

EXTRACTION OF ESSENTIAL OIL FROM *CINNAMOMUM*
ZEYLANICUM BY VARIOUS METHODS AS A PERFUME OIL

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ABSTRACT

Cinnamomum Zeylanicum is a very popular spice and very useful substances in medicines and food, said to be originated from the island Sri Lanka, southeast of India. The plant is also playing an important role in aromatherapy due to its chemical constituent and also its aroma and scent. It contains cinnamaldehyde, an aromatic compound that have a very pleasant smell that can relax and soothe the mind and body, and also eugenol that have a strong aromatic odor and a spicy, pungent taste. The aims of this research are to extract and obtain essential oils from *Cinnamomum zeylanicum* using hydro distillation technique and ultrasonic extraction method, to analyze the chemical compound present in the essential oil using Gas Chromatography-Mass Spectrometer (GCMS), and to use the extracted essential oil in aromatherapy as a perfume oil. The hydro distillation method is used to obtain the essential oil from *Cinnamomum Zeylanicum* by grinding the leaves into a fine powder, weighing and then extracted the essential oil by Soxhlet apparatus while by ultrasonic extraction, the samples will soak in a mixture of ethanol and water in ultrasonic bath then will centrifuge to separate the solid and liquid. Next, the sample will be analyzed by GS/MS technique after rotary evaporating to separate between oil and water, in order to determine the chemical composition in the leaves of the plant. The percentage of essential oil yield is calculated as the weight of essential oils divided by the weight of leaf powder. Then, the essential oil will be tested as aromatherapy oil by using sensory evaluation. The result showed only essential oil by hydrodistillation contains eugenol and others 29 volatile and aromatic compounds while the essential oil by ultrasonic extraction, it contains no eugenol but more antioxidant compound. The time of extraction and weight of dry leaves should be varied in order to get better results in term of yield and active compound in the essential oil.

ABSTRAK

Cinnamomum Zeylanicum adalah rempah yang sangat popular dan sangat berguna dalam ubat-ubatan dan makanan, berasal dari pulau Sri Lanka, tenggara India. Tumbuhan itu juga memainkan peranan yang penting dalam aromaterapi kerana komposisi kimia dan juga kerana aroma dan bau. Ia mengandungi cinnamaldehyde, sebatian aromatik yang mempunyai bau yang sangat menyenangkan yang boleh merehatkan dan menenangkan fikiran serta dan tubuh badan, dan juga mengandungi eugenol yang mempunyai bau aromatik yang kuat dan rasa yang pedas. Matlamat kajian ini adalah untuk mendapatkan pati minyak dari *Cinnamomum zeylanicum* dengan menggunakan teknik penyulingan hidro dan kaedah pengekstrakan ultrasonik, untuk menganalisis sebatian kimia yang terdapat di dalam pati minyak dengan menggunakan Gas Chromatography Mass Spectrometer (GCMS), dan untuk menggunakan minyak pati yang diekstrak sebagai minyak wangi. Cara penyulingan hidro yang digunakan untuk mendapatkan pati minyak dari *Cinnamomum Zeylanicum* dengan mengisar daun ke dalam serbuk halus, menimbang dan kemudian mengekstrak pati minyak dengan menggunakan radas Soxhlet, manakala cara pengekstrakan ultrasonik, serbuk daun *Cinnamomum Zeylanicum* akan dicampur dengan campuran etanol dan air dan akan direndam di dalam larutan ultrasonik, kemudian akan disentrifuse untuk memisahkan pepejal dan cecair. Seterusnya, sampel akan dianalisis oleh GC/MS teknik setelah proses pengewapan putaran untuk memisahkan antara minyak dan air, untuk menentukan komposisi bahan kimia yang terdapat di dalam tumbuhan tersebut. Peratusan hasil minyak pati dikira sebagai berat minyak pati dibahagikan dengan berat serbuk daun. Kemudian, minyak pati akan diuji sebagai minyak aromaterapi dengan menggunakan kaedah penilaian deria. Hasil yang diperoleh menunjukkan hanya minyak pati yang diekstrak oleh kaedah penyulingan hidro mengandungi eugenol dan 29 sebatian aromatik yang lain manakala minyak pati oleh pengekstrakan ultrasonik, tidak mengandungi eugenol tetapi mengandungi banyak sebatian antioksidasi. Masa pengekstrakan dan berat daun kering perlu diubah untuk mendapatkan keputusan yang lebih baik untuk peratusan hasil minyak dan bahan kimia yang aktif di dalam minyak pati.

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CHAPTER 1

INTRODUCTION

1.1 Overview of *Cinnamomum Zeylanicum*

Cinnamon, a very popular culinary spice and a very useful substance in medicines, is said to be originated from the island Sri Lanka, southeast of India. It is also native to south-west India and the Tenasserim Hills of Burma. It is also now cultivated in many tropical countries such as Vietnam and Madagascar and Mexico. Cinnamon is scientifically named as *Cinnamomum Zeylanicum Blume* or *Cinnamomum Verum J.S. Presl* and also known as Ceylon cinnamon, true cinnamon, Ceylon-Zimtbaum and cannelle de Ceylan.

1.2 Physical Properties of *Cinnamomum Zeylanicum*

The name cinnamon is consequent from the Greek word 'kinnamon'. *Cinnamomum Zeylanicum* belong to the diminutive evergreen tree in the Laurel (Lauraceae) family. The tree can grow up and reach a height in a range 6 m to 12 m. The stem is robust with between 30 – 60 cm diameters. Mature trees have a thick skin brown or gray and have many branches low with the tapered and rounded leaves. Cinnamon has the shape of small diameter stem and has a king-size or short. Exterior and interior color of cinnamon is light brown and the chemical properties of cinnamon are spicy, slightly sweet, warm and fragrant.



Figure 1.1 Bark of *Cinnamomum Zeylanicum*



Figure 1.2 Leaves of *Cinnamomum Zeylanicum*

1.3 Usage of *Cinnamomum Zeylanicum*

Cinnamon has been used for many purposes since very early times. Since the 16th century, cinnamon has been used as cooking spices and as prevention of food from stale. Cinnamon also used as flavoring in cookies, biscuits and cakes. It is also widely used in traditional and modern medicine, perfumes and aromatherapy.

In addition, in medicinal uses, it is used as a treatment for diarrhea, stomach upset, against respiratory ailments and gastric ulcers. The cinnamon bark may also possess a potentiating on insulin and can be used to treat type 2-diabetes and to lower serum cholesterol. (Khan et. al, 2003).

Next, the oil that extracted from cinnamon is employed in aromatherapy as a rub to promote blood circulation because the presence of blood thinning compound in it which help to reduce and remove pain. The pleasant aroma of cinnamon oil makes it a very effective to produce an effect to soothe and relax the mind, body and soul. It is also used as a room freshener and often added in potpourris and also can be used for the treatment of hypertension.

The essential oil of cinnamon contains both antifungal and antibacterial principles that can be used to prevent food spoilage due to bacterial contamination. (Fabio et. al., 2003). Furthermore, it is also proven that cinnamon oil is effective against some species of toxicogenic fungi (Junglal et. al., 2001) and respiratory tract pathogens. (Viollon and Chaumont, 1994).

1.4 Usual Method of Obtaining Cinnamon Essential Oil

In *Cinnamomum Zeylanicum*, there are many constituents such as cinnamaldehyde, eugenol, linalool and cinnamic acid. There is various types of extraction that used to obtain essential oils, which are solvent extraction, hydro distillation ultrasonic extraction and also shaking and stirring along with solvent. Solvent extraction method is particularly hard to control. In this method, the less polar of components presents in the herbs or spices which are called oleoresins are extracted together with the essential oils. In addition, small amounts of organic solvents can pollute the extraction product of essential oil and usually the final extract product is very viscous. Ultrasonic extraction uses ultrasonic vibrations to extract samples with polar solvents in an ultrasonic bath. This is often used for chemical extraction from solid samples because it's simple. These methods are in essence derivations of liquid-liquid extraction. Sample extraction is achieved by simply placing solid samples in centrifuge containers with organic solvents and shaking. After extraction, separate sample matrix and extracts by centrifugation or filtration.

1.5 Hydro distillation

The most popular physical method for essential oil isolation is distillation. Prior to distillation, plant materials are in most cases dried and are then suitably ground so that the oil sacs are broken and a maximum of surface area is exposed for efficient oil release. Suitably ground plant materials are placed in a boiler with water completely covering them. As heat is slowly applied, the steam alone will be initially formed, and the distillate will be clear. With continued heating, the essential oil starts

to distill over with the steam, and the distillate becomes milky white. Distillation is continued until the distillate becomes clear, with no more oil distilling over from the material. Even though the essential oils have relatively high boiling points, co distillation like this brings about a satisfactory recovery of the oil because, in accordance with Dalton's law, a mixture boils when the sum of the vapour pressures of the individual components equals the atmospheric pressure. It is because hydro distillation at reduced pressure is better because lower temperature is used. During the process, the essential oil components form an azeotropic mixture with water. Most of the essential oil does not mix well with water in the liquid phase so, after condensation, they are separated by decantation. However, this method is somewhat slow and requires close manual attention and separation of the oil and aqueous phases of the condensate.

1.6 Problem Statement

Generally, there are few problems that arise in *Cinnamomum Zeylanicum* extraction. They are due to too many types of extraction process can be done to obtain essential oil. The traditional method used to produce essential oils is from hydrodistillation process but solvent extraction is also a good extraction process because it will produce a very similar scent to the material from which it was derived. However, the extracted product can be polluted because small amounts of organic solvent so it is not safe anymore. New method of extraction is introduced for example ultrasonic extraction that didn't require much time to extract the essential oil and also a straightforward method. But, different method will give a different yield of the active compound in the essential oil. So, it needs to be determined which

method will produce a higher percentage of the active compound in essential oil of *Cinnamomum Zeylanicum*.

Most of essential oils have medicinal properties and it had been used for a thousand years ago. Today, essential oils from *Cinnamomum Zeylanicum* are widely used in medical field and also in aromatherapy. The aromatic properties of the *Cinnamomum Zeylanicum* which is eugenol and cinnamaldehyde it will give the best scent and fragrance in order to soothe and relax the both mind and soul just by healing them. Furthermore, it also can be used for the treatment of hypertension. Then, it's also used as an aromatherapy oil in order to circulate the blood circulation and remove the pain. (Tisserand, 1995). Therefore, with the aromatic compound found in essential oil of *Cinnamomum Zeylanicum*, it is needed to determine whether it can be aromatherapy or perfume oil or not.

1.7 Research Objectives

The main objectives of this research are:

1. To extract and obtain essential oils from *Cinnamomum Zeylanicum* using various method of extraction.
2. To analyze the chemical compound present in the essential oil using Gas Chromatography-Mass Spectrometer (GCMS).
3. To compare the yield between the essential oil of *Cinnamomum Zeylanicum* from different method of extraction.
4. To use the extracted essential oil as a perfume oil.

1.8 Scope of Proposed Study

This research is an experimental study of hydro distillation method using *Cinnamomum Zeylanicum* as raw material. In order to realize the objectives, three scopes have been identified. The scopes are:

- i. To know the yield of *Cinnamomun Zeylanicum* essential oils by different method of extraction.

The methods that will uses are hydrodistillation, ultrasonic extraction and shaking and stirring along with solvent. The part of plant which is leaves will used in order to get better yield of essential oils.

- ii. To analyze the product using chromatography.

This study is focusing on using the gas chromatography to analyze the active compound in the extracted essential oil of *Cinnamomum Zeylanicum* by different methods of extraction.

- iii. To determine whether the extracted essential oils of *Cinnamomum Zeylanicum* can be used as a perfume oil

The essential oil will be tested using the sensory analysis test based on scent whether it can be perfume oil or not.

1.9 Expected Outcome

The expected outcome of this research is expected that the essential oil of *Cinnamomum Zeylanicum* can be used in aromatherapy as perfume oil in order to promote blood circulation and relaxing the mind and body just by smell the odor.

1.10 Significance of Proposed Study

This research intends to produce essential oil by of *Cinnamomum zeylanicum* from leaves using the various processes of extraction and some other test such as sensory evaluation test. Essential oil also can be used in aromatherapy as perfume oil that can help to soothe and relax the mind and body. In addition, it can be used to promote good blood circulation which it will help to help significantly in removing pain.

1.11 Contribution of the Study

The hydro distillation is expected to produce the best quality of essential oils from *the Cinnamomum zeylanicum bark* and leaves. There are some expected results from this research:

- i. The equipment of extraction process will be more efficient and effective to obtain essential oils.
- ii. Potential savings in the operational cost.
- iii. The environmental friendly experiment will be conducted.
- iv. *Cinnamomum zeylanicum* in aromatherapy can be contributed to a modern of application in this world.

CHAPTER 2

LITERATURE REVIEW

2.0 Introduction

This chapter is about literature reviews of the articles of production of essential oils from *Cinnamomum zeylanicum* extraction by hydro distillation. There are six sub-chapters to look through about overview of *Cinnamomum zeylanicum*, essential oils, aromatherapy, extraction process, essential oil production method and sensory analysis.

2.1 Overview of *Cinnamomum Zeylanicum*

Cinnamomum zeylanicum is an herbal substance. The scientific name is *Cinnamomum verum* J. S. Presl or *Cinnamomum zeylanicum* Nees). it's characteristics are dried bark, freed from the outer cork and the underlying

parenchyma (ESCOP, 2003; European Pharmacopoeia 6.2, 2009). *Cinnamomum verum* J. S. Presl. is also known by the synonym *Cinnamomum zeylanicum* Blume and is member of Lauraceae family (Keller et al., 1992).

2.1.1 History of *Cinnamomum zeylanicum*

Cinnamon has been used as a spice for thousands of years. In Egypt, cinnamon was a spice used in embalming fluids for preserving cadavers. In Ayurvedic medicine, the world's oldest system, cinnamon bark was used as an antiemetic, anti-diarrheal, anti-flatulent and also as a general stimulant. Moreover, The Portuguese found cinnamon trees growing in Sri Lanka (Ceylon) in the early 16th century; they imported cinnamon to Europe during the 16th and 17th centuries afterwards. The Dutch occupied Sri Lanka in the mid-17th century until the British captured the island in 1796. The East India Company then became the main exporter of cinnamon to Europe. The Dutch cultivated cinnamon in Java and the exports of Ceylon cinnamon decreased as a result of heavy export duties. In spite of that, Sri Lanka is the only regular supplier of cinnamon bark and leaf oils. The food industry chooses Ceylon cinnamon, but pharmaceutical manufacturers use both, oils from Ceylon cinnamon (cinnamon oil) and from Chinese cinnamon (cassia oil) interchangeably. China is the foremost exporter of cassia cinnamon. (Barceloux, 2009).

Cinnamon belongs to the herbal substances with one of the longest medicinal traditions. Conversely, there is no real reference to Ceylon as its

original source. Dioskorides described 5 cassia and 7 cinnamon species. The latter ones were reported as possessing diuretic and digestive properties. From the 8th century, cinnamon was introduced in Europe as an expensive spice that only could be used by kings and popes. There is a written report on the use of cinnamon in an Arabic source from around 1275. In 1310, Johannes of Montevino confirmed the existence of cinnamon trees in Ceylon. About 100 years later, Nicolo Conit accommodated a real prescription of the cinnamon tree.

Introduced by the Portuguese, cinnamon of Ceylon remained the preferred substance in Europe. It has been described, that in 1536 cinnamon of Ceylon would have cost 40 times more than the cinnamon from Java or the Philippines. The herbal substance was first accumulated from wild growing trees. The farming of cinnamon began around 1765, when Holland took the lead in the origin area. The manufacturing was streamlined in the way that a better quality was obtained and Holland could contribute enough cinnamon to cover the European needs. When the English took over in 1796, the cinnamon cultivation and trading became a dominance of the English Eastern-Indian Company. Around 1833, when Holland developed the cultivation of cinnamon in Java and Sumatra, the herb and the essential oil were introduced into medicine as a stimulant. The oil was contrived for the first time by St Amando of Doornyk at the end of the 15th century.

2.1.2 Chemical Constituents in *Cinnamomum Zeylanicum*

Barceloux, 2008 mentions that volatile oils are products from the distillation process of the bark, leaves, flowers, or buds of *Cinnamomum* species and the chemical composition of these oils varies depending on the part of the plant used for the distillation process. Cinnamon bark and leaf oil are steam distillation products obtained from the inner bark and leaves, respectively, of *Cinnamomum Verum*. The main components of cinnamon bark oil are cinnamaldehyde while eugenol is the main components about 81-85% of cinnamon leaf oil. A commercial sample of essential oils from *Cinnamomum verum* contained approximately 63% cinnamaldehyde, 8% limonene, 7% eugenol, 5.5% cinnamaldehyde propylene, and 1-2% of a variety of terpenoid compounds which are pinene, camphene as measured by gas chromatography/mass spectrometry.

According to R. Wang et al., 2009, main constituents of the *Cinnamomum zeylanicum* leaf are 79.75% eugenol, 16.25% trans-cinnamaldehyde, 0.14% Linalool, Neohexane, cinnamyl alcohol and others various compounds of aldehyde, ester, alcohol, ketone and alkanes. Cinnamon bark contains up to 4% of essential oil consisting primarily of cinnamaldehyde (60-75%), cinnamyl acetate (1-5%), eugenol (1-10%) (WHO Vol. 1, 1999), β -caryophyllene (1-4%), linalool and (1-3%) and 1.8-cineole (1-2%). (ESCOP, 2003). Other constituents are oligopolymeric procyanidins, cinnamic acid, phenolic acids, pentacyclic diterpenes cinnzeylanol and its acetyl derivative cinnzeylanine and the sugar mannitol, L-arabino-D-

xylanose, L-arabinose, D-xylose, α -D-glucose as well as mucilage polysaccharides (Hänsel et al., 1992; ESCOP, 2003).

The essential oil of the bark is described in the European Pharmacopoeia, 2009. There exists a summary report on the essential oil of cinnamon bark by the Committee for Veterinary Medicinal Products. According to this information, the oil mainly contains cinnamaldehyde (55-76%), eugenol (5-18%) and saffrole (up to 2%). This document refers also to human use (CVMP 2000).

2.1.3 Cinnamaldehyde

3-Phenyl-2-propenal is known as cinnamaldehyde, is a pale yellow liquid with a warm, sweet,spicy odor and pungent taste reminiscent of cinnamon. It is found naturally in the essential oils of Chinese cinnamon (*Cinnamomum cassia*, Blume) (75–90%) and Ceylon cinnamon (*Cinnamomum zeylanicum*, Nees) (60–75%) as the primary component in the steam distilled oils. It also occurs in many other essential oils at lower levels.

Greater than 95% of the consumption of cinnamaldehyde occurs in flavor uses where a spicy, cinnamon character is needed. It is used in a large range of products including bakery goods, confection, and beverages and also in toothpastes, mouthwashes and chewing gum. Furthermore, it is also used effectively in air fresheners where odor neutralization

can be achieved by reacting with sulfur and nitrogen malodorants. Other uses include its capability as an animal repellent, its use in compositions to attract insects and demonstration of a positive antifungal activity. Cinnamaldehyde has been efficiently isolated in high purity by fractional distillation from cassia and cinnamon bark essential oils and also used in manufacturing protocols for the preparation of natural benzaldehyde through a retro-aldol process.

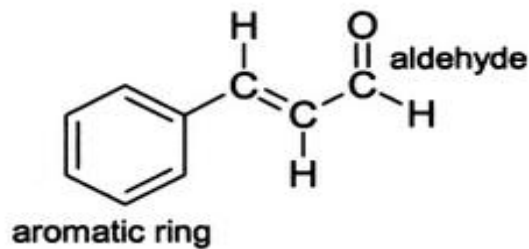


Figure 2.1 Cinnamaldehyde

2.1.4 Eugenol

According to Wikipedia, eugenol is a phenylpropene, an allyl chain-substituted guaiacol. Eugenol is a member of the phenylpropanoids class of chemical compounds. It is a clear to pale yellow oily liquid extracted from certain essential oils especially from clove oil, cinnamon, basil and bay leaf. It is slightly soluble in water and soluble in organic solvents and has a spicy, clove-like aroma. The name is derived from the scientific name

for clove, *Eugenia aromaticum* or *Eugenia caryophyllata*. Eugenol is responsible for the aroma of cloves. Eugenol is used in perfumeries, flavorings, essential oils and in medicine as a local antiseptic and anesthetic. It was used in the production of isoeugenol for the manufacture of vanillin, though most vanillin is now produced from phenol or from lignin. Eugenol derivatives or methoxyphenol derivatives in wider classification are used in perfumery and flavoring. They are used in formulating insect attractants and UV absorbers, analgesics, biocides, and antiseptics. Eugenol possesses significant antioxidant, anti-inflammatory and cardiovascular properties, in addition to analgesic and local anesthetic activity. (Pramod, Ansari and Ali, 2010).

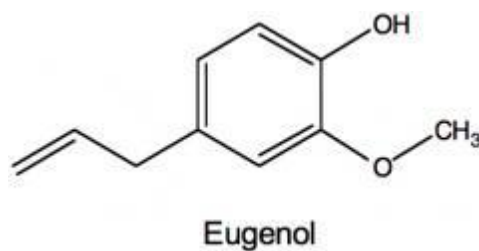


Figure 2.2: Eugenol

2.1.5 Usage of *Cinnamomum Zeylanicum*

When going back in history, cinnamon was used as a stomachic and in case of nerve weakness. As the gastrointestinal user is concerned, cinnamon was used in case of diarrhea, dyspepsia, and hyperacidity with reflux, vomiting and bloating (Madaus, 1938). It is also used poultices to treat minor bacterial and fungal infections of the skin externally and as a rub in

aromatherapy to promote blood circulation. (Tisserand, 1995). It is because the presence of blood thinning compound in it that will help to remove the pain and ensure the oxygen to supply to the body cells leading to higher metabolic activity. It is also suitable to use to increase the appetite and dyspepsia. (Blumenthal, 1998). Khan et. Al, 1990 state that cinnamon bark posses a potentiating effect on insulin which is a water soluble polyphenol compound and can be used in the treatment of type 2 diabetes as well as to lower triglyceride levels and serum cholesterol. (Khan et. al., 2003, Broadhurst et al. 2000, Onderolgu et al, 1999). It also can be used as perfume, room fresheners and also added in potpourris because of the pleasant smell and refreshing aroma that can soothe and relax the mind and body.

2.2 Essential oils

Shahzad, 2008 mentions that the essential oils carry the scent of the spices or aromatic plant in a concentrated form. The essential oils are volatile and do not contain the very high boiling or non-volatile components that are responsible for the characteristic taste of certain spices. For a thousand years ago, essential oil has played an important role in our daily lives. It is said to be used as anointing and healing the sick. (Doterra, 2008). The first people used the essential oils is the Egyptians in medical practice, beauty treatment, food preparations and also in religious ceremony. Apart from that, the Greek uses the essential oil in therapeutics massage and aromatherapy practices meanwhile the Romans use the essential oils to promote health and personal hygiene. It is followed by Chinese and Ayurvedic