

EXTRACTION OF ESSENTIAL OIL FROM ORANGE PEELS**ONG HUI FONG**

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ABSTRACT

The microwave-assisted solvent extraction (MASE) technique is a promising technique which is highlighted by increased extraction yield, decreased time and solvent consumption; moreover the reproducibility is better. Isolation of citrus essential oils from orange peels was analyzed by gas chromatography-mass spectrometry (GC-MS). It was found that main constituents in orange oil which separated by using deionized water solvent is limonene. Further studies also revealed that yield of essential oils were mainly influenced by different extraction temperature, time and power. Limonene products are being used in a wide range of industrial, institutional, commercial, medical and residential cleaning applications. In this study, this product has been developed as a natural soap and air freshener .

ABSTRAK

Pengekstrakan pelarut bantuan gelombang mikro (MASE) ialah teknik yang menjanjikan peningkatan hasil pengekstrakan, menjimatkan jangka masa dan penggunaan pelarut, lebih-lebih lagi reproducibility adalah lebih baik. Pengasingan minyak sitrus penting dari kulit oren telah dianalisis dengan kromatografi gas-spektrometri jisim (GC-MS). Ia mendapati bahawa unsur-unsur yang utama dalam minyak oren yang dipisahkan dengan menggunakan pelarut deionized air ialah limonene. Kajian lanjutan juga mendedahkan bahawa hasil minyak pati kebanyakannya dipengaruhi oleh suhu, masa, dan kuasa pengekstrakan yang berbeza. Produk limonene sedang digunakan dalam pelbagai industri, institusi, perdagangan, permohonan pembersihan perubatan dan kediaman. Dalam kajian ini, produk ini telah dibangunkan sebagai sabun semula jadi dan penyegar udara.

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LIST OF SYMBOLS

°C	Degree Celsius
%	Percentage
W	Watts
min	Minutes
g	Grams
L	Liter
mL	Mili Liter
m	Meter
μL	Micro –Liter
w	Weight
w/w	Weight of Oil /Weight of raw materials
μL	Micro –Liter

LIST OF ABBREVIATIONS

HD	Hydrodistillation
MAHD	Microwave-Assisted Hydrodistillation
SI	International System of unit
W	Watt
MASE	Microwave-Assisted Solvent Extraction
GC-MS	Gas Chromatography- Mass Spectrometer
MSD	Mass selective detector

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Orange juice is one of the most widely-consumed beverages today in Malaysia. Approximately 50-60% of the processed fruits is transformed into citrus peel, which is composed of peels, seeds and membrane residues. With the increase in production of processed fruit wastes generated is increasing enormously. Large amounts of these wastes poses the problem of disposal without causing environmental pollution. These wastes can be effectively disposed by manufacturing useful by products from them.

Orange peel oil has been chosen for extraction because it provides a great potential for further commercial form. Peel of citrus fruit has numerous glands that contain oil that is typically recovered as major by product. Each citrus fruit has its own characteristic set of compounds that comprise the oil and that are responsible for its flavor and aroma to products such as carbonated drinks, ice-creams, cakes, air-fresheners and perfumes.(Braun & Cohen 2007). In this research, peel oranges waste were collected separately from the East

Coast Bottling factory which is located at Kawasan Perindustrian Semambu , 25350 Kuantan , Pahang.

Recently developed extraction methods like supercritical fluid extraction, microwave assisted extraction and Soxhlet method has been used for oil extraction. The basic parameters influencing the quality of an extract are the solvent used for extraction, the manufacturing process (extraction technology) used with the type of equipments employed. The use of appropriate extraction technology, plant material, manufacturing equipment, extraction method and solvent and the adherence to good manufacturing practices certainly help to produce a good quality extract. In this research, microwave assisted extraction of oil of orange peel has been chosen because it processes many advantages over conventional methods in terms of costing, yielding and time and reproducing better natural aroma of orange essential oil.

1.2 PROBLEM STATEMENT

The orange peels if treated as waste materials, may create environmental problems for local communities since the presence of biomaterials in orange peel . Every ton of food waste means 4.5 ton of CO₂ emissions. (Kesterson, J. W. and R. J. Braddock. 1976). There is a great need for development of new and environmental friendly design processing techniques .could be turned into an asset, if potentially marketable bioproducts such as pectin could be extracted from the peels. The peels, after extraction, could be sold as a high protein stock

feed in dry form, increasing the potential return for the orange juice industry and reducing the pollution load to the environment.

Thus, the demand of extraction of orange oil is very high compared to other fruit oil because orange oil will provide a great potential for further commercial use. Extraction using conventional method or innovative extraction technique may either cause degradation of the targeted compounds due to high temperature and long extraction time. So Microwave Assisted Solvent Extraction is suggested to be used to extract oil from orange peels in order to lower the operating & investment cost and to increase the profit as it allows more complete extraction at lower temperature.

1.3 RESEARCH OBJECTIVES

- 1.3.1 To determine the optimum time, temperature and power to extract orange essential oil for the highest yield and quality by using Microwave Assisted Solvent Extraction.
- 1.3.2 To apply as natural soap and air freshener after extracting the main component of essential oil

1.4 SCOPE OF STUDY

The scope of study is to solve the problem of disposal orange peels as waste materials which will cause to the environmental problems. In addition, we can analyze the characterization of this Microwave Assisted Extraction method in the yield. Thus we can evaluate the effect of different temperature, time

and power on extracted the oil from orange peels. Thus, the components in essential oil in peels from orange are analyzed by using Gas chromatography mass spectrometry (GC-MS). Application of the main components of orange peels (limonene) as non-toxic, environmentally friendly cleaner product .

1.5 SIGNIFICANCE OF STUDY

Effective solid waste management is one of the most essential element for an industry to achieve a sustainable development. Improper treatment of waste will affect peoples' health and the environment. For the citrus processing industry the disposal of fresh peels has become a major concern for many years Orange peels are the major solid by-product of the overall process. Orange peels are the major source of orange peel oil and these oils can be used for various industrial application. In order to produce good quality of extracts or essential oil from the orange peel waste, the most appropriate extraction technology must be applied.

There are various types of methods for extraction. There are many researches which have been carried out in the field of orange peel oil extracton. Orange peel oil extraction methods are still being researched. The goal of such researches is to discover methods to extract a greater percentage of peels oil than the current methods. Microwave Assisted Solvent Extraction method is considered as one of the most effective extraction technique would yield greater quantities of oil than any other conventional methods.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Citrus fruits belong to six genera (*Fortunella*, *Eremocitrus*, *Clymendis*, *Poncirus*, *Microcitrus* and *Citrus*) which are native to the tropical and subtropical regions of Asia, but the major commercial fruits belong to genus *Citrus*. The genus *Citrus* includes several important fruits such as oranges, mandarins, lime, lemons and grape fruits. The essential oils are present in fruit flavonoids in great quantities. The *Citrus* essential oils are a mixture of volatile compounds and mainly consist of monoterpene hydrocarbons (Sawamura et al 2004). *Citrus* oils are mixtures of over a hundred compounds that can be approximated into three fractions: terpene hydrocarbons, oxygenated compounds and nonvolatile compounds. The terpene fraction can constitute from 50 to more than 95% of the oil. However, it makes little contribution to the flavor and fragrance of the oil. Oranges are now commonly enjoyed by many and used for other purposes besides general consumption. It can be made into juice or incorporated into food products. It is a fruit with significant history, and oranges will remain a popular choice among consumers as a source of vitamin and to aid in wellbeing maintenance.

There are two modern species of orange, *Citrus aurantium* (the bitter or Seville orange) and *Citrus sinensis* (the sweet orange). Their wild ancestors originated in China and India, but these days they are cultivated mainly in Brazil and the USA, with contributions from Israel, Spain, South Africa, Australia and Malta. Interestingly, the early Chinese valued them for their peel rather than their pith. Perfume and flavouring were harvested in preference to the edible flesh, and orange oil is still highly valued among aromatherapists, perfumiers and herbalists. The current world production of oranges is around 50 million tons annually.

The remaining orange peels are around 45% of the total bulk. Consequently, significant amounts of orange peels are available as a by-product. The orange peels if treated as waste materials, may create environmental problems, particularly water pollution, since the presence of biomaterials in orange peels such as peel oil, pectin, as well as sugar, stimulate aerobic bacteria to decompose the biodegradable organic matters into products such as carbon dioxide, sulfates and phosphates in water. This problem could be turned into an asset, if potentially composition such as limonene could be extracted from orange peels which can be applied as natural products.

2.2 IMPORTANCE OF ORANGE OIL

Orange oil is a pure, essential oil produced by glands which found in the rinds of orange fruit and is well-known for its powerful antiseptic and solvent properties. Orange oil is considered to be relatively safe, very effective and

environmentally friendly. Essential oil of Orange (*Citrus sinensis*) is fun cheery, sweet, warm, sensuous, radiant and alive. The liquid which comes in the packet of orange flavored soft drink concentrates is sometimes this oil. The main components of this oil are Alpha Pinene, Citronellal, Geranial, Sabinene, Myrcene, Limonene, Linalool and Neral. Essential oil of orange has a wide variety of domestic, industrial and medicinal uses. Domestically, it is used to add orange flavor to beverages, desserts and sweetmeats. Industrially, it is used in soaps, body lotions, creams, anti mark and wrinkle lifting applications, concentrates for soft beverages, room fresheners, sprays, deodorants, biscuits, chocolates, confectionary and bakery items and many such.

In addition, orange oil restores balance to dry or oily skin. It maintains healthy, youthful skin by promoting the production of collagen. It reduces puffiness and discourages dry or wrinkled skin. Orange oil stimulates circulation to the skin. It also clear blemishes and improves acne-prone skin. It tends to decrease perspiration thus assisting the release of lymphatic fluids and help releases tissue swelling and fluid ventilation. It improves cellulite, which is sometimes called orange-peel skin.

Besides, orange oil can upset stomach, especially if nerve or stress related and can aid in digestion and restore appetite. It regulates bowels and relieves diarrhea and constipation. Orange oil encourages elimination of wastes and promotes urination, making it helpful in treating obesity, fluid retention and premenstrual syndrome (PMS). It regulates a body temperature and either cools a fever or warms a chilli. Orange oil soothes inflammation from psoriasis as well as eczema and other types of dermatitis.

Furthermore, orange oil also used in medical fields. Orange oil relieves the discomfort of bronchitis and the flu. It aids in absorption of vitamin C, boasts immunity, help prevent colds and flu, and relieves some of the symptoms associated with chronic fatigue syndrome. Orange oil heals mouth ulcers and gingivitis. It also settles painful muscles and joints. The Chinese treat anorexia nervosa, colds , coughs and malignant breast sores with dried orange peels.

Hence, orange oil is an excellent additive to synthetic furniture and wood care products. It is gentle yet it powerfully protects wood from insect damage and easily cleans fingerprints and grime off of the furniture without damaging it. For penetration beneath the wood surface and as protective against insect damage, it is mixed with linseed oil. Nothing is more gentle or protective for wooden furniture and instruments than these 3 essential oils which is orange, Swisspine and cypress mixed in jojoba oil.

2.3 SOLVENT EXTRACTION TECHNIQUE

It is the technique of removing one constituents from a solid by means of a liquid solvent also called leaching. The process may be employed either for the production of a concentrated solution of a valuable solid material or in order to free an insoluble solid, such as pigment from a soluble material with which it is contaminated. In this process, a chemical solvent such as n hexane is used to saturate the crushed seed and pull out the oils. After completion of the extraction process the solvent is condensed and reclaimed. There are many factors influencing the rate of extraction like particle size influences the extraction rate

in a number of ways. The smaller the size the greater is the interfacial area between the solid and liquid, and therefore the higher is the rate of the transfer of material. The liquid chosen should be a good selective solvent and its viscosity should be sufficiently low for it to circulate freely.

Temperature also affects the extraction rate, in most cases, the solubility of the material which is being extracted will increase with temperature. Agitation of the solvent also affects, it increases the eddy diffusion and therefore increases the transfer of material from the surface of the particles.

2.4 MICROWAVE-ASSISTED SOLVENT EXTRACTION (MASE)

Microwave-assisted solvent extraction (MASE), also called microwave extraction, is a new extraction technique, which combines microwave and traditional solvent extraction. Study shows that microwave-assisted extraction has many advantages, such as shorter time, less solvent, higher extraction rate, better products with lower cost. The apparatus of microwave-assisted extraction is simpler and cheaper and can be used to more materials with less limit of the polarity of extractant.

The applications of microwave technique in microwave-assisted solvent extraction as an alternative to the conventional solvent extraction have been introduced. This method was used as a new method for extraction of artemisinin from *Artemisia annua* L (Hao *et al.*, 2002). Recently, the microwave-assisted solvent extraction was used to extract thymol from seeds of *Trachyspermum ammi* (TA) (Gujar *et al.*, 2009) and oil from olive cake using hexane as solvent (Armani and Kadi, 2010).

The conventional solvent extraction (CSE) method that used to extract oil from vegetal materials is not efficient due to the long extraction time and solvent consumption. The microwave-assisted solvent extraction (MASE) system consisted of a domestic microwave oven, a reactor and a stirrer.

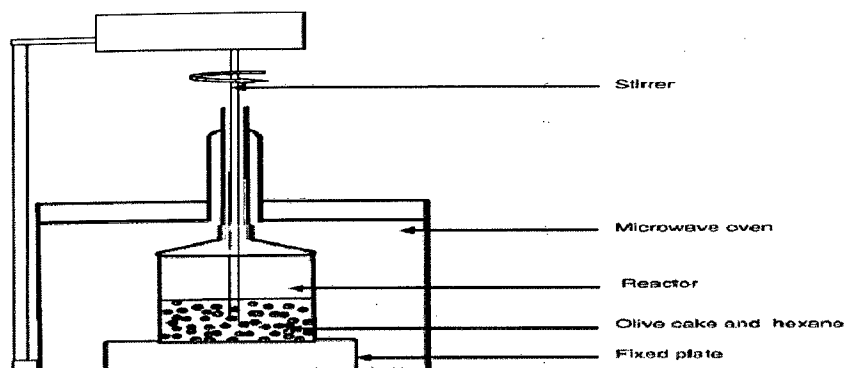


Figure 2.1: Schematic diagram of microwave-assisted extraction system (MASE) (*Armani and Kadi, 2010*).

The conventional solvent extraction system was the same as that used in the MASE system but a thermostated bath was used for the immersion of the reactor to control the temperature.

By applying microwave technique in the microwave-assisted extraction system, there was significance difference in the extraction time. The conventional solvent extraction needs a longer extraction time and thus a low efficiency. The enhanced extraction in microwave-assisted solvent extraction is due to the internal heating of the in situ water within the sample and cause the temperature rise rapidly and eventually accelerates cell rupture to release constituents.