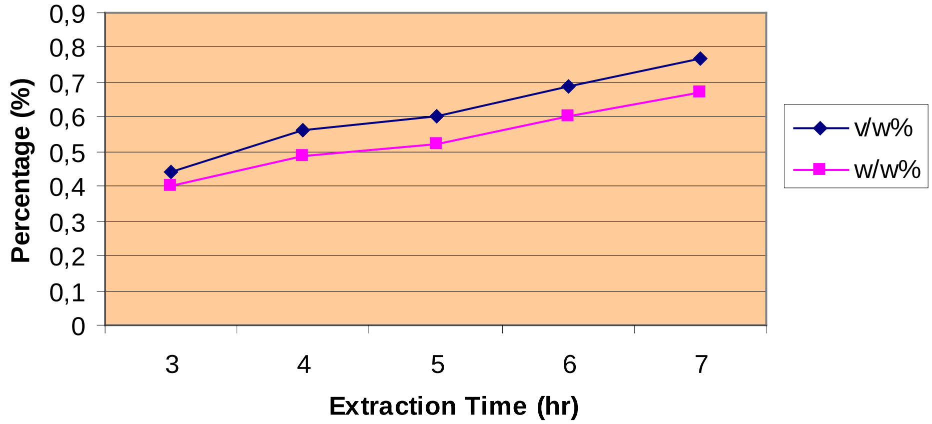

Figure 4.6: Oil yield percentage for the effect of different extraction time

For this parameter, the odor become stronger for longer extraction time and the colour become darker proportional to longer extraction time because at longer extraction time the higher boiling point component cause such characteristics to the oil yield.

4.4.2 Effect of Different Sample Mass on Oil Yield

Figure 4.7, shows the volume at different sample mass range from 2 kg to 4 kg. At 2 kg sample mass, the least volume of oil that can be collect at the end of last collection, 7 hours extraction time. For the 4 kg sample mass, the graph show rapid increment of volume yield comparing between this 3 samples mass. Hence, for this range of extraction time, the 3 kg sample mass is the optimum because have the constant rise of volume thus the economical oil yield collection.

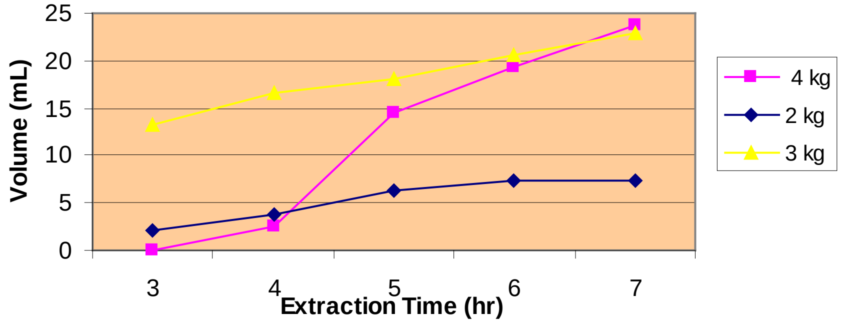

Figure 4.7: Volume of oil yield for the effect of different sample mass

The same goes for the mass of oil yield for this data collection, Figure 4.8. The 3 kg sample mass is the optimum for 3 hours to 7 hours range of extraction time with 2 kg to 4 kg of sample mass. Both, mass and volume of oil yield for 4 kg sample mass does not meet the literature where supposedly between this 3 samples mass, it should be the optimum oil yield collection is at 4 kg sample mass. The reason for this to happen is that at 4 kg sample mass, the temperature rising to 100^{o}C need longer time. For this equipment the plant material needs to be stacked compactly due to space constraint of the equipment. Therefore, the steam need longer time to penetrate to the plant especially to force open every pocket of the plant containing the patchouli essential oil component.

It is expected that at longer time which is more than 7 hours extraction time, more oil can be collected from 4 kg sample mass comparing to 3 hours extraction time because the graph intercept at 6.8 hours extraction time. Whereas, the 2 kg sample mass show that the value constant after 6 hours which indicate that there is no more oil after 7 hours extraction times.

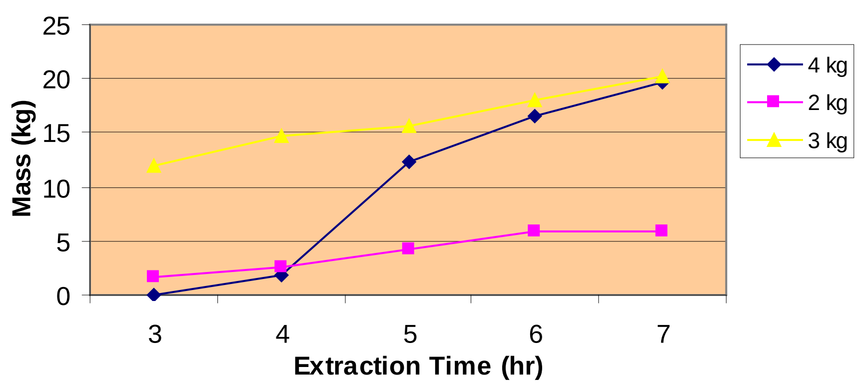

Figure 4.8: Mass of oil yield for the effect of different sample mass

Based on Figure 4.9 and Figure 4.10, the optimum oil yield collection for this range of parameter is proved to be at 3 kg sample mass. The highest oil yield percentage in v/w% and w/w% is at 3 kg sample mass. In effect, it complies with the earlier statement.

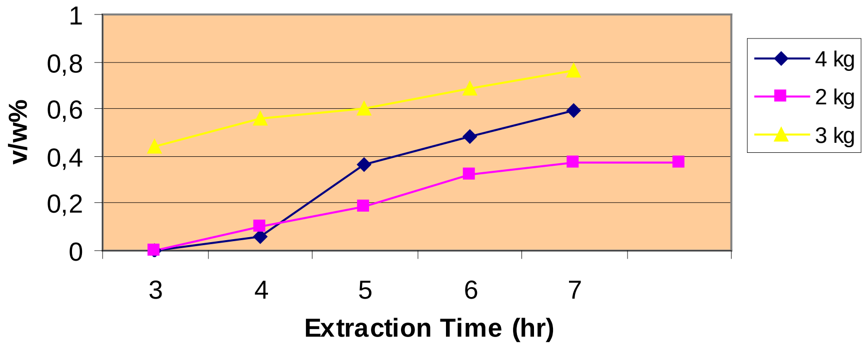

Figure 4.9: Volume-weight percentage (v/w%) of oil yield for the effect of different sample mass

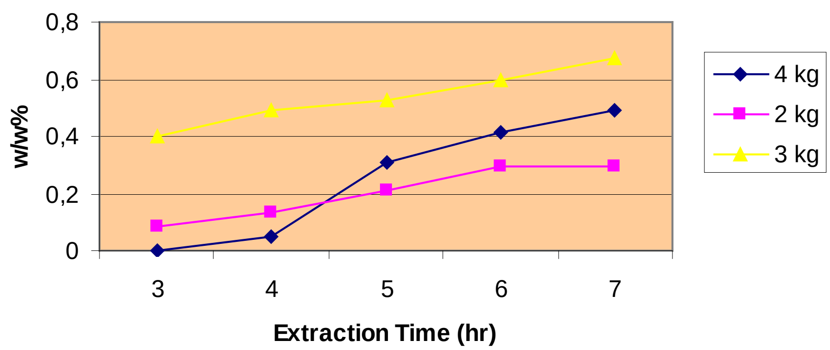

Figure 4.10: Weight-weight percentage (w/w%) of oil yield for the effect of different sample mass

4.4.3 Gas Chromatography Mass Spectrometer Analysis

In this case of study, the GC/MS is run mainly to determine that the yield is patchouli essential oil. From the chromatogram Figure 4.11 and Figure 4.12, the analysis shows that the highest peak, at retention time 14.741 and 14.891, is prove to be patchouli alcohol which have the biggest percent are, 11.05% and 14.58% respectively. This meet the characteristic of patchouli oil which major contain is patchouli alcohol. In this chromatogram however have many peak due to the present of other component such as β-patchoulene, other components of patchouli oil and some component due to contamination before GC/MS run.

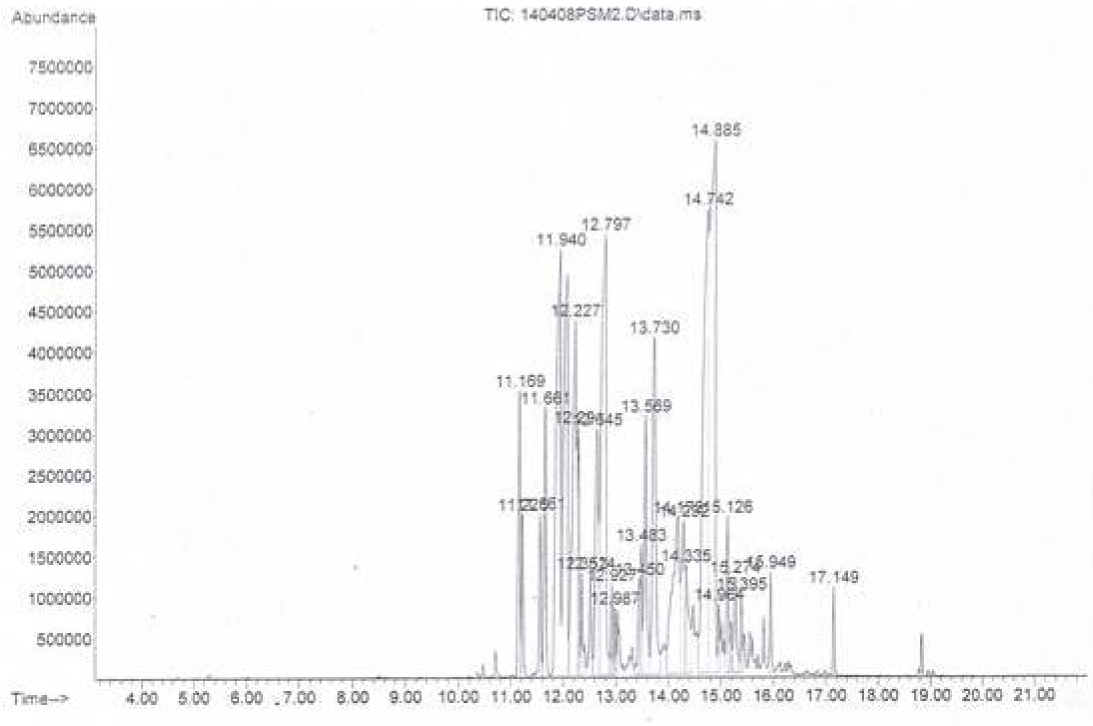

Figure 4.11: Chromatogram of patchouli oil analysis

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21  14.741 11.05 C:\Database\NIST05a.L
                  Patchouli alcohol                    72914 005986-55-0 86
                  Azulene, 1,2,3,3a,4,5,6,7-octahydr   60067 022567-17-5 83
                  o-1,4-dimethyl-7-(1-methylethenyl)
                  -, [1R-(1.alpha.,3a.beta.,4.alpha.
                  ,7.beta.)]-
                  Patchouli alcohol                    72910 005986-55-0 78

22  14.891 14.58 C:\Database\NIST05a.L
                  Patchouli alcohol                    72916 005986-55-0 93
                  Patchouli alcohol                    72914 005986-55-0 91
                  Patchouli alcohol                    72910 005986-55-0 90
```

Figure 4.12: Mass spectral interpretation of patchouli oil analysis

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 CONCLUSION

The purpose for this research is to obtain the feasibility of the maximum condition of steam distillation in patchouli oil extraction process. Therefore two parameter that possible to be study for this equipment have been identified which is effect of different extraction time on oil yield and the effect of different sample mass on oil yield. For the effect of extraction time, longer extraction time yield more quantity of oil. Second parameter, sample mass experiment also proved that higher sample mass is proportional to higher oil yield provided that longer extraction time. However, for economical of this equipment, it is proved that 3 kg sample is the optimum sample mass between 2 kg, 3 kg and 4 kg sample mass and at 7 hours extraction time.

Therefore, for this equipment with the range of 7 hours extraction time and 2 kg to 4 kg sample masses, the optimum extraction time is at 7 hours with 3 kg sample mass.

5.2 RECOMMENDATION

Recommendation is more on the equipment modification. The equipment need to have a steam flowrate meter indicator and controller. The steam pipeline to the equipment also need to be insulated because of heat loss to the environment will decrease the pressure and temperature to the equipment. Thus, lead to inefficient extraction. Chiller to the condenser also need a flowarate meter indicator because sometime while running the experiment, the chiller flowrate is not constant that lead to insufficient condensation of oil to liquid phase.

For separation process of water and oil, it is advisable to find another separation method because for little amount of oil is adhere to the rotary evaporator wall. The oil that already separated from oil need to be keep in cool place to preserve the quality and kept tightly in a bottle so that it would not vaporize to the air.

In this experiment, the raw material was already dried when delivered. The exact number of moisture content is not recordable. For next study, it is advisable to do the pre-treatment process before extracting so that a proper drying process conducted to determine moisture content thus increase the extraction efficiency. Also, the raw material in this study did not grinded earlier due to lack of proper pilot plant grinding equipment. Therefore, for next study, grinded raw material is best to be used for extraction of patchouli plant using steam distillation. Lastly, try to extend the extraction time for more than 7 hours to collect the remaining oil because it is believed that for extraction more than 2 kg sample masses there is significant amount of oil left.

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Table 4.1: Volume of oil yield for the effect of different extraction time

Volume (mL)	Time (hr)									
	3		4		5		6		7	
	V_1	$\Sigma (V_1)$	V_2	$\Sigma (V_1+V_2)$	V_3	$\Sigma (V_1+V_2+V_3)$	V_4	$\Sigma (V_1+V_2+V_3+V_4)$	V_5	$\Sigma (V_1+V_2+V_3+V_4+V_5)$
First Run	12	12	2	14	1	15	3.5	18.5	2.2	20.7
Second Run	15	15	4	19	1	20	1	21	2	23
Third Run	13	13	4.2	17.2	2	19.2	3	22.2	3	25.2
Average	$\frac{12+15+13}{3}$ = **13.3**		$\frac{14+19+17.2}{3}$ = **16.7**		$\frac{15+20+19.2}{3}$ = **18.1**		$\frac{18.5+21+22.2}{3}$ = **20.6**		$\frac{20.7+23+25.2}{3}$ = **23**	

V_1, V_2, V_3, V_4 and V_5 = oil yield at 3, 4, 5, 6 and 7 hours respectively in mL

$\Sigma (V_1)$ = total oil yield that can be extracted at 3 hours in mL

$\Sigma (V_1+V_2)$ = total oil yield that can be extracted at 4 hours in mL

$\Sigma (V_1+V_2+V_3)$ = total oil yield that can be extracted at 5 hours in mL

$\Sigma (V_1+V_2+V_3+V_4)$ = total oil yield that can be extracted at 6 hours in mL

$\Sigma (V_1+V_2+V_3+V_4+V_5)$= total oil yield that can be extracted at 7 hours in mL

Table 4.2: Mass of oil yield for the effect of different extraction time

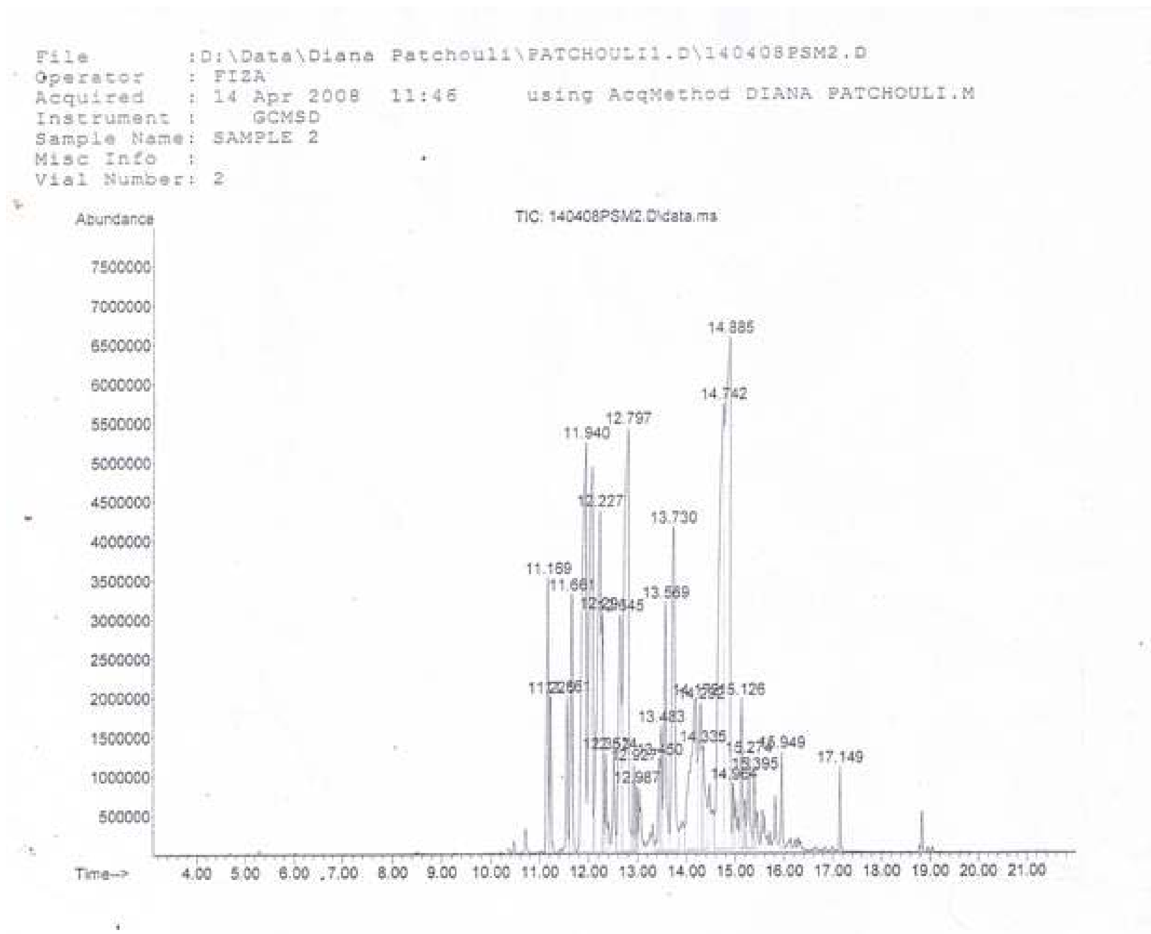
Mass (g)		**Time (hr)**				
		3	4	5	6	7
First Run	x	21.2024	21.1430	21.1485	21.1694	21.2119
	y	32.1042	23.0213	22.0951	23.8996	23.5557
	z	10.9018	1.8783	0.9466	2.7302	2.3438
	Σ z	10.9018	12.7801	13.72670	16.4569	18.8007
Second Run	x	21.2481	21.0817	21.0561	21.2501	21.1561
	y	34.3502	24.7280	22.0851	23.0452	23.4541
	z	13.1021	3.6463	1.029	1.7951	2.298
	Σ z	13.1021	16.7484	17.7774	19.5725	21.8705
Third Run	x	21.1717	21.0795	21.6541	21.3341	21.4210
	y	33.2838	23.6026	22.6115	23.4925	23.6187
	z	12.1121	2.5231	0.9574	2.1584	2.1977
	Σ z	12.1121	14.6352	15.5926	17.7510	19.9487
Average	$\frac{\Sigma z}{3}$	**12.0387**	**14.7212**	**15.6989**	**17.9268**	**20.2066**

x = mass of empty bottle in grams

y = mass of bottle with patchouli oil in grams

z = y-x = mass of patchouli oil in grams

APPENDIX A



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Time-->
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13.730
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12.987

APPENDIX B

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                 [1S-(1.alpha.,4.alpha.,7.alpha.)]-
  β-patchoulene  4,7-Methanoazulene, 1,2,3,4,5,6,7,      60013 000514-51-2 91
                 8-octahydro-1,4,9,9-tetramethyl-,
                 [1S-(1.alpha.,4.alpha.,7.alpha.)]-
                 4,7-Methanoazulene, 1,2,3,4,5,6,7,      60012 000514-51-2 90
                 8-octahydro-1,4,9,9-tetramethyl-,
                 [1S-(1.alpha.,4.alpha.,7.alpha.)]-

2   11.227  1.30 C:\Database\NIST05a.L
                 Cyclohexane, 1-ethenyl-1-methyl-2,      60003 000515-13-9 60
                 4-bis(1-methylethenyl)-, [1S-(1.al
                 pha.,2.beta.,4.beta.)]-
                 1H-Pyrrole, 1-(2-furanylmethyl)-        21692 001438-94-4 46
                 1,5-Cyclooctadiene, 1,5-dimethyl-       15260 003760-14-3 43

3   11.558  1.32 C:\Database\NIST05a.L
                 4-Fluorobenzoic acid, pent-2-en-4-      59609 1000299-15-2 47
                 ynyl ester
                 Bioallethrin                           126072 000584-79-2 43
                 2-Cyclohexene-1-carbonyl chloride,      47007 100131-18-8 43
                  1,4,4-trimethyl-

4   11.665  2.81 C:\Database\NIST05a.L
                 Caryophyllene                           59797 000087-44-5 99
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                 Bicyclo[5.2.0]nonane, 2-methylene-      59917 242794-76-9 92
                 4,8,8-trimethyl-4-vinyl-

5   11.942 15.58 C:\Database\NIST05a.L
                 Azulene, 1,2,3,4,5,6,7,8-octahydro      60026 003691-12-1 99
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                 , [1S-(1.alpha.,4.alpha.,7.alpha.)
                 ]-
                 Azulene, 1,2,3,4,5,6,7,8-octahydro      60027 003691-12-1 98
                 -1,4-dimethyl-7-(1-methylethenyl)-
                 , [1S-(1.alpha.,4.alpha.,7.alpha.)
                 ]-
                 Azulene, 1,2,3,4,5,6,7,8-octahydro      60028 003691-12-1 87
                 -1,4-dimethyl-7-(1-methylethenyl)-
                 , [1S-(1.alpha.,4.alpha.,7.alpha.)
                 ]-

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                 1H-3a,7-Methanoazulene, 2,3,6,7,8,      60042 000560-32-7 86
                 8a-hexahydro-1,4,9,9-tetramethyl-,
                  (1.alpha.,3a.alpha.,7.alpha.,8a.b
                 eta.)-
                 1H-3a,7-Methanoazulene, 2,3,6,7,8,      60044 000560-32-7 83
                 8a-hexahydro-1,4,9,9-tetramethyl-,
                  (1.alpha.,3a.alpha.,7.alpha.,8a.b
                 eta.)-
                 1H-Benzocycloheptene, 2,4a,5,6,7,8      59997 003853-83-6 38
                 ,9,9a-octahydro-3,5,5-trimethyl-9-
                 methylene-, (4aS-cis)-

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                  -1,4-dimethyl-7-(1-methylethenyl)-
                  , [1S-(1.alpha.,4.alpha.,7.alpha.)
                  ]-
                  .beta.-Humulene                      59811 000116-04-1 58
                  1H-Cycloprop[e]azulene, 1a,2,3,5,6   60086 021747-46-6 46
                  ,7,7a,7b-octahydro-1,1,4,7-tetrame
                  thyl-, [1aR-(1a.alpha.,7.alpha.,7a
                  .beta.,7b.alpha.)]-

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                  ydro-1,1,3a-trimethyl-7-methylene-
                  , [1aS-(1a.alpha.,3a.alpha.,7a.bet
                  a.,7b.alpha.)]-
                  Azulene, 1,2,3,3a,4,5,6,7-octahydr   60066 022567-17-5 90
                  o-1,4-dimethyl-7-(1-methylethenyl)
                  -, [1R-(1.alpha.,3a.beta.,4.alpha.
                  ,7.beta.)]-
                  1H-Cyclopropa[a]naphthalene, 1a,2,   60074 000489-29-2 90
                  3,3a,4,5,6,7b-octahydro-1,1,3a,7-t
                  etramethyl-, [1aR-(1a.alpha.,3a.al
                  pha.,7b.alpha.)]-

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                  1,1,7-trimethyl-4-methylene-, [1aR
                  -(1a.alpha.,4a.beta.,7.alpha.,7a.b
                  eta.,7b.alpha.)]-
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                  etramethyl-, [1aR-(1a.alpha.,3a.al
                  pha.,7b.alpha.)]-
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                  o-1,4-dimethyl-7-(1-methylethenyl)
                  -, [1S-(1.alpha.,7.alpha.,8a.beta
                  )]-
                  Azulene, 1,2,3,5,6,7,8,8a-octahydr   60031 003691-11-0 98
                  o-1,4-dimethyl-7-(1-methylethenyl)
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                  Azulene, 1,2,3,5,6,7,8,8a-octahydr   60035 003691-11-0 98
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3	12.989	0.45	C:\Database\NIST05a.L			
			Isoaromadendrene epoxide	71364	1000159-36-6	25
			Caryophyllene oxide	71352	001139-30-6	25
			2,3-Hexadiene, 2-methyl-	2824	029212-09-7	25
4	13.448	0.81	C:\Database\NIST05a.L			
			Phenol, 2-ethyl-	9606	000090-00-6	30
			Phenol, 3,5-dimethyl-	9631	000108-68-9	30
			Phenol, 2,5-dimethyl-	9630	000095-87-4	30
5	13.481	1.08	C:\Database\NIST05a.L			
			Caryophyllene oxide	71352	001139-30-6	42
			2-Isopropylidene-3-methylhexa-3,5-dienal	22844	1000191-76-5	30
			(1H)Imidazole-4-acetonitrile	5005	018502-05-1	30
6	13.566	3.17	C:\Database\NIST05a.L			
			1-Pentene, 5-(2,2-dimethylcyclopropyl)-2-methyl-4-methylene-	32238	1000150-39-5	22
			Phenol, 2-ethyl-	9598	000090-00-6	18
			3,3-Dimethyl-6-methylenecyclohexen	9738	020185-16-4	18
7	13.726	5.47	C:\Database\NIST05a.L			
			Caryophyllene oxide	71352	001139-30-6	91
			Cyclohexene, 3-methyl-6-(1-methylethenyl)-, (3R-trans)-	15383	005113-87-1	55
			Bicyclo[6.1.0]nonane, 9-(1-methylethylidene)-	32218	056666-90-1	53
8	14.175	5.32	C:\Database\NIST05a.L			
			Isoaromadendrene epoxide	71364	1000159-36-6	49
			Alloaromadendrene oxide-(1)	71377	1000156-12-8	43
			Cyclohexane, 1-methyl-2,4-bis(1-methylethenyl)-	41749	061142-58-3	41
9	14.292	2.39	C:\Database\NIST05a.L			
			1H-Cycloprop[e]azulene, decahydro-1,1,7-trimethyl-4-methylene-, [1aR-(1a.alpha.,4a.alpha.,7.alpha.,7a.beta.,7b.alpha.)]-	60079	000489-39-4	53
			Naphthalene, decahydro-4a-methyl-1-methylene-7-(1-methylethenyl)-, [4aR-(4a.alpha.,7.alpha.,8a.beta.)]	60015	017066-67-0	49
			1,4-Methanoazulene, decahydro-4,8,8-trimethyl-9-methylene-, [1S-(1.alpha.,3a.beta.,4.alpha.,8a.beta.)]	60020	000475-20-7	49
0	14.335	1.72	C:\Database\NIST05a.L			
			1H-Cycloprop[e]azulen-7-ol, decahydro-1,1,7-trimethyl-4-methylene-, [1ar-(1a.alpha.,4a.alpha.,7.beta.,	71465	006750-60-3	64

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			dro-1,1,7-trimethyl-4-methylene-,			
			[1ar-(1a.alpha.,4a.alpha.,7.beta.,			
			7a.beta.,7b.alpha.)]-			
			Isoaromadendrene epoxide	71364	1000159-36-6	47
21	14.741	11.05	C:\Database\NIST05a.L			
			Patchouli alcohol	72914	005986-55-0	86
			Azulene, 1,2,3,3a,4,5,6,7-octahydr	60067	022567-17-5	83
			o-1,4-dimethyl-7-(1-methylethenyl)			
			-, [1R-(1.alpha.,3a.beta.,4.alpha.			
			,7.beta.)]-			
			Patchouli alcohol	72910	005986-55-0	78
22	14.891	14.58	C:\Database\NIST05a.L			
			Patchouli alcohol	72916	005986-55-0	93
			Patchouli alcohol	72914	005986-55-0	91
			Patchouli alcohol	72910	005986-55-0	90
23	14.965	0.49	C:\Database\NIST05a.L			
			.alpha.-Farnesene	59827	000502-61-4	42
			Cyclohexane, 1,5-diethenyl-2,3-dim	32246	068779-14-6	35
			ethyl-, (1.alpha.,2.alpha.,3.alpha			
			.,5.beta.)-			
			Longifolenaldehyde	71344	019890-84-7	30
24	15.126	1.37	C:\Database\NIST05a.L			
			2H-Cyclopropa[a]naphthalen-2-one,	69993	006831-17-0	50
			1,1a,4,5,6,7,7a,7b-octahydro-1,1,7			
			,7a-tetramethyl-, (1a.alpha.,7.alp			
			ha.,7a.alpha.,7b.alpha.)-			
			1H-Cycloprop[e]azulene, decahydro-	60077	025246-27-9	48
			1,1,7-trimethyl-4-methylene-, [1aR			
			-(1a.alpha.,4a.beta.,7.alpha.,7a.b			
			eta.,7b.alpha.)]-			
			2(3H)-Naphthalenone, 4,4a,5,6,7,8-	69988	019598-45-9	46
			hexahydro-4a,5-dimethyl-3-(1-methy			
			lethylidene)-, (4ar-cis)-			
25	15.275	1.35	C:\Database\NIST05a.L			
			Isoaromadendrene epoxide	71364	1000159-36-6	68
			Ledene oxide-(I)	71329	1000151-93-3	62
			.beta.-Humulene	59811	000116-04-1	60
26	15.393	0.56	C:\Database\NIST05a.L			
			Eudesma-4(14),11-diene	59851	1000152-04-3	83
			1-Cycloheptene, 1,4-dimethyl-3-(2-	59937	1000159-38-6	70
			methyl-1-propene-1-yl)-4-vinyl-			
			Ledol	72882	000577-27-5	64
27	15.948	0.75	C:\Database\NIST05a.L			
			Benzene, 1-isothiocyanato-4-methyl	22616	000622-59-3	38
			6-Methylthieno[2,3-b]pyridine	22608	001759-30-4	38
			N,N,2,4-Tetramethylaniline	22424	000769-53-9	38
28	17.144	0.54	C:\Database\NIST05a.L			
			Pentadecanoic acid, 14-methyl-, me	105662	005129-60-2	98
			thyl ester			

Library Search Report

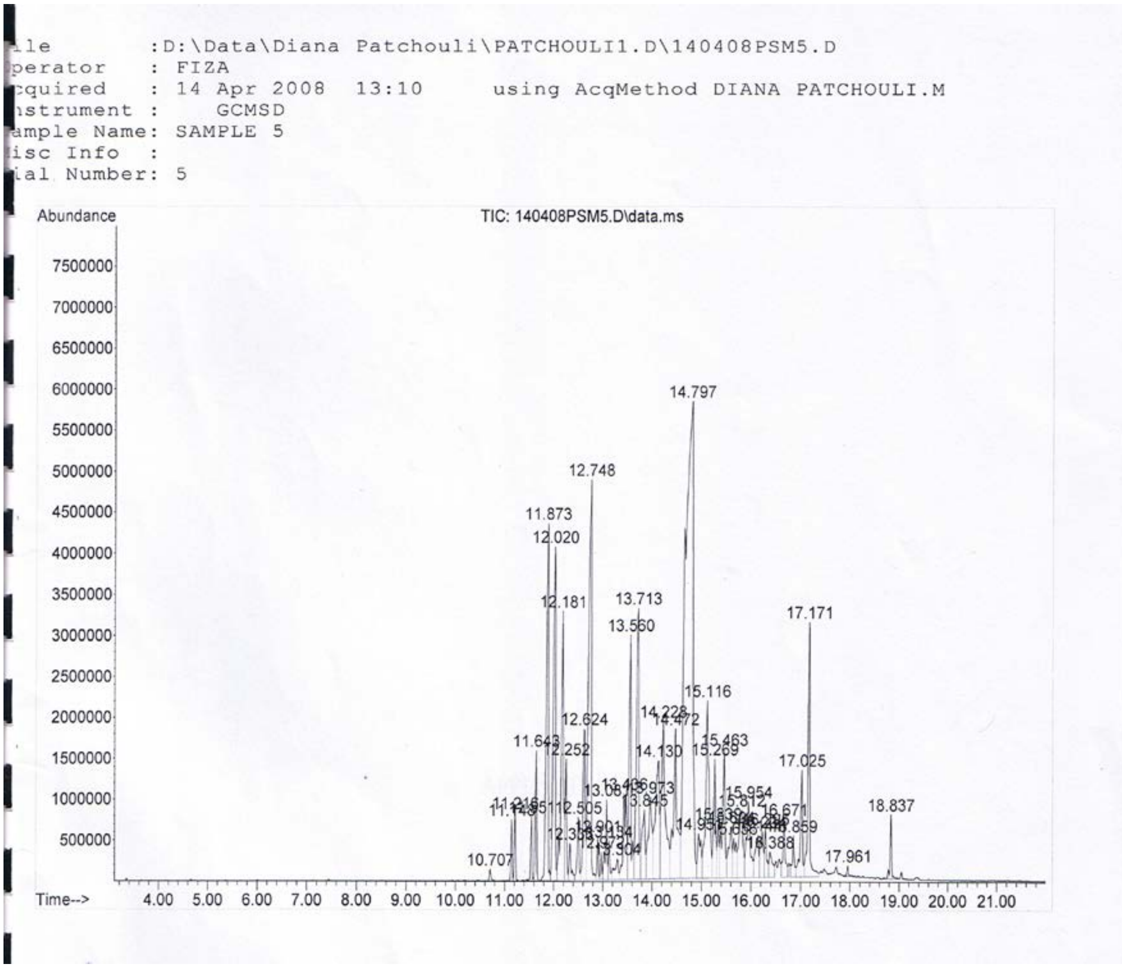
Data Path : D:\Data\Diana Patchouli\PATCHOULI1.D\
Data File : 140408PSM2.D
Acq On : 14 Apr 2008 11:46
Operator : FIZA
Sample : SAMPLE 2
Misc :
ALS Vial : 2 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

:#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			Hexadecanoic acid, methyl ester	105639	000112-39-0	97
			Hexadecanoic acid, methyl ester	105644	000112-39-0	96

APPENDIX C


ile :D:\Data\Diana Patchouli\PATCHOULI1.D\140408PSM5.D
perator : FIZA
cquired : 14 Apr 2008 13:10 using AcqMethod DIANA PATCHOULI.M
nstrument : GCMSD
ample Name: SAMPLE 5
isc Info :
ial Number: 5
Abundance
TIC: 140408PSM5.D\data.ms
Time-->
14.797
12.748
11.873
12.020
12.181
13.713
13.560
17.171
15.116
12.624
17.025
18.837
17.961
10.707

APPENDIX D

```
Library Searched : C:\Database\NIST05a.L
Quality          : 99
ID               : Patchouli alcohol
```