A STUDY ON MICROWAVE-ASSISTED EXTRACTION OF ZINGIBER AROMATICUM

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" I declare that this thesis 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."

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Dedicated, in thankful appreciation for support, encouragement and understanding to my beloved family, friends and my supervisor.

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ABSTRACT

The objective of this research is to extract zingiber aromaticum essential oil by using a microwave assisted extraction (MAE) with solvent. Rhizome of zingiber aromaticum accumulates large amount of essential oil that contains bioactive sesquiterpenoid *zerumbone* which is widely used in medicine sectors. This research has focused on the influence of microwave power (500 Watt) at various natures of solvents and times of the extraction of zingiber aromaticum essential oil. Four types of solvents are used in this research which is ethanol, methanol, hexane and water. In this research, the methods of drying, grinding, extraction, separation and analysis are used and the sample is separated from solvents by using a rotary evaporator to get the essential oil. The sample was analyzed by using a GC-MS to identify the component of zingiber aromaticum oil. The optimum yield of zerumbone is 17.72 % at microwave power of 500 W and 10 minutes of extraction by using methanol as solvent.

ABSTRAK

Objektif penyelidikan ini adalah bagi mengekstrak minyak pati zingiber aromaticum dengan menggunakan satu gelombang mikro bagi membantu pengekstrakan dengan pelarut. Rizom zingiber aromaticum berkumpul jumlah luas minyak pati yang mengandungi bioaktif sesquiterpenoid zerumbone yang adalah digunakan secara meluas dalam sector ubat-ubatan. Penyelidikan ini telah tertumpu kepada pengaruh gelombang kuasa mikro (500 Watt) pada sifat semulajadi pelbagai pelarut di pelbagai masa bagi pengekstrakan minyak pati zingiber aromaticum. Empat jenis pelarut adalah digunakan dalam penyelidikan ini yang adalah etanol, metanol, heksana dan air. Dalam penyelidikan ini, kaedah-kaedah bagi pengeringan, pengisaran, pengekstrakan, pemisahan dan analisis adalah digunakan dan sampel dipisahkan daripada pelarut dengan menggunakan satu penyejat berputar untuk mendapat minyak pati. Sampel itu telah dianalisis dengan menggunakan satu GC-MS untuk mengenal pasti komponen zingiber aromaticum minyak. Hasil optimum zerumbone adalah 17.72 % pada gelombang kuasa mikro 500 W dan 10 minit pengekstrakan dengan menggunakan metanol sebagai pelarut.

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LIST OF ABBREVIATIONS

MAE	-	Microwave Assisted Extraction
MAP	-	Microwave Assisted Process
GC	-	Gas Chromatograph
MS	-	Mass Spectrometer
W	-	Watt

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CHAPTER 1

INTRODUCTION

1.1 Zingiber Aromaticum

Zingiber aromaticum also called ‘Puyang’ is a medicinal plant, a beautiful ginger, originally from Indonesia. The bioactive sesquiterpenoid *zerumbone* has anticancer properties. This plant may be effective as an anticancer agent and it also exhibits HIV-inhibitory [1].Zerumbone is a sesquiterpene phytochemical from a type of edible ginger known as ‘Zingiber Zerumbet Smith’ grown in Southeast Asia or ‘Zingiber aromaticum’[2]. It also currently being explored for its effects on cancer in general, Leukemia in particular, as well as HIV [2].

Figure 1.1: Zingiber Aromaticum plant [2].

Figure 1.2: Zingiber Aromaticum rhizome

1.2 Microwave Assisted Extraction(MAE)

Extraction is the first step for the preparation of medicine from raw plant materials and significantly affects the cost of the whole manufacture process[3].Microwave assisted extraction(MAE) is a new separation technique that combines the uses of energy of microwave radiation and traditional solvent extraction. Microwave extraction of biologically active compounds was first presented by Ganzler as a novel and effective sample preparation technique in 1986[4].By using a closed system, extraction can be performed at higher temperatures and extraction time can be reduced drastically. This technique can be applied to both liquid and gas phase extraction. Several studies show that microwave assisted extraction has many advantages, such as shorter time extraction, less solvent needed, higher extraction rate and better products with lower cost[5,6].

Figure 1.3: Microwave Extractor

Table 1.1 : Summary of Microwave Extraction Process

	Minimum	**Maximum**
Extraction Time(min)	10	300
Microwave Power(W)	30	1000
Spectrum Frequency(GHz)	30	300

1.3 Problem Statement

The concentration and purification of *zerumbone* have been important processes for many years. The conventional methods used so far are mainly based on solvent extraction and steam distillation. The drawbacks linked to these techniques have led to the searching for new alternative extraction processes. The commercial methods currently used for folding or unfolding essential oils are fractional vacuum distillation, selective solvent extraction and chromatographic separation. All these methods have drawbacks such as low yield, formation of by-products owing to the time exposure to high temperature and the presence of toxic organic residues in the extracts.

1.4 Objective

The objective of this research is to extract *zerumbone* with microwave assisted extraction (MAE) by using solvents extraction.

1.5 Scope of Research Work

This research focus on two main scopes:

i) Investigate the influence of extraction time.

ii) Investigate the influence of nature of solvent on extraction of *zerumbone.*

1.6 Rationale and Significance

It is very efficient to extract zerumbone using microwave assisted extraction rather than conventional technique.

CHAPTER 2

LITERATURE REVIEW

2.1 Essential Oil

Essential oil is define as concentrated, hydrophobic liquid containing volatile aroma compounds from plants, which are called aromatic herbs or aromatic plants. They are also known as volatile or ethereal oils, or simply as the "oil of" the plant material from which they were extracted. An oil is ‘essential’ in the sense that it carries a distinctive scent, or essence, of the plant. They are mixtures of more than 200 compounds that can be grouped into two fractions, a volatile fraction, that constitutes 90 – 95 % of the whole oil and contains monoterprenes and sesquiterprene hydrocarbon and their oxygenated derivatives, along with aliphatic aldehydes, alcohols and esters, and a non-volatile residue, that constitutes from 5 to 10 % of the world oil and contains hydrocarbons, fatty acids, sterols, carotenoids, waxes, coumarins, psoralens and flavonoids [7].

Essential oils are generally extracted by distillation. Other processes include expression, or solvent extraction. They are used in perfumes, cosmetics and bath products, for flavoring food and drink, and for scenting incense and household cleaning products.

Various essential oils have been used medicinally at different periods in history. Medical applications proposed by those who sell medicinal oils range from skin treatments to remedies for cancer, and are often based on historical use of these oils for these purposes.

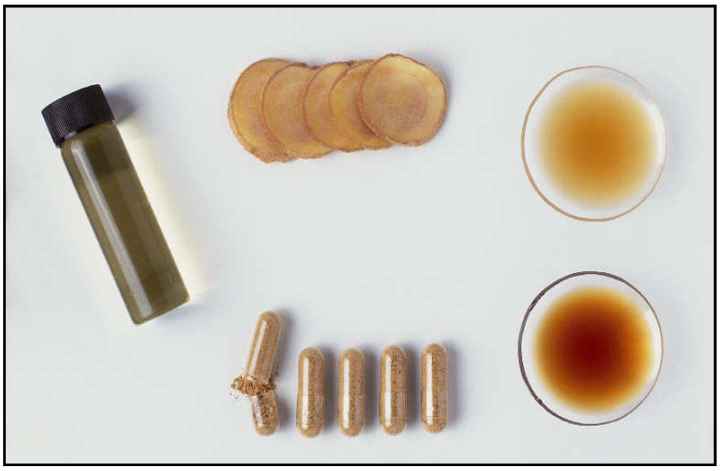

Figure 2.1: Applications of essential oil

Interest in essential oils has revived in recent decades, with the popularity of aromatherapy, a branch of alternative medicine which claims that the specific aromas carried by essential oils have curative effects.

2.2 *Zerumbone*

Zerumbone is the main component of the essential oil of a Zingiber Aromaticum, is a monocyclic sesquiterpene containing a cross-conjugated dienone system. It exhibits a variety of interesting reactions. It was found that some of the new zerumbone derivatives possessed intriguing bioactivities [8, 9]. Much of its chemistry remains to be explored in order to exploit the ready availability of this substance as a versatile starting material for conversion to other useful compounds. Figure 2.1 shows the chemical structure of zerumbone.

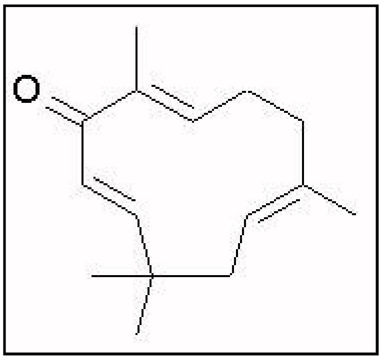

Figure 2.2: Structure of *Zerumbone*($C_{15}H_{22}O$) [10].

The Zingiber aromaticum essential oil contains of Zerumbone (2, 6, 9 humulatriene-8-one), 2, 3-epoxy-6, 9-humuladien-5-ol-8-one, 2, 3-epoxy-6, 9-humuladien-5-ol-8-one, these are all sesquiterpenoids. kaempferol-3-O-(2, 3-di-O-acetyl- -L-rhamnopyranoside), kaempferol-3-O-(2, 3, 4-tri-O-acetyl- L-rhamnopyranoside) these are flavonol glycosides.

2.2.1 Uses of *Zerumbone*

There are many uses of zerumbone recorded especially in medical field. There are several researches that had been done in making zerumbone as medical treatment.

The medicinal properties of Zerumbone can be summarized as follows as treatment for cancer[11], treatment for Leukemia[12], treatment for tumor[13], anti-inflammatory[14], exhibited HIV-inhibitory[15] and as a new anticancer for liver cancer[16].

2.3 Microwave Assisted Extraction (MAE)

Microwave Assisted Extraction (MAE) is a simple technique that provides a novel way of extraction soluble products into a fluid, from a wide range of materials, helped by microwave energy [17].The technique can be applied to both liquid phase extraction like isolation of essential oil is based on a basic physical principle, namely the different ability to absorb microwave energy depending on the chemical nature of the species being subjected to microwave irradiation. The parameter generally used as a measurement of this property is the dielectric constant. Thus, liquid phase extraction assisted by microwaves is based on the fact that it is possible to immerse the matrix to be extracted into a solvent that is characterized both by flow at low dielectric constant and relative transparency to microwave.

The first applications of the technique dealt with the extraction of essential oils from plant product [18].The kinetics of the microwave extraction of rosemary leaves in hexane, ethanol, or hexane-ethanol mixtures, as well as the influence of factors such as the source of the leaf, the microwave energy, duration of irradiation and sample load, on the rate of extraction of the compounds have been reported

[19].In more research, MAE has been coupled with liquid chromatography for the determination of herbicides in plant tissue [20].

Moreover, MAE offers other advantages over conventional technique, such as reduced energy consumption, smaller volumes of chemical solvent, uses of less toxic solvents and a smaller quantity of waste products. Compared with the traditional methods, MAE has many advantages, such as shorter time, less solvent, higher extraction rate, better products with lower cost because the microwave heat the solvent or solvent mixture directly, and the direct interaction of microwave with the free water molecules presents in the glands and vascular systems, which results in the subsequent rupture of the plant tissue and release of the active compounds into the organic solvent.

2.3.1 Microwave Theory

Microwaves are non-ionizing electromagnetic waves of frequency between 300 MHz to 300 GHz and positioned between the X-ray and infrared rays in the electromagnetic spectrum [21]. In modern day science, microwave serves two major purposes: communication and as energy vectors. The latter application is direct action of waves on materials that has the ability to convert a part of the absorbed electromagnetic energy to heat energy.

Microwaves are made up of two oscillation perpendicular field's for heating [21]. Unlike conventional heating which depends on conduction where convection loses the heat energy to the environment as the heating occurs in a closed system. This unique heating mechanism can significantly reduce the extraction time which normally less than 30 minutes as compare to Soxhlet [22].Figure 2.3 shows the electromagnetic spectrum in terms of frequency, wavelength and photon energy and the common names given to each region of the spectrum.

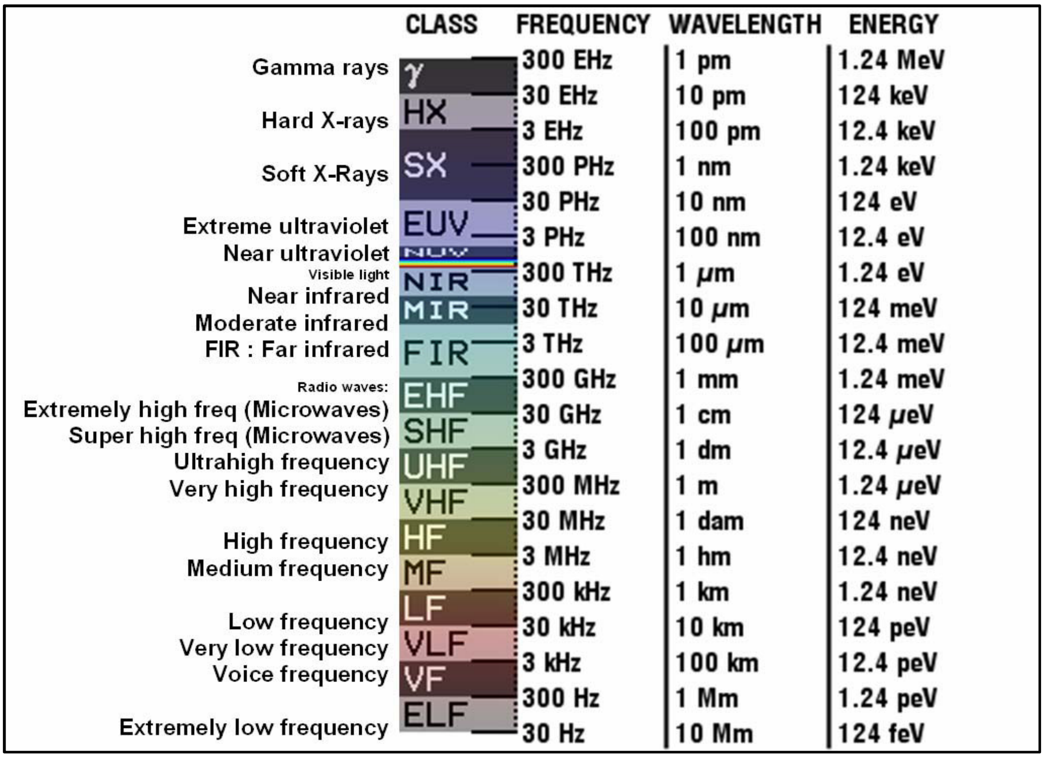

Figure 2.3: The electromagnetic spectrum [23]

The principle of heating using microwave is based upon its direct impact with polar materials/solvents and is governed by two phenomenons': ionic conduction and dipole rotation, which in most cases occurs simultaneously [21, 24].

Ionic conduction refers to the electrophoretic migration of ions under the influence of the changing electric field. The resistance offered by the solutions to the migration of ions generates friction, which eventually heats up the solution. Dipole rotation means realignment of the dipoles of the molecule with the rapidly changing electric field.