UNIVERSITI MALAYSIA PAHANG

BORANG PENGESAHAN STATUS TESIS

JUDUL : **EXTRACTION OF LEMON OIL FROM *Citrus limon* PEELS VIA ULTRASONIC ASSISTED EXTRACTION;**

SESI PENGAJIAN: **2007/2008**

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Extraction of Lemon Oil from *Citrus limon* Peel via Ultrasonic assisted Extraction

DHANAKUMAR A/L SUPPIAH

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

University Malaysia Pahang

MAY 2008

I declare that this thesis entitled "Extraction of Lemon Oil from *Citrus limonium* Peel v*ia* Ultrasonic assisted Extraction" 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 of Candidate	: Dhanakumar a/l Suppiah
Date	: 16 MAY 2008

DEDICATION

I dedicated this research to my self

ACKNOWLEDGEMENT

‘To take action without thinking is something a stupid does, to take action after thousand times thinking is something a scientist does’. This word resembles what I’ve done for the past one year in completing this research. In completing this research, I have gained many in exchange of time,energy and money that I had.

They say you cannot achieve any thing alone in life, same goes with me. I get many helps from friends and lectures. They helped me in various kind of ways .I would like to take this opportunity to pour my gratitude’s to each of them .First and foremost I would like to thank my family for their help and for being at my site every time. Secondly, I would like to thank my supervisor for teaching me all the things about plant extraction and also I would like to say thanks Io other lectures for being very nice to me.

It is honored to have many loyal good friends that always been very helpful. I thanking them for the all the things they had done for me.

ABSTRACT

Ultrasound-assisted extraction was evaluated as a simpler and more effective alternative to conventional extraction methods for the isolation of lemon oil from peels of *Citrus limon*. The samples were extracted with different solvents, under indirect sonication in an ultrasound cleaning bath. The ultrasonic extraction was not only more efficient but also convenient for the recovery and purification of the active ingredients of plant materials. In addition, the sonication-assisted extraction can be carried out at lower temperatures which are favorable for the thermally unstable compounds. It is found that the optimum yield was achieved by diethyl ether with extraction time of 30 minutes compare to dichloromethane and n-hexane. Oil extracted with n-hexane has highest quality as the amount of limonene in oil high.

ABSTRAK

Pengekstrakan berbantu gelombang bunyi ultra dinilai sebagai suatu kaedah alternatif yang lebih mudah dan berkesan bebanding kaedah pengekstrakan tradisional yang lain bagi pengeluaran pati minyak dari kulit *Citrus limonium.* Sampel diekstrak dengan mengunakan bahan kimia pelarut yang berlainan melalui kaedah pengekstrakan bebantu gelombang bunyi ultra. Kaedah in bukan sahaja berkesan ,malah amat mudah bagi penemuan dan penulenan sebatian aktif dalam tumbuhan. Malah,kaedah pengekstrakan berkenaan boleh dilakukan pada suhu rendah yang amat bersesuaian bagi komponen bahan yang tidak mempunyai kestabilan terhadap haba. Adalah diketahui bahawa hasil maksimum dicapai bagi minyak yang diekstrak dengan mengunakan pelarut diethyl ether dalam masa 30 minit berbanding pelarut dichloromethana dan n-heksana .Minyak yang diekstrak menggunakan n-heksana mempunyai kualiti yang tertinggi kerana kandungan limonene dalam minyak adalah yang tertinggi .

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LIST OF ABBREVIATIONS AND SYMBOLS

cm	Centimeter
mmHg	Millimeter Mercury
µL	Micro liter
g	Gram
°C	Degree Celsius
%	Percentage
GC	Gas Chromatography
MS	Mass Spectrometry
α	Alpha
β	Beta

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Ultrasonic extraction is a new technology for extraction of plant material that can be carried out at lower temperatures, avoiding thermal damage to extracts and loss of volatile components in boiling.

The improvement of solvent extraction from plant material by ultrasound is due mainly to the mechanical effects of acoustic cavitations, which enhances both solvent penetration into the plant material and the intracellular product release by disrupting the cell walls.

The extraction of organic compounds from various plant materials can be significantly improved with the aid of intense ultrasound, achieving higher product yields at reduced processing time and solvent consumption.

More recently, application of ultrasonic technology in food processing attracted widely attentions Comparative investigation of the influence of classical and ultrasonic techniques on the yield and the structural features of extracts from wheat straw and the root of valerian have shown a higher yield and stability of functional properties of lignin and water-soluble polysaccharides using the latter method (Sun *et al.*, 2002). Ultrasonic extraction carnosic acid from *Rosmarinus officinalis* using ethanol was effective in producing a greater yield and shortening of extraction time (Albu and Mason, 2004).

Sun and Tomkinson reported a procedure for ultrasonic extraction of hemicelluloses from wheat straw,

Romdhane and Gourdou isolated pyrethrines from pyrethrum flowers and oil from woad seeds using ultrasound. The optimization of ultrasound variables according to a specific plant matrix is also of importance for achieving high extraction yield.

1.2 Problem Statement

Citrus fruit holds a unique place in plant kingdom and occupies a resulting solitary position in the human diet and playing an important role in food processing. Citrus peels represent a potential material for pharmaceutical and food industry since they contain significant flavonoids that are bioactive compounds with health-related properties. They have several hydroxyls in different position of rings, where there is strong chemical activity. Such components as antioxidants in various biological systems can display anti-allergic and anti-inflammatory activity

The common commercial methods to produce the oils from lemon oil are mostly based on the correct choice of solvents and the use of heat and agitation to increase the solubility of materials and the rate of mass transfer. Essential oils derived from steam distillation of lemon fruits and also from various other method are widely in use as ointments, bathing oils or inhaling drugs for curing a wide range of skin and muscle-disorders of infectious, rheumatic or neuralgic origin Those oils comprise various amounts of monoterpenes such as pinene, camphene , limonene, neral and myrcene as major components.

Usually, the traditional techniques require long extraction hours and have low efficiency. Moreover, many natural products are thermally unstable and may degrade during thermal extraction.

These earlier works have proved that ultrasonic extraction is a potential technology in food processing and pharmaceutical industry. Ultrasonic technique for lemon oil extraction is seems to be an attractive process.

In this work, the influence of solvents and time on the extraction rate of lemon peel (*Citrus limon*) via USE is studied.

1.3 Research Objective

The research objective is to study the effect of solvent and time in extraction of lemon peel oil from *Citrus limon* via Ultrasound extraction (USE).

1.4 Scope of Research

There are several scopes on this research which is:

(i)To study the effect of sonication time of extraction on the yields of lemon oil via ultrasonic extraction

(ii)To study the effect of low boiling point solvent (n-hexane, dichloromethane and diethyl ether) on yield of oil

(iii)To analyze the constituents in lemon oil by using GC-MS.

(iv)To determine the quality of oil produced by comparing the percent area of limonene. peak in chromathogram

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The lemon (*Citrus limon*) is a hybrid in cultivated wild plants. It is the common name for the reproductive tissue surrounding the seed of the angiosperm lemon tree. The lemon is used for culinary and non culinary purposes throughout the world. The fruit is used primarily for its juice, though the pulp and rind (zest) are also used, primarily in cooking and baking.

2.1.1 Botanic of Lemon

Table 2.1: Botany details of lemon

Scientific name	*Citrus limonium*
Common name	Lemon
Family	*Rutacea*
Origin	China ,Italy

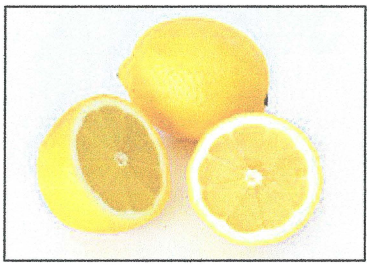

Figure: 2.1: Lemon fruits

2.2 Lemon Essential Oil

Lemon essential oil possesses strong germicidal disinfectant properties, and it is a pleasant addition to formulations intended to purify the air. The astringency of lemon makes it useful for combating oily skin and hair. It can also help with bronchial problems and asthma, and it stimulates concentration.

Lemon essential oil has a refreshing scent that is thought to stimulate the liver and has a gentle, calming effect. Lemon oil is thought to promote clarity of mind and purpose, as well as generally increasing one's well-being and physical energy.

Lemon essential oil is a powerful antiseptic. Not only has it been used to clean contaminated surfaces, such as a butcher's cutting block, but it has a powerful antibacterial and antiviral effect attributable to its limonene content.

Lemon essential oil is use to eliminate many types of bacteria, and may be the most effective oil for disinfecting a room using a diffuser.

It is often used in disinfecting blends with other antibacterial oils what makes lemon oil particularly pleasing for this use is that its antiseptic properties are present with a lovely aroma. Lemon is a great modifier for medicinal-smelling oils like tea tree and eucalyptus. Lemon works synergistically on a therapeutic, aesthetic and emotional level.

It also contains antifungal compounds. Some people have even used lemon to disinfect questionable drinking water. Because of its vitamin content, lemon oil offers strong support to the immune system.

Rather than exhibit an extreme acidic quality, lemon essential oil works as an excellent stabilizer of the body's acidity-alkalinity content. And its astringent qualities make lemon oil useful in the home for many conditions, including insect bites, fever and skin conditions.

2.2.1 Medicinal Uses

The vaporized essence of lemon can kill *meningococcus* bacteria in 15 minutes, typhoid bacilli in just one hour, Staphylococcus *aureus* in two hours, and *Pneumococcus* bacteria within three hours. A mere 0.2 percent solution of lemon oil can eliminate diphtheria bacteria in 20 minutes and completely inactivate tuberculosis bacteria.

Lemon oil is also beneficial for anxiety, blood pressure, digestive problems, sore throats, and respiratory infections. It helps improves memory, strengthens nails, promotes a sense of well-being, and cleans the skin.

There is no known virus or bacteria that can live in the presence of any therapeutic-grade essential oil. With antibiotics, viruses can mutate and develop an immunity but not with essential oils. Lemon oil is even diffused extensively throughout hospitals in Europe and England. Recently, a unique blend of essential oils was tested at Weber State University for its potent antimicrobial properties, and amazingly, it was found to have a 99.96 percent kill rate against airborne bacteria. (www.refluxremedy .com)

2.3 Major Compounds

The major compound of lemon oil is, alpha pinene, beta pinene, camphene, limonene, alpha-terpinene, citral, sabinene, myrcene, linalool, b-bisabolene, trans-a-bergamotene, nerol and neral

2.4 Extraction Procedures

To obtain extracts from vegetal materials several methods are available:

1. Distillation:

(a) Direct essential oil distillation;

(b) Water steam distillation;

(c) Water and steam distillation.

2. Solvent extraction:

(a) Solvent extraction (percolation)

(b) Maceration with solvent

(c) Boiling with water (infusion)

(d) Extraction with cold fat (effleurage)

(e) Extraction with hot fat.

3. Cold compression, which is the usual method for the natural oil industry

4. Non-conventional extraction techniques:

(a) Supercritical fluid extraction

(b) Vertical (turbo) extraction

(c) Extraction by electrical energy

(d) Ultrasonically assisted extraction

Not all of the classical extraction processes are suitable for ultrasonic enhancement. The main procedures leading to bioactive products from plants and its constituents (seeds, flowers and leaves.) are percolation, maceration, water steam distillation, Soxhlet extraction, infusion and boiling. The water steam distillation to produce essential oil for example is not amenable to ultrasonic enhancement but extraction with light solvent (petroleum ether) or with water or water-alcohol extracts (maceration) are possible (Vinatoru *et al.,* 1997). These methods lead to the types of extract suitable for cosmetics, pharmaceuticals as well as for food industry.

2.5 Ultrasonic Extraction

Ultrasound is probably the most simple and most versatile method for the disruption of cells and for the production of extracts. Ultrasound is efficient, safe and reliable.

Today, thermal treatment is the most common processing method for food extraction or microbial inactivation that leads to longer shelf-life (preservation). Because of the exposure to high temperature this method has often disadvantages for many food products. Thermal treatment can cause undesirable alterations of sensory attributes, example texture, flavour, colour, smell, and nutritional qualities, example vitamins and proteins. Other use of ultrasonis includes

(i) Ultrasonic improvement of oil extraction from oil seeds

(ii) Cell membrane permeabilization of fruits, such as grapes, plums, mango

(iii) Ultrasonic processing of fruit juices purees sauces, and dairy products

(iv) Improves stability of dispersions, such as in orange juice

Unlike other non-thermal processes, such as high hydrostatic pressure (HP), compressed carbon dioxide (cCO2) and supercritical carbon dioxide (ScCO2) and high electric field pulses (HELP), ultrasound can be easily tested in lab or bench-top scale - generating reproducible results for scale-up. The intensity and the cavitations characteristics can be easily adapted to the specific extraction process to target specific objectives. Amplitude and pressure can be varied in a wide range, such as to identify the most energy efficient extraction setup (Vinotoru *et al.,* 1997)

When high frequency ultrasound is employed, the extraction yield did not increase significantly however the degradation of the herb constituents was diminished. In the case of low frequency sonication degradation becomes more important, especially when alkaloids are being extracted. This effect could be employed as a tool to help in the extraction of medicinal compounds by using lower frequencies to assist in the degradation of toxic alkaloids during the process.

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. An example showing how ultrasound can help solvent extraction of essential oils from dill seeds is given in Table 2 .1

2.6 Sonication-assisted Extraction

2.6.1 Principles and Mechanisms

Two general designs of ultrasound-assisted extractors are ultrasonic baths or closed extractors fitted with an ultrasonic horn transducer. The mechanical effects of ultrasound induce a greater penetration of solvent into cellular materials and improve mass transfer. Ultrasound in extraction can also disrupt biological cell walls, facilitating the release of contents. Therefore, efficient cell disruption and effective mass transfer are cited as two major factors leading to the enhancement of extraction with ultrasonic power (Paniwnyk and Lorimer 1996). Scanning electron micrographs (SEM) have provided evidence of the mechanical effects of ultrasound, mainly shown by the destruction of cell walls and release of cell contents. In contrast to conventional extractions, plant extracts diffuse across cell walls due to ultrasound, causing cell rupture over a shorter period (Vinatoru *et al.*, 1999).

2.6.2 Practical Issues For Sonication-assisted Extraction

It is necessary to take into account plant characteristics such as moisture content and particle size, and solvent used for the extraction in order to obtain an efficient and effective ultrasound-assisted extraction. Furthermore, many factors govern the action of ultrasound including frequency, pressure, and temperature and sonication time.

2.6.3 Effects of Ultrasound Characteristics

Ultrasound frequency has great effects on extraction yield and kinetics. However, the effects of ultrasound on extraction yield and kinetics differ depending on the nature of the plant material to be extracted. A small change in frequency can increase the yield of extract about 32% for ultrasound-assisted solid-hexane extraction of pyrethrines from *pyrethrum* flowers. However, ultrasound has weak effects on both yield and kinetics for the extraction of oil from *woad* seeds (Romdhane and Gourdon, 2002).

The ultrasonic wave distribution inside an extractor is also a key parameter in the design of an ultrasonic extractor. The maximum ultrasound power is observed in the vicinity of the radiating surface of the ultrasonic horn. Ultrasonic intensity decreases rather abruptly as the distance from the radiating surface increases. Also, ultrasound intensity is attenuated with the increase of the presence of solid particles (Romdhane, Gourdon, and Casamatta 1995).

2.6.4 Operating Conditions

The use of ultrasound allows changes in the processing condition such as a decrease of temperature and pressure from those used in extractions without ultrasound (Wu *et al.,* 2001). For solid-hexane extraction of pyrethrines from *pyrethrum* flowers without ultrasound, extraction yield increases with the extraction temperature and maximum yield is achieved at 66 °C. With ultrasound, the effect of temperature in the range of 40 to 66 °C on the yield is negligible, such that optimal extraction occurs across the range of temperature from 40 to 66 °C. Therefore, use of ultrasound-assisted extraction is advisable for thermo labile compounds, which may be altered under Soxhlet operating conditions due to the high extraction temperature (Romdhane and Gourdon, 2002). However, it should be noted that since ultrasound generates heat, it is important to accurately control the extraction temperature (Toma *et al.,* 1997). The sonication time should also be considered carefully as excess of sonication can damage the quality of extracts.

2.6.5 Advantages and Disadvantages of Sonication-assisted Extraction

Ultrasound-assisted extraction is an inexpensive, simple and efficient alternative to conventional extraction techniques. The main benefits of use of ultrasound in solid–liquid extraction include the increase of extraction yield and faster kinetics. Ultrasound can also reduce the operating temperature allowing the extraction of thermo labile compounds. Compared with other novel extraction techniques such as microwave-assisted extraction, the ultrasound apparatus is cheaper and its operation is easier. Furthermore, the ultrasound-assisted extraction, like Soxhlet extraction, can be used with any solvent for extracting a wide variety of natural compounds.

However, the effects of ultrasound on extraction yield and kinetics may be linked to the nature of the plant matrix. The presence of a dispersed phase contributes to the ultrasound wave attenuation and the active part of ultrasound inside the extractor is restricted to a zone located in the vicinity of the ultrasonic emitter. Therefore, those two factors must be considered carefully in the design of ultrasound-assisted extractors.

2.6.6 Potential Applications of Sonication-assisted Extraction

Ultrasound-assisted extraction has been used to extract nutraceuticals from plants such as essential oils and lipids dietary supplements (Wu *et al.,* 2001).Ultrasound can increase extraction yield. Sharma and Gupta (2004) found that ultrasonication was a critical pretreatment to obtain high yields of oils from almond, apricot and rice bran. The yield of oil extracted from soybeans also increased significantly using ultrasound (Li *et al.*, 2004). For ultrasound-assisted extraction of saponin from ginseng, the observed total yield and saponin yield increased by 15% and 30% (Hui *et al.*, 1994).

Ultrasound can increase extraction kinetics and even improve the quality of extracts. Cravotto *et al.* (2004) found that rice bran oil extraction can be efficiently performed in 30 min under high-intensity ultrasound either using hexane or a basic aqueous solution. Extraction rates of carvone and limonene by ultrasound-assisted extraction with hexane were 1.3–2 times more rapid than those by the conventional extraction depending on temperature (Chemat *et al.*, 2004). Furthermore, the yield and quality of carvone obtained by the ultrasound-assisted extraction were better than those by a conventional method. The ultrasound was also applied to the cartridge of a Soxhlet extraction for the extraction of total fat from oleaginous seeds such as sunflower, rape and soybean seeds. The use of ultrasound reduced the extraction at least to half of the time needed by conventional extraction methods without any change in the composition of extracted oils (Luque-Garcia and Luque de Castro, 2004). Wu *et al.* (2001) found the ultrasound-assisted extraction of ginseng saponins occurred about three times faster than traditional Soxhlet extraction.

Ultrasound-assisted extraction was considered as an efficient method for extracting bioactive compounds from *Solvia officinalis* (Salisova *et al.*, 1997) and *Hibiscus tiliaceus L.* flowers (Melecchi *et al.*, 2002), antioxidants from *Rosmarinus officinalis* (Albu *et al.*, 2004), and steroids and triterpenoids from *Chresta spp.* (Schinor, *et al.,* 2004). The use of ultrasound as an adjunct to conventional extraction provides qualitatively acceptable tocols from *amaranthus caudatus* seeds but much more quickly, more economically and using equipment commonly available (Bruni *et al.*, 2002).

2.7 Laboratory and Large Scale Ultrasonic Extraction

Performing ultrasonic assisted extraction is not difficult on a laboratory scale can be done by using a simple cleaning bath (figure. 2.2 and figure. 2.3). Using such equipment it is possible to obtain good extraction yields by direct or indirect extraction .In both cases it is preferable to use a mechanical stirrer and to cool the extraction mixture since the absorption of ultrasonic energy can increase temperature. By indirect sonication, only small amounts of vegetal material can be extracted, whereas using the direct procedure, large amounts of vegetal material can be employed.

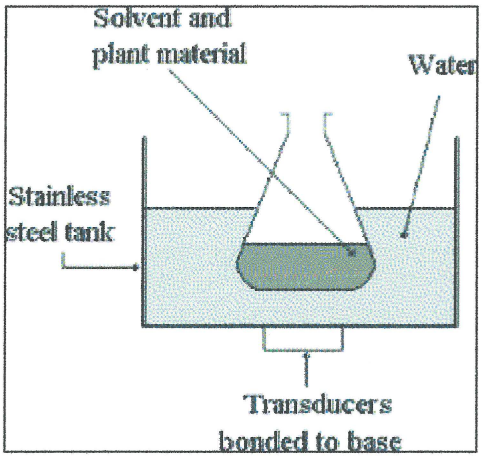

Figure2.2: Experimental setup for indirect extraction using a cleaning bath.

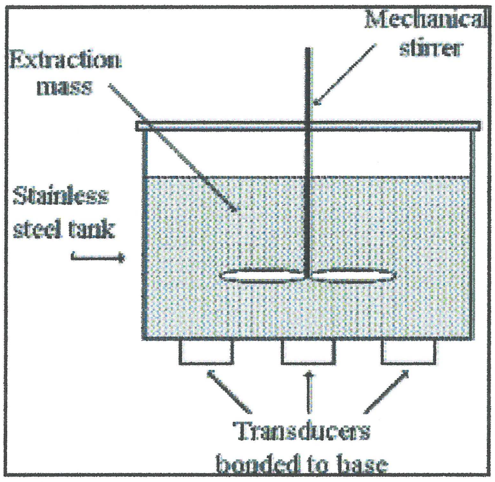

Figure2.3: Experimental setup for direct extraction using a cleaning bath.

CHAPTER 3

METHODOLOGY

3.1 Raw Material

Lemon fruit is purchased from local market; the outer yellow part is cut into small pieces (1cm in length per piece) is air-dried in the shade before being introduced into a flask added with solvents.

3.2 Solvents

One liter of each low boiling points solvents; n-hexane, diethyl ether and dichloromethane, were used for the extraction of lemon peel.

3.3 Ultrasound Assisted Extraction

Plant material and solvent were placed into an Erlenmeyer flask (1000ml) fitted with a condenser. The flask was immersed into the cleaning bath, at four centimeters distance from the bottom of the tank, and sonicated.

The temperature was kept constant (25°C) by a cooling coil immersed into the bath. Indirect sonication (fixed frequency, 35 kHz) was performed in a closed reactor, fitted with condenser.

Entire raw material (150 g) were extracted with 300 ml solvents via indirect sonication for, (15 minute, 30 minute and 45minute) .The marc was removed from the solvent by filtering, through Whatman filter paper in a Büchner funnel. Oil was recovered by drying down at boiling point of each solvent, using a rotary vacuum evaporator (RVE) with low vacuum pressure of 300mmHg.

The yields were recorded after filtration and evaporation of solvent. Main components of aroma compounds were successfully removed and collected in 2 mL solvent.

For control experiment, lemon peel is macerated for 60 minutes at room temperature with each solvent using same amount of raw material

3.4 Gas Chromatography and Mass Spectrometry (GC-MS)

The thick oil was diluted with solvent n-hexane and prepared in ratio of 1:10 in sample bottle.

The extracts were examined by GC-MS, using a Hewlett-Packard 5890 series II gas chromatograph equipped with Hewlett-Packard 5972 mass detector with electron impact ion source (Hewlett-Packard, Rockville).

The system was assisted with the Hewlett-Packard MS Chemstation software, version B.02.05. The column used was a HP-5 M.S. (Crosslinked 5% pH Me Silicone, 30 m × 0.25 mm, 0.25 μm film thickness). Helium was used as carrier gas at a constant flow of 0.84 ml min^{-1}. In all runs, 2 μl of sample were injected in split mode with a split ratio of 1:14 at 280 °C.

The oven temperature was programmed from 35 °C (1 min hold) to 270 °C (10 min hold) at 4 °C min^{-1}. The MS interface was heated at 320 °C. The compounds were identified by their mass spectra (computerized comparison with reference spectra in the MS Chemstation library) and retention time in the capillary column.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

The data were obtained from the sample for each experiment conducted with different conditions. Each experiment with different conditions will yield different production of essential oil. It will also differ in the chemical constituents for each sample. The chemical constituents of each sample were identified by their mass spectra and compared with reference spectra in the MS Chemstation library and also from retention time in the capillary column

4.2 Effects of Solvent and Sonication Time

Table 4.1 show the yields of oil are dependent on different solvents. Average result is taken account from two trials to reduce the percentage error in readings

Table 4.1: Average result for quantity analysis of lemon oil

Solvent	Mass of sample (g)	Extraction Time (minute)	Weight of Oil (g)	Volume of Oil (ml)	Yield (wt %)
Dichloromethane	150	15	1.79	1.8	1.12
	150	30	2.25	2.6	1.5
	150	45	1.83	2.0	1.21
Diethyl ether	150	15	5.85	6.0	3.9
	150	30	12.86	13.	8.57
	150	45	12.06	12.4	8.04
N-hexane	150	15	3.12	3.2	2.08
	150	30	5.0	5	3.33
	150	45	4.38	5	2.91

For diethyl ether (sonicated for 15 minutes) it was observed that solvents turns yellow and layer of oil could be seeing floating on top of solvents .For sample sonicated for 30 minutes, bubbles appears and layer of oil turns a pale yellow. This shows rapid release of oil from sample .For time 45 minutes, sample turns brown and dark layer of oil appears

For n-hexane, all the sample solvents turn yellowish and only sample sonicated for 30 minutes and 45 minutes shows slight layer of oil on top of solvents layer.

For dichloromethane (sonicated for 15minutes), solvents turns yellow and layer of oil could be seeing floating on top of solvent .Sonication for 30 minutes and 45 minutes has led to dryness of solvent and only some oil is left behind.

For diethyl ether the yield increased by 4.67% from at t=30 minutes compared to n-hexane which increased by 1.22% and dichloromethane by 0.48%

For sonication time of 45 minutes, oil extracted has reduced by 0.53% for diethyl ether, 0.42% for n-hexane and 0.39% for dichloromethane.

Diethyl ether appears to be the most effective extraction solvent under the same ultrasonic conditions and temperature followed by n-hexane and dichloromethane. The different yields of extracts might be caused by polarities of solvents.

Diethyl ether has high polarity and immiscible in water thus could extract most of hydrophobic component from lemon peel N-hexane is only suitable in extraction involving maceration for longer period and for this experiment ,n-hexane only show less quantity of oil as extraction time varies from 15 minutes to 45minutes.

This is also due to mechanism for the ultrasonic enhancement of extraction which depends to the increase of mass transfer and easier access of the solvent to the cell material of the peel. Although cavitational collapse, n-hexane does not produce high energies but it is expected that it would still produce some cell disruption together with a good penetration of the solvent into the cells, through the ultrasonic

For dichloromethane, the oil is obtained less as the affinity of solute to react with solvent is less than diethyl ether and it also because the solvents is tend to vaporize or dry out before the extraction is complete.

Extraction of oil with ultrasonic time of 30minutes from 100g of dill seed with n-hexane shows 3.40 g of yield (Table 2.2).In this experiment, the yield of lemon oil from (150 g of raw material) from n-hexane with same extraction conditions is 5.0g, shows that somehow n-hexane has produces good yield. in extraction from lemon peel

Lemon peels in the entire sample shows only a small amount of extraction in the first 15 minutes whereas the rest of the sample showed a rapid extraction within 30 minutes. This can be explained through the disruptions of the cell walls thus take some time (~30min) after which time the release of cell content is much more rapid. After 30 minutes the oil extracted has show slight reduction .as the longer extraction hour has made some of the oil vaporized. The optimum time for ultrasonic extraction is about 30min, ensuring nearly the maximum amount of oil in liquid extracts

High temperature is not beneficial for ultrasonic extraction because of evaporation of low boiling point solvent. So, 25°C is chosen as an optimal temperature in the extraction procedures this also could avoid thermal degradation of lemon oil properties. The results suggested the advantages of ultrasound-assisted extraction, which can achieve at lower temperature and can efficiently reduce extraction time.

In this experiment the ultrasonic frequencies are maintained at moderate level of 35 kHz .This level of frequency could also avoid disruption of plant molecule and heat that could be generated from high intensity

4.2.1 Control Procedure

In the control procedure, the mechanism is via normal diffusion through the cell walls. A process which require control experiments using classical extraction obtained by mixing the mixture of plant material and solvent were allowed to stand (maturate) for 60 minutes

Table 4.2: Maceration and ultrasonication

Sample	Weight of Sample (g)	Weight of oil(g)	Yield (wt %)
US 30minutes (Diethyl ether)	150	12.86	8.57
Maceration with diethyl ether (60minutes)	150	No oil obtained	-
US 30 minutes (n-hexane)	150	5.00	3.33
Maceration with n-hexane (60minutes)	150	No oil obtained	-
US 30 minutes (dichloromethane)	150	2.25	1.5
Maceration with dichloromethane(60 minutes)	150	No oil obtained	-

The conventional method shows no yield for 60 minutes of maceration compares to ultrasonication for 30 minutes. This is due to mechanical effects of ultrasound, mainly appearing on cell walls and by the destruction of cells, enhances the release of their contents, in contrast to conventional maceration which involve diffusion of plant extracts.

The ultrasonic extraction at 25°C is more effective than the classical maceration at room temperature, ensuring higher yields of oil at much shorter time, especially if an extracting solvent of higher polarity is used. The ultrasonic procedure thus seems to be a significant improvement when extraction time is taken into account

4.3 Quality Analysis of Lemon Oil

Table 4.3, 4.4 and 4.5 shows the data obtained from the qualitative analysis with the retentions time which represents chemical constituents that consist in the samples. The compounds were identified by their mass spectra (computerized comparison with reference spectra in the MS Chemstation library) and retention time in the capillary column.

Table 4.3: Major compound and quality of each constituent in oil extracted with diethyl ether (30minutes time of extraction)

Peak	MSChemstation library ID	Quality	Area %
5	Alpha pinene	96	0.36
10	**limonene**	**91**	**17.30**
3	camphene	96	0.02
5	Beta pinene	94	3.05

Table 4.4: Major compound and quality of each constituent in oil extracted with dichloromethane (30 minute time of extraction)

Peak	MS Chemstation library ID	Quality	Area %
2	Alpha pinene	96	0.28
9	**limonene**	**94**	**16.91**
4	Beta pinene	96	2.85
5	Beta-mycrene	95	1.00

Table 4.5: Major compound and quality of each constituent in oil extracted with n-hexane (30minute time of extraction)

Peak	MS Chemstation library ID	Quality	Area %
5	Alpha pinene	96	0.69
14	**limonene**	**94**	**17.93**
6	camphene	96	0.04
33	nerol	16	2.94
9	Beta pinene	96	3.51
10	mycrene	59	1.61
16	linalool	40	0.37

Lemon oil contains many constituents in its essential oil. Each chemical constituent from the lemon peel is extracted differently due to type of solvents.

Table shows constituents in lemon oil such as α-pinene, camphene, beta -pinene, and limonene. Comparison with each samples shows that oil extracted with n-hexane has highest quality as the percentage area of limonene which represent the amount of limonene in oil is large than other sample that is 17.93%.Limonene in oil extracted with diethyl ether has 17.30% area and dichloromethane has lowest quality with 16.91% area. The difference of quality is due to type of solvents which plays important role in extracting chemical compound from plant .The result shows that n-hexane is efficient in term of oil quality.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The results from this study showed that type of solvent and sonication time in ultrasonic extraction was the most effective influence on the yield of the extracts of solid -liquid extraction.

In this experiment, the most efficient ultrasonic parameters of extracting oil from lemon peel were determined as diethyl ether for extraction time of 30minutes. The best quality of oil is from oil extracted by n-hexane.

Two main conclusions can be drawn from the information presented in this paper:

(i)Ultrasound has been proven to assist solvent extraction by reducing time and temperature of extraction

(ii)Ultrasound has proved to be a powerful tool for the phytochemical extraction from plant.

5.2 Recommendation

The most widely used solvent to extract edible oils from plant sources is hexane. Hexane is available at low cost and is efficient in terms of oil and solvent recovery (Mustakas *et al.*, 1980) but the most appropriate solvent that is recommended in extraction of lemon peel (in terms of yield) at low boiling point is diethyl ether. In terms of oil quality, it is strongly recommended to use n-hexane.

The selection of parameter such as extraction temperature and time should carefully consider the evaporation of oil and the solubility of compounds extracted in order to avoid loss of solvent in high temperature and reduce costs. In experiment it is recommended to make sure to maintain the temperature of the ultrasonic water bath so that the volatile oils do not vaporize

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Appendix A

Table A1: Solvent Boiling Point Chart

Solvent	Boiling Point (ºC)	Solvent	Boiling Point (ºC)
Acetic Acid	118.0	Ethyl Acetate	77.1
Acetic Acid Anhydride	139.0	Ethyl Ether	34.6
Acetone	56.3	Ethylene Dichloride	83.5
Acetonitrile	81.6	Ethylene Glycol	197.5
Benzene	80.1	Heptane	98.4
iso-Butanol	107.7	n-Hexane	68.7
n-Butanol	117.7	Hydrochloric Acid	84.8
tert-Butanol	82.5	Methanol	64.7
Carbon Tetrachloride	76.5	Methylene Chloride	-96.7
Chlorobenzene	131.7	MTBE	55.2
Chloroform	61.2	Pentane	36.1
Cyclohexane	80.7	Petroleum Ether	35.0-60.0
Cyclopentane	49.3	iso-Propanol	82.3
Dichloromethane	39.8	n-Propanol	97.2
Diethyl Ether	34.6	Pyridine	115.3
Dimethyl Acetamide	166.1	Tetrahydrofuran	66.0
Dimethyl Formamide	153.0	Toluene	110.6
Dimethyl Sulfoxide	189.0	Trifluoroacetic Acid	71.8
Dioxane	101.0	Water	100.0
Ethanol	78.3	Xylene	140.0

(All boiling points at standard pressure)

Appendix B

Table B1: Result for quantity analysis

Solvent	**Mass of sample (g)**	**Extraction Time (minute)**	**Weight of Oil (g)**	**Volume of Oil (ml)**	**Yield (wt %)**
Dichloromethane	150	15	1.86	1.8	1.24
	150	30	2.3	2.4	1.533
	150	45	1.88	2.0	1.2
Diethyl ether	150	15	6.59	6.0	4.4
	150	30	12.7	13.	8.47
	150	45	11.56	12.4	7.69
N-hexane	150	15	3	3.2	2.0
	150	30	5.230	5	3.4866
	150	45	5.01	5	3.34

Solvent	**Mass of sample (g)**	**Extraction Time (minute)**	**Weight of Oil (g)**	**Volume of Oil (ml)**	**Yield (wt %)**
Dichloromethane	150	15	1.71	2.5	1.14
	150	30	2.51	2.6	1. 667
	150	45	1.78	2.1	1.2
Diethyl ether	150	15	5.03	5.20	3.353
	150	30	13.01	13.2	8.667
	150	45	12.6	12.8	8.4
N-hexane	150	15	3. 3	3.6	2.2
	150	30	4.8	5.0	3.2
	150	45	3.765	4.0	2.51

Appendix C

MS-Chemestation Library ID

Library Search Report

ta Path : D:\Data\LEMON OIL\
ta File : PSMP01.D
g On : 24 Mar 2008 13:16
erator : NORA
nple : PINE1
sc :
 Vial : 6 Sample Multiplier: 1

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

known Spectrum: Apex
egration Events: ChemStation Integrator - autoint1.e

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		Bicyclo[3.1.0]hexane, 4-methyl-1-(1-methylethyl)-, didehydro deriv.	15391	058037-87-9	93
		.alpha.-Phellandrene	15203	000099-83-2	91
		Bicyclo[3.1.0]hex-2-ene, 2-methyl-5-(1-methylethyl)-	15375	002867-05-2	91
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		1R-.alpha.-Pinene	15186	007785-70-8	97
		1R-.alpha.-Pinene	15186	007785-70-8	96
		1S-.alpha.-Pinene	15185	007785-26-4	96
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		Bicyclo[2.2.1]heptane, 2,2-dimethyl-3-methylene-, (1S)-	15387	005794-04-7	98
		Camphene	15160	000079-92-5	96
		Camphene	15161	000079-92-5	96
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		Bicyclo[3.1.0]hexane, 4-methylene-1-(1-methylethyl)-	15378	003387-41-5	91
		.beta.-Phellandrene	15200	000555-10-2	91
		Bicyclo[3.1.0]hexane, 4-methylene-1-(1-methylethyl)-	15379	003387-41-5	91
4.433	3.05	C:\Database\NIST05a.L			
		Bicyclo[3.1.1]heptane, 6,6-dimethyl-2-methylene-, (1S)-	15390	018172-67-3	97
		.beta.-Pinene	15171	000127-91-3	96
		.beta.-Pinene	15175	000127-91-3	94
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		.beta.-Myrcene	15177	000123-35-3	95
		Bicyclo[3.1.0]hex-2-ene, 4-methyl-1-(1-methylethyl)-	15374	028634-89-1	80
		Pyridine, 2-propyl-	9271	000622-39-9	59
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		Octanal	12031	000124-13-0	91
		Octanal	12028	000124-13-0	87
		Octanal	12030	000124-13-0	83
4.679	0.05	C:\Database\NIST05a.L			
		.alpha.-Phellandrene	15204	000099-83-2	91
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		.alpha.-Phellandrene	15202	000099-83-2	87
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		Bicyclo[4.1.0]hept-2-ene, 3,7,7-trimethyl-	15319	000554-61-0	97
		Bicyclo[4.1.0]hept-2-ene, 3,7,7-trimethyl-	15317	000554-61-0	96
		(+)-4-Carene	15169	029050-33-7	96
5.031	17.30	C:\Database\NIST05a.L			
		Limonene	15153	000138-86-3	91
		Limonene	15154	000138-86-3	91
		D-Limonene	15165	005989-27-5	90

Library Search Report

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		Octanal	12028	000124-13-0	87
		Octanal	12030	000124-13-0	83
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		.alpha.-Phellandrene	15203	000099-83-2	91
		.alpha.-Phellandrene	15202	000099-83-2	91
4.807	0.22	C:\Database\NIST05a.L			
		(+)-4-Carene	15169	029050-33-7	97
		1,3-Cyclohexadiene, 1-methyl-4-(1- methylethyl)-	15349	000099-86-5	95
		Bicyclo[4.1.0]hept-2-ene, 3,7,7-tr imethyl-	15319	000554-61-0	95
4.988	16.91	C:\Database\NIST05a.L			
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		Limonene	15154	000138-86-3	94
		D-Limonene	15165	005989-27-5	93
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		1,3,6-Octatriene, 3,7-dimethyl-, (Z)-	15281	003338-55-4	95
		1,3,7-Octatriene, 3,7-dimethyl-	15243	000502-99-8	94

Library Search Report

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		Bicyclo[3.1.0]hex-2-ene, 2-methyl-5-(1-methylethyl)-	15380	002867-05-2	91
		Bicyclo[3.1.0]hex-2-ene, 2-methyl-5-(1-methylethyl)-	15375	002867-05-2	91
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		1R-.alpha.-Pinene	15186	007785-70-8	96
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		Camphene	15161	000079-92-5	98
		Bicyclo[2.2.1]heptane, 2,2-dimethyl-3-methylene-, (1S)-	15387	005794-04-7	97
		Camphene	15160	000079-92-5	95
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		Bicyclo[3.1.0]hexane, 4-methylene-1-(1-methylethyl)-	15378	003387-41-5	91
		.beta.-Phellandrene	15198	000555-10-2	90
4.433	2.84	C:\Database\NIST05a.L			
		Bicyclo[3.1.1]heptane, 6,6-dimethyl-2-methylene-, (1S)-	15390	018172-67-3	97
		.beta.-Pinene	15171	000127-91-3	96
		.beta.-Pinene	15176	000127-91-3	94
4.497	1.10	C:\Database\NIST05a.L			
		.beta.-Myrcene	15177	000123-35-3	95
		Pyridine, 2-propyl-	9271	000622-39-9	59
		2-Pyridineethanamine	9505	002706-56-1	59
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		Octanal	12031	000124-13-0	91
		Octanal	12028	000124-13-0	91
		Octanal	12030	000124-13-0	83
4.679	0.05	C:\Database\NIST05a.L			
		.alpha.-Phellandrene	15202	000099-83-2	87
		.alpha.-Phellandrene	15203	000099-83-2	87
		Bicyclo[3.1.0]hex-2-ene, 2-methyl-5-(1-methylethyl)-	15380	002867-05-2	86
4.807	0.24	C:\Database\NIST05a.L			
		Cyclohexene, 1-methyl-4-(1-methylethylidene)-	15340	000586-62-9	97
		1,3-Cyclohexadiene, 1-methyl-4-(1-methylethyl)-	15349	000099-86-5	96
		Bicyclo[4.1.0]hept-2-ene, 3,7,7-trimethyl-	15317	000554-61-0	96
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		Limonene	15154	000138-86-3	94
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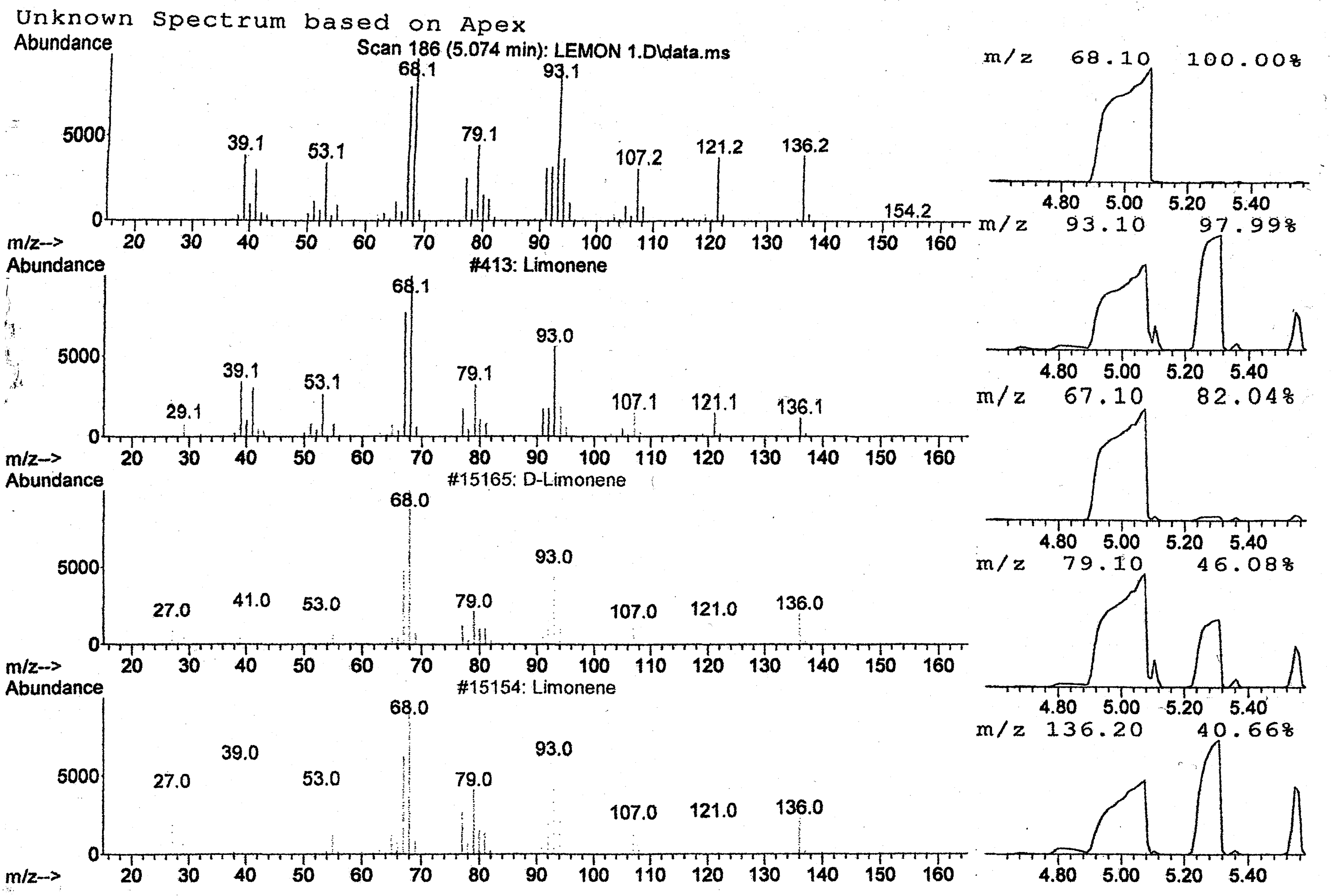
		D-Limonene	15162	005989-27-5	90

Library Search Report - ChemStation Integrator



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2 D-Limonene	15165	005989-27-5	93
3 Limonene	15154	000138-86-3	91
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2 Terpinyl acetate	234	000080-26-2	64
3 Linalyl butyrate	246	000078-36-4	40