# STUDIES ON MICROWAVE STEAM DISTILLATION PROCESS FOR EXTRACTION OF LEMONGRASS ESSENTIAL OIL

By

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**(2015-18-002)**

**THESIS**

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**KERALA, INDIA**
**2017**

## DECLARATION

I hereby declare that this project report entitled **"STUDIES ON MICROWAVE STEAM DISTILLATION PROCESS FOR EXTRACTION OF LEMONGRASS ESSENTIAL OIL"** is a bonafide record of research work done by me during the course of research and that the report has not previously formed the basis for the award to me of any degree, diploma, associateship, fellowship or other similar title, of any other University or Society.

**Place: Tavanur**
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## CERTIFICATE

Certified that this thesis, entitled, **"STUDIES ON MICROWAVE STEAM DISTILLATION PROCESS FOR EXTRACTION OF LEMONGRASS ESSENTIAL OIL"** is a record of research work done by Ms. Claudia K. L. under my guidance and supervision and that it has not previously formed the basis for the award of any degree, fellowship, associateship to her.

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**CLAUDIA K.L.**

*Dedicated*

*to*

*God Almighty*

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## LIST OF SYMBOLS AND ABBREVIATIONS

| | | |
|---|---|---|
| $^{o}C$ | : | Degree Celsius |
| % | : | Per cent |
| & | : | And |
| / | : | Per |
| *et al.* | : | and others |
| etc. | : | Etcetera |
| GC | : | Gas Chromatography |
| GHz | : | Giga Hertz |
| G | : | Gram |
| H | : | Hour |
| Ha | : | Hectare |
| i.e. | : | that is |
| I.U | : | International Unit |
| J | : | Joules |
| K.C.A.E.T. | : | Kelappaji College of Agricultural Engineering and Technology |
| Kcal | : | kilo calorie |
| Kg | : | Kilogram |

| | | |
|---|---|---|
| MAE | : | Microwave Assisted Extraction |
| MHz | : | Mega Hertz |
| MSD | : | Microwave Steam Distillation |
| M | : | Meter |
| Min | : | Minute (s) |
| Ml | : | Milli Litre |
| mm | : | Milli Meter |
| mPas | : | Milli Pascal Second |
| P | : | Probability |
| SEM | : | Scanning Electron Microscopy |
| SD | : | Steam Distillation |
| S | : | Second (s) |
| T | : | Tonne |
| V | : | Volt |
| W | : | Watt |
| μ | : | Micro |
| U | : | Frequency |
| λ | : | Lamda |

# *Introduction*

# CHAPTER I

## INTRODUCTION

Highly concentrated aromatic oily liquids distilled from different parts of aromatic plants are called essential oils. These volatile oils represent the typical flavour and aroma of a particular plant from which they are obtained. They are found in flowers, leaves, roots, seeds and barks of aromatic plants. These volatile compounds have the property to solubilise in fatty oils and fats; hence they are known as essential oils. Hydrophobic and viscous characteristics is represented by the term 'oil' and the native essence and typical fragrance of the plant is denoted by 'essential'. The essential oil content of plant material is low, normally 1 to 3% of the plant weight. Hence they are low-volume with complex mix of components and are very high value products.

Essential oils have become an important part of everyday life. These oils are used principally in perfumery and food flavourings. It can be used in variety of ways like natural additives in foods and food products, air fresheners, feed additives, cosmetics, deodorisers and active compounds in packaging materials. Essential oils possess high antimicrobial potential which led to its use as biocides and insect repellents and they are good natural sources of several bioactive compounds. Essential oils can serve as the alternative additives or processing aid as green technology. The rate of essential oil is determining by the amount of essential oil from different parts of plants which is different. The quantities of essential oils produced around the world vary widely. Essential oil production is the key source of exports for many countries, mainly in Africa and Asia. Essential oil export figures for Indonesia, Sri Lanka, Vietnam, and India are very high.

Several extraction methods are applied in the manufacture and extraction of essential oils, and the method employed is normally dependent on what type of botanical material is being used. Essential oils are isolated by physical means such as distillation, squeezing or dry distillation (pyrolysis) of natural materials. Distillation:

mainly steam and hydro-distillation, is the most used conventional method of isolating volatile compounds from plant material into essential oil. Among these methods, steam distillation is extensively utilised for commercial scale production (Burt, 2004).

In steam distillation, the most volatile compounds of the aromatic material were carried by steam generated from water. Then the steam is chilled in the condenser to form the hydrosol. The essential oil is then collected effectively by draining the water. The measure of essential oil produced relies upon the length of distillation time, the temperature, and mainly, the type and quality of the plant material. The yield of essential oils from plants is between 0.005% and 10% usually (Chemat *et al.*, 2013).

These existing conventional extraction methods may encounter loss of some volatile compounds, low extraction efficiency, degradation of unsaturated compounds through thermal or hydrolytic effects and toxic solvent residue in the extract. Low yields, long extraction time, high energy utilisation, high cost and environmental pollution are other deficiencies of these techniques. These deficiencies have prompted the thought of the utilisation of new innovative technique in essential oil extraction, which normally utilise less solvent and energy, like microwave assisted extraction, supercritical fluid extraction, ultrasound extraction, controlled pressure drop process, etc. These advanced innovations which developed gradually conquered the limitation of conventional methods, and enhanced the extraction efficacy.

Microwave energy has been advanced recently for the extraction of organic compounds from environmental matrices. The technological progress in microwave energy has occurred as a result of a requirement for a quick, protected and cheap method. Microwave technique which has the benefit of utilising kinetic effects inside the sample to bring about heating, has enhanced effectiveness and hence utilised in numerous industrial processes. Microwave energy as a non-contact alternative heat source is being used proficiently in the field of extraction. A few classes of compounds like essential oils, aromas, pigments, antioxidants have been extracted

progressively by consuming fraction of energy in contrast with traditional extraction techniques. Microwave extraction methods by giving high purity of final product have also secured the intensive energy expenditure in the purification and separation step.

The portability and controllability via mild increments of heating makes the microwave mediated processes more desirable in the essential oil extraction. The internal heating of the in-situ water within the plant material in the microwave extraction process extends the plant cells and prompts breakage of the of the glands and oleiferous receptacles. The essential oil is oozes out by the process, and vaporised with the water. The distillate is condensed by the cooling system outside the microwave oven to isolate the essential oil. Microwave extraction is a green innovation and appears as a better option for the extraction of essential oils from spice crops (Alam and Sandhya, 2012). In microwave steam distillation process, while the plant material is subjected to microwave radiation, steam produced outside crosses the plant material, evaporates and carries the essential oil towards the condenser where it is isolated and collected.

Lemongrass (*Cymbopogon flexuosus*), a perennial tropical grass with thin, long leaves is one of the main medicinal and aromatic plants. It is cultivated mostly in tropical and subtropical regions of Asia, Algeria, South America, and Africa for its essential oil. In India, lemongrass is cultivated in about 4000 hectare area -largely in Kerala, Karnataka, Uttar Pradesh and Assam (Srivastava *et al.*, 2013). Lemongrass leaves present a characteristic flavour of lemon due primarily to citral. There exists huge international demand for the essential oil of *Cymbopogon* species. Steam distillation is used for extraction of lemongrass essential oil from the dried or fresh leaves of the plant. It is utilised in the manufacture of fragrances, perfumery, cosmetics, detergents, and pharmaceuticals. It is useful in culinary flavorings. Most of the major food categories including alcoholic and nonalcoholic beverages, frozen dairy desserts, candy baked foods, gelatins and puddings, meat and meat products and fat and oils make use of lemongrass oil. Flavour of some fish and can be improved

and wines, sauces etc can be flavoured by using lemongrass oil. (Boukhatem *et al.*, 2014).

Some recently published studies have utilised the microwave energy along with steam distillation for extraction of essential oil from various aromatic plant materials. It is conceivable to maintain mild conditions and have prevalent extraction by utilising microwaves to mediate the extraction and steam. Though each biomaterial reacts distinctively to microwave radiation, they all heated rapidly and more consistently in microwaves than the different thermal treatments. Therefore, the process parameters leading to the efficient extraction and quality of oil needs to be optimised for each material extracted using microwave steam distillation technology. In this context, it may be noted that such optimisation studies pertaining to lemongrass oil has not been found reported.

Taking the above facts into consideration, this research entitled **"Studies on microwave steam distillation process for extraction of lemongrass essential oil"** was undertaken with the following objectives:

1. To develop a microwave steam distillation system for extraction of lemongrass oil.
2. Evaluation of the system developed towards extraction of lemongrass oil, and optimisation of the process parameters.
3. Characterisation of the microwave steam distilled lemongrass oil in comparison with conventional steam distilled oil.

# *Review of literature*

# CHAPTER II

# REVIEW OF LITERATURE

This chapter comprises of various reviews of research work related to the study of microwave steam distillation of lemongrass essential oil. Reviews on lemongrass, its benefits, essential oil, conventional methods of distillation and application of microwave technology in extraction of essential oil has been elaborately presented.

## 2.1 LEMONGRASS

Lemongrass (*Cymbopogon flexuosus*) is a tall, fast-growing, lemon-scented, perennial grass. It has distinct, dark-green foliage. The culm may grow upto 1.8 meter high. Long green leaves are linear tapering upwards and along the margins. This short day plant produces plentiful flowering in South India. The inflorescence is one meter long spike (Srivastava *et al.*, 2013).

### 2.1.1 Area and Production

Lemongrass (barbed wire grass, silky heads, Cochin grass or Malabar grass), a native aromatic tall sedge (family: Poaceae) grows in many parts of tropical and sub-tropical South East Asia, Africa, Sri Lanka and South India. In India, it is widely cultivated in Maharashtra, Kerala, Karnataka, Andhra Pradesh, Tamil Nadu, Uttar Pradesh, Uttaranchal, Assam, Arunachal Pradesh and Sikkim. Currently the world production of oil of lemongrass is around 1000 t annum$^{-1}$ from an area of 16000 ha. At present, India grows this crop in 4,000 ha area and the annual production is around 250 t annum$^{-1}$. India, the largest producer of lemongrass, exports 80% of the produce. Its essential oil has been traditionally exported to West Europe, U.S.A. and Japan (AMPRS, 2013).

### 2.1.2 Varieties

Lemongrass is extensively cultivated in the tropics and subtropics. The two species that are for the most part cultivated are: East Indian lemongrass, *Cymbopogon flexuosus*, which is also known as Cochin or Malabar grass, and West Indian lemongrass *Cymbopogon citratus*. There are two known types of *C. flexuosus* on the

basis of stem colour: The red-stemmed grass, which is known as the true lemongrass and is suggested for higher oil yields (oil content: 0.3-0.5%, Citral: 80-86%), and the white-stemmed (*C. flexuosus* var. *albiscens*) yields oil of lower citral content (Oil content: 0.4-0.7%, Citral: 55-70%). There are also a few types of *C. citratus* of which do not yield enough essential oil.The varieties of lemongrass grown in the country includes Sugandhi (OD 19), Pragati, Praman, Jama Rosa, RRL 16, CKP 25, OD-408 and Kaveri (Dutta *et al.*, 2014).

**2.1.3 Lemongrass Essential Oil**

Essential oils are mixtures of organic chemicals formed as by-products of plant metabolism. There is a huge international demand for the essential oil of *Cymbopogon* species. Major constituents of lemongrass essential oil are tabulated below (Table 2.1). These are known to have antifungal, antiseptic, insecticidal and counterirritant properties. The essential oil of lemongrass has strong lemon-like odour, because of high percentage (over 75%) of citral in the oil. Lemongrass essential oil is extracted by steam distillation for 1.5 to 2 h from the dried or fresh leaves of the plant. The distillate is condensed by the cooling system to isolate the essential oil and collected by decantation. Normal oil yield is 50-70 kg $acre^{-1}$ $year^{-1}$. Lemongrass oil, viscous liquid, yellow/ dark yellow/ dark amber in colour, is caustic in nature and volatile under ambient temperatures.

Anhydrous sodium sulphate at the rate of 2 g $kg^{-1}$ oil is added to take out moisture in the distilled oil. It ought to be kept overnight and filtered thereafter through filter paper or muslin fabric. The oil turns red on prolonged storage. To avoid this, oil can be kept in shaded or cool area away from direct heat and daylight. The oil can be easily stored in amber coloured glass bottles if the amount is less and stainless steel or aluminium containers are preferred for vast amounts (AMPRS, 2013)

Table 2.1. Major constituents of lemongrass essential oil (AMPRS, 2013).

| **East Indian Lemongrass Essential oil - Chemical Constituents** | |
|---|---|
| Oil content | 0.2 – 0.4 % |
| Main constituent | Citral |
| Citral | 70% |
| Geraniol | 5.00% |
| Limonene | 2.42% |
| Citronellol, nerol | 2.20% |
| Linalyl acetate, geranyl acetate | 1.95% |
| Methyl heptanone | 1.50% |
| Linalool | 1.34% |
| Caryophyllene, β-pinene, β-thujene, myrcene | 0.46% |
| Citronellal | 0.37% |
| α-terpineol | 0.24% |
| α-Pinene | 0.24% |
| β-Ocimene | 0.06% |
| Terpenolene | 0.05% |

### 2.1.4 Utilisation of lemongrass

Lemongrass contains several important bioactive compounds which are useful in several health issues (Olorunnisola *et al.,* 2014). Roacha *et al.* (2011) identified that lemongrass can be utilise for medicinal purposes, especially as infusion. Its essential oil is used in perfume, food and pharmaceuticals. It can be used for flavouring chicken and rice preparation. It is unique for flavouring green tea. The oil has very good aroma therapeutic properties and good medicinal properties. Research work by Vinesh *et al.* (2014) concluded that lemongrass oil has anti-bacterial, anti-

fungal, anti-oxidant, anti-proliferative, anti-viral and anti-inflammatory properties, hence it can be used to treat various diseases in human. Jafari *et al.* (2012) stated that lemongrass essential oil has the ability to control bacterial growth and fungal pollutants in food such as *Staphylococcus aureus* and *Escherichia coli*. Essential oil of lemongrass has good antibacterial activity against *A.baumannii* and might be considered as an alternative treatment option against multi-drug resistant infections (Aparna *et al.*, 2015).

Pure citral, separated from lemongrass oil, is utilised as a starting material in the synthesis of Vitamin A and other industrially imperative products. The generation of soft drinks, desserts, confectioneries, beverages, bakery items, chewing gums etc make utilisation of the pure citral. A little amount of oil is utilised, as such in soaps, detergents and other preparations (NHB: Lemongrass).

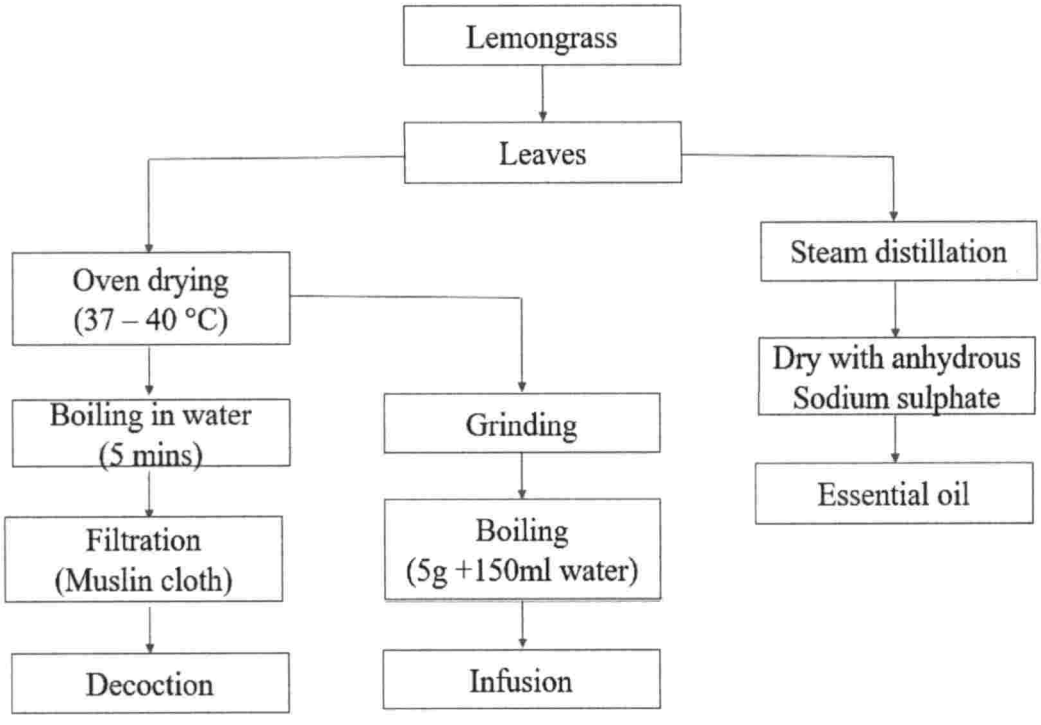


Figure 2.1. Methods of extracting bioactive compounds from lemongrass (Olorunnisola *et al.*, 2014)

Spent lemongrass, the residue got after extraction, is utilised as roughage for dairy cattle, for ensilaging, for mulching or manuring crops as such or after enriched composting. In some plantations in India, it is utilised after drying as a fuel for distillation. It is utilised as cheap packaging material and also for the manufacture of cardboard and paper. The spent lemongrass contains 7.4% crude protein, 0.17% Ca and 0.09% P (AMPRS, 2013).

## 2.2 CONVENTIONAL METHODS OF EXTRACTION

Essential oils are commonly extracted by distillation, mostly hydro distillation and steam distillation. Other processes include expression, solvent extraction, cold pressing etc. In hydro distillation method, the herbage is packed in a vessel which is halfway loaded with water and heated by direct fire without an external boiler. This method is less proficient, however the unit is simple and less expensive. Steam distillation utilises an external boiler to produce steam which is to be brought into the chamber. In spite of the fact that the technique is more effective, it includes higher capital cost, yet the quality of oil obtained is superior. Thus the most usually utilised method is steam distillation.

The steam distillation method involves placing of plant part, from which essential oil to be extracted (which can be either fresh or dried), into a vessel. Then, pressurised steam is allowed to concentrate in the area, where the items are placed, and allowed to saturate. After saturation, the essence (volatile aroma compounds) of plant part is sweated out due to bursting of cell walls and passes through a condenser along with steam. Essential oil can be collected by separating the hydrosol.

The main shortcomings of these conventional methods include low yields, long extraction time, high energy consumption, high cost, thermal and hydrolytic degradation and environmental pollution (Tongnuanchan and Benjakul, 2014). These inadequacies prompted new innovative techniques in essential oil extraction. Modern technologies which grew continuously conquered the limitation of conventional methods, and upgraded the extraction efficacy. Microwave assisted extraction is one of such techniques on the basis of interaction between water in the plant material and

microwaves generated by the energy source. Contrasted with regular techniques microwave assisted extraction has high extraction rates, reduced energy consumption and more environment friendliness.

## 2.3 MICROWAVES

Datta & Anantheswaran (2000) cited by Chandrasekharan *et al.* (2013) outlines that microwaves are electromagnetic waves with frequency varies from 300 MHz to 300 GHz and wavelength of 1 mm to 1 m. In order to prevent interferences of radio communications, domestic and industrial microwaves normally work at a frequency of 2450 MHz whereas industrial microwave systems work at frequencies of 915 MHz.

The energy of the radiation, E, is directly related to the frequency, $\upsilon$, the number of oscillations per second, by Planck's constant, $h = 6.63 \times 10^{-34}$ J/s:

$$E = h / \upsilon$$

The frequency, $\upsilon$, is inversely related to the wavelength, $\lambda$, by:

$$\upsilon = c / \lambda$$

where c: velocity of the radiation (Scaman and Durance, 2005).

There are wide applications of microwaves in the field of food processing. Effective destruction of pathogenic microorganisms is conceivable in microwave heating technology for pasteurisation and sterilisation. Utilisation of microwave heating preserved food with better nutritional quality in food processing applications like blanching, cooking, baking etc. Heat is generated volumetrically all through the product rapidly in microwave heating unlike the traditional thermal processing techniques. Contrasted with conventional methods, processing time is much reduced without serious damage in overall quality of liquid food (Okeke *et al.*, 2014).

## 2.4 MICROWAVE HEATING

Kratchanova *et al.* (2004) investigated the effect of microwave pretreatment of fresh orange peels. Pretreatment led to destructive changes in the plant tissue which is proved by scanning electron micrographs. The changes resulted in an increase in the capillary-porous characteristics and the water absorption capacity of

the plant material which facilitated the pectin extraction with improved quality and attained considerable increase in pectin yield.

The study conducted by Cheng *et al.* in 2011 demonstrates that microwave pre-treatment combined with solvent extraction is a clean and dry process for the production of crude palm oil (CPO). The results shows that microwave heated palm fruits (for three min) achieved an oil yield similar to the conventional and commercial palm oil milling methods. Fatty acid composition (FAC) of the CPO implies an increment in lauric acid with the increase of microwave exposure time. Palm fruits subjected to microwave heating for two min noted the higher concentration of vitamin E and carotene content. Results revealed that the quality of CPO produced utilising microwave treatment combined with hexane extraction possesses better qualities than the conventional palm oil milling process.

Rodrigues *et al.* conducted an experiment in 2012 to monitor the protective effects of *Lavandula latifolia* essential oil during soybean oil microwave heating. Several parameters of soybean oil with and without spike lavender essential oils which is exposed to microwave heating for different intervals of time (1, 3, 5, 10 and 15 min; 1000 W) were evaluated. The microwave heating impacts were unique in the samples with essential oils addition, permitting segregation from plain soybean oils by a principal component analysis.

### 2.4.1 Principles of Microwave Heating

The basis of the principle of heating utilising microwave energy is the direct effects of microwaves on molecules of the material. The ability of the materials to absorb microwave energy and convert it into heat causes the microwave heating. The change of electromagnetic energy into heat in microwave heating of food materials, happens by two mechanisms: ionic conduction and dipole rotation.

Ionic conduction occurs because of the oscillatory movement of ions in the food which produces heat in the presence of a high frequency oscillating electric field. Conversion of kinetic energy to thermal energy is caused by the resistance of

the solution to the flow of ions and the collision between ions. This results in heating up of the solution.

The dipole rotation is related to alternative movement of polar molecules having dipole moments. When an oscillating electric field is collided on the water molecules in the food material, the permanently polarised dipolar molecules try to arrange themselves towards the electric field. because the high frequency the electric field, this realignment occurs at a million times per second and causes internal friction of molecules resulting in the volumetric heating of the material (Datta & Davidson, 2000).

There are many factors which influence microwave heating and its heat distribution and the most important of them are the dielectric properties and penetration depth.

### 2.4.2 Dielectric Properties of Food Material

By knowing dielectric properties of material, the ability of a material to convert microwave energy into heat can be understood. The ability to store electric energy indicates dielectric constant - the real part of dielectric property and the ability to convert electric energy into heat is dielectric loss – the imaginary part of dielectric property.

Ikediala *et al.* (2000) specified that the dielectric properties of materials can be defined in terms of their relative permittivity. The permittivity related to free space is known as relative complex permittivity (Ɛ*), and it is represented as:

$$\varepsilon^* = \varepsilon' - j\varepsilon''$$

where, Ɛ' is dielectric constant, Ɛ" is dielectric loss and $j = \sqrt{-1}$ .

The loss tangent gives ratio of dielectric loss to dielectric constant and is expressed as,

$$\tan \delta = \frac{k''}{k'} = \frac{\varepsilon''}{\varepsilon'}$$

where k': relative dielectric constant and k": relative dielectric loss and are given as $k' = \varepsilon'/\varepsilon_0$ and $k'' = \varepsilon''/\varepsilon_0$, ($\varepsilon_0$ is the permittivity of free space; $\varepsilon_0 = 8.854 \times 10^{-12}$F/m).

Main factors that affects the dielectric properties of material are operating temperature and the microwave frequency. Another factor deciding the dielectric properties of the food material is the amount of moisture in a food material because water is a better absorber of microwaves.

Penetration depth and electrical conductivity are the are alternate properties identified with dielectric parameters. The power penetration depth (Dp) is characterised as the distance at which the power density drops to a value of 1/e (e = 2.718) from its value at the surface. The penetration depth is a function of ε' and ε" (Sosa-Morales *et al.*, 2009).

Icier and Baysal, 2004 cited by Chandrasekaran *et al.*, 2013 described that the dielectric properties and the penetration depth are the chief factors which affect microwave food processing. There are other factors like microwave frequency, microwave oven design (oven size and geometry), moisture content, density, load, composition, shape and the size of food materials and the placement of food material inside the oven.

## 2.5 MICROWAVE ASSISTED EXTRACTION (MAE)

Microwave energy could be used effectively to mediate extraction of essential oil in place of steam or water heating in order to introduce its inherent advantages. It has been found that the use of microwaves for extraction of active components could result in enhanced performance in terms of quality and quantity such as high extraction and efficiency, less extraction time and yield with quality of the extracted oil superior to that of other conventional methods due to the mild conditions. Besides, microwave extraction may be classified as a green technology which is energy efficient and controlling the process is easy.

Abu-Samra *et al.* in 1975 mentioned the utilisation of microwave energy for the first time. Domestic ovens in the laboratory for the treatment of biological samples for metal trace analysis was utilised by them. Ganzler *et al.* in 1986 depicted the extraction of organic compounds utilising microwave irradiation. Pare in 1995 documented the first patent for extraction of a natural product by microwaves (Destandu *et al.*, 2013).

Microwave assisted extraction (MAE) is one of the advanced method of extraction, in which extraction happens as the consequence of changes in the cell structure caused by electromagnetic waves. MAE has gained acceptance as a mild and controllable processing tool. In MAE, the heat is dissipated volumetrically inside the irradiated medium while in traditional extraction the heat is transferred from the heating medium to the interior of the sample. Thus MAE exhibits process acceleration and high extraction yield. MAE is a simple, quick and low-solvent-consuming process. These might be the aftereffect of a synergistic combination of two transport phenomena: heat and mass gradients working similar way. Then again, in conventional extractions, the mass transfer happens from inside to the outside, although the heat transfer happens from the outside to the within the substrate (Veggi *et al.*, 2013).

Guo *et al.* (2001) worked on microwave-assisted extraction of the puerarin from the Chinese herbal medicine Radix puerariae and found that the process can be finished in one minute. Solvent, time of extraction and the temperature and pressure in the sample vessel are the principle factors that influence MAE of effective constituents from Radix puerariae . The study revealed that the microwave irradiation did not destroy the structure or molecule of the puerarin.

Pan *et al.* (2003) utilised microwave-assisted extraction (MAE) method for the extraction of tea polyphenols and tea caffeine from green tea leaves. The extraction for four min was higher than those of extraction at room temperature for 20 h, ultrasonic extraction for 90 min and heat reflux extraction for 45 min, respectively. They found that MAE was better than the conventional extraction methods in terms

of the extraction efficiency, the extraction time and the percentages of tea polyphenols or tea caffeine in extracts.

Saoud *et al.* (2006) carried out the investigation of microwave extraction of eucalyptus essential oil using ethanol as solvent. The results showed that the highest concentration was obtained at the ratio of 6 g leaves per 100 ml ethanol. The yield of eucalyptus essential oil was 4.5 mg $g^{-1}$ fresh leaves using an exposure time of three min, 10 doses of exposure and 1000 W microwave power.

Microwave-assisted extraction (MAE) for pectin extraction from the dried apple pomace was studied by Wang *et al.* (2007). The pectin yield was 0.315 g from 2 g dried apple pomace under optimised conditions (pH of 1.01, microwave power of 499.4 W, extraction time of 20.8 min, solid: liquid ratio of 0.069). Response surface methodology was utilised to optimise the effects of processing parameters of extraction. The process of MAE reduces extraction time dramatically.

In 2012, Karabegovic *et al.* studied on optimisation of a microwave-assisted extraction of cherry laurel (*Prunus laurocerasus*) fruit using methanol by utilising response surface methodology. The maximum yield of 41.85 g per 100g fresh plant material was achieved at optimised condition (microwave power: 600 W, plant material to solvent ratio: 0.2 g $cm^{-3}$ and extraction time: 26 min). The outcomes demonstrated that process parameters were effective on the extraction efficiency, predominantly extraction time.

The microwave-assisted extraction (MAE) process of phenolic antioxidants from grape seeds (*Vitis vinifera*) was optimised by Krishnaswamy *et al.* (2012) utilising a central composite design. Microwave power, extraction time and solvent concentration and their interactions on total phenols and the antioxidant activity were chosen as independent variables and their influence on MAE process were determined. The response variables were maximised with a desirability function of 0.947 (six min, 32.6% ethanol and 121 W).

An optimised procedure based on microwave-assisted extraction is developed in 2013 by Svarc-Gajic *et al.* for the extraction of total polyphenols, flavonoids,

anthocyanins, monomeric and condensed anthocyanins from rosemary. By using 70 % of methanol, highest yield of total polyphenols was obtained while flavonoids were most efficiently extracted in the microwave field with 70% ethanol. Anthocyanins, monomeric and condensed anthocyanins, were all best extracted with 70% ethanol and 1% acetic acid. Microwave power of 800 W was found best for total polyphenols, whereas for flavonoids, anthocyanins, monomeric and condensed anthocyanins the power of 320 W given the best extraction efficiency.

Taghvaei *et al.* (2014) evaluated the influence of MAE on oxidative stability and physicochemical properties of cottonseed oil. MAE cottonseed oil were found not significantly different ($P > 0.05$) than conventionally-extracted cottonseed oil by GC analysis. The results showed that MAE oil samples from whole cottonseed had the long-term stability, more than BHT contained oil samples.

Nde *et al.* (2016) conducted experiments on microwave assisted extraction (MAE) of butter from sheanut kernels utilising the Doehlert's experimental design. Under optimum conditions (heating time: 23 min, temperature: 75°C and solvent/solute ratio: 4:1) more than 88% of the oil with a free fatty acid (FFA) value less than two was extracted by MAE method contrasted to soxhlet extraction (10 h and solvent/solute ratio: 10:1). MAE process was found to be beneficial in terms of extraction time and volumes of solvent utilised and oil of suitable quality.

Thakker et al. (2016) extracted essential oil from the leaves of palmarosa using microwave radiation, for maximisation of oil yield and geraniol yield and zone of inhibition (ZOI) as responses. The optimised extraction conditions were attained at, solid loading of 35 g, water volume of 300 ml, microwave power of 850 W and extraction time of 20 min. Under optimised conditions, 2.4400% (w/w) yield of essential oil, 2.1700% (w/w) yield of geraniol and 20 mm ZOI were obtained.

### 2.5.1 Methods of Microwave Assisted Extraction (MAE)

Several microwave assisted extraction methods have been developed to conquered the shortcomings of conventional extraction techniques like microwave assisted hydro distillation (MAHD), microwave-accelerated steam distillation

(MASD), microwave steam distillation (MSD), solvent free microwave extraction (SFME), microwave hydro diffusion and gravity (MHG), vacuum microwave hydro distillation (VMHD) and so on.

A dissolvable free microwave extraction (SFME) was created by Lucchesi et al. (2004). They SFME and a regular method, hydro-refining (HD), for the extraction of fundamental oil from three sweet-smelling herbs: basil (Ocimum basilicum L.), cultivate mint (Mentha crispa L.), and thyme (Thymus vulgaris L.). The basic oils extricated by SFME for 30 min were quantitatively (yield) and subjectively (fragrant profile) like those got by ordinary hydro-refining for 4.5 h. The SFME technique yields a fundamental oil with higher measures of more significant oxygenated mixes and permits considerable investment funds of expenses, as far as time, vitality and plant material.

A solvent-free microwave extraction (SFME) was developed by Lucchesi *et al.* (2004) for the extraction of essential oil from three aromatic herbs: basil (*Ocimum basilicum* L.), garden mint (*Mentha crispa* L.), and thyme (*Thymus vulgaris* L.). They contrasted SFME with hydro-distillation (HD). The essential oils extracted by SFME (30 min) and by conventional hydro-distillation (4.5 h) were quantitatively (yield) and qualitatively (aromatic profile) similar. An essential oil with higher amounts of more valuable oxygenated compounds were yielded by SFME with considerable savings in costs, in respect of energy, time and plant material.

An improved solvent free microwave extraction (SFME) was developed by Wang *et al.* (2006). The sample was mixed with carbonyl iron powders (CIP) and extraction of essential oil from the dried plant materials was applied without any pre-treatment. They compared improved SFME with conventional SFME, microwave-assisted hydro-distillation (MAHD) and conventional hydro distillation (HD) for the extraction of essential oil from dried *Cuminum cyminum* L. and *Zanthoxylum bungeanum* Maxim. The time of extraction in Improved SFME was 30 min, Conventional SFME (85 W) was 50 min, Microwave assisted hydro-distillation was 90 min and Hydro distillation was 180 min.

Lucchesi *et al.* (2007) conducted a study on the solvent-free microwave extraction (SFME) of cardamom essential oil. Essential oils extracted by SFME are more valuable and made out of highly odoriferous aromatic compounds and dominated by the oxygenated fraction.

MAHD was prevalent in regard of sparing extraction time (75 min, rather than 4 h in HD) and vitality. Confirmations as to a rapid burst of fundamental oil organs with MAHD were given by Scanning electron microscopy (SEM) of thyme leaves experienced HD and MAHD. Gas chromatography– mass spectrometry investigation of the extricated basic oils drew out the way that the utilization of microwave illumination did not unfavorably impact the creation of the basic oils. They discovered MAHD as a green innovation.

Vian *et al.* (2008) developed MHG for the extraction of essential oil from two aromatic herbs: spearmint *(Mentha spicata* L.*)* and pennyroyal *(Mentha pulegium* L.*)*. They compared MHG with hydro-distillation (HD). The essential oils extracted by MHG (15 min) were similar to those obtained by HD (90 min) in terms of quantity and quality. MHG saves energy (90%) and thus prevents pollution leads to beneficial as greenhouse gas emission.

In 2009, Bousbia *et al.* developed a new and original process for the extraction of natural substances using microwave hydro-diffusion and gravity (MHG) and applied MHG, HD (Hydro-distillation) and CP (Cold Pressing) techniques to extract essential oil from citrus peels. The energy required to perform the two extraction methods are respectively 3 kWh for HD, and 0.2 kWh for MHG. MHG (15 min) is clearly faster than HD (180 min) with comparable yields.

Zill-e-Huma *et al.* (2011) studied on solvent free microwave hydrodiffusion and gravity (MHG) method of four varieties of *Allium cepa* (red, yellow, white and grelot onion) and evaluated for the total content of reducing compounds (TCRC) of quercetin (flavonol) glycosides, flavonol content and antioxidant activity. They analysed the extracts obtained by (MHG) technique and conventional solvent extraction (CSE) with high performance liquid chromatography (HPLC). The highest

antioxidant capacity was obtained for red onion. MHG was superior to the conventional method yet it gives low recovery of extractable flavonoid. All the samples obtained under MAE exhibited the highest antioxidant activities. The microscopic observations of extracted tissues provided evidences to promote the effectiveness of MHG that microwaves induced disruptions of vacuoles and cell walls at cellular level.

Solvent-free microwave extraction (SFME) of the essential oil from *Dryopteris fragrans* and its antioxidant activity were investigated by Xiao-Juan *et al.* in 2012. They applied a central composite design with response surface methodology to study the effect of irradiation power, extraction time and humidity (proportion of water pretreatment). Under optimal conditions (time of extraction 34 min, irradiation power 520 W and humidity 51%), the best extraction yield of 0.33% was achieved. The results suggested that SFME is alternate protocol for production of essential oils.

Solvent-free microwave extraction (SFME) of essential oil from *Schisandra chinensis* (Turcz.) Baill was studied by Ma *et al.* in 2012. The effect of three major variables influencing the performance of SFME was evaluated utilizing central composite design (CCD). The essential oil yield of 11 ml $kg^{-1}$ was obtained under the optimal conditions of extraction time of 30 min, irradiation power of 385 W and 68% moisture content of the fruits. More evidences were provided by GC-MS, scanning electron microscopy (SEM) and thermo-gravimetric analysis (TGA) to establish SFME is an effective technique than HD and SD.

For extracting cassia oil and proanthocyanidins from *Cortex cinnamomi*, a study was conducted by Liu *et al.* (2012) on ILMSED method (ionic liquid-based microwave-assisted simultaneous extraction and distillation). They selected 0.5 M 1-butyl-3-methylimidazolium bromide ionic liquid as solvent. The yield of essential oil was 1.24 ± 0.04% and proanthocyanidins was 4.58 ± 0.21% under the optimum conditions of microwave power of 230 W, 15 min microwave extraction time and 10 liquid–solid ratio for 20 g sample. The outcomes disclosed that ILMSED method is a

better alternative for the extraction of both the essential oil and proanthocyanidins from *Cortex cinnamomi*, with less energy consumption and better extraction yield.

Bittar *et al.* (2013) studied on extraction of of grape juice by-product utilising Microwave Hydro diffusion and Gravity (MHG). They analysed MHG extract (MHGE) using HPLC. They prepared innovative grape juice (IJ) by adding MHGE to the natural juice (NJ). For evaluating three juices, total polyphenol content (TPC), total anthocyanin content (TAC) along with sensorial characteristics was found out. MHGE revealed the highest values of TPC and TAC.

Abdurahman *et al.* (2013) investigated the performance of MAHD in the extraction of essential oil from Ginger (*Zingiber officinale* Roscoe) and Lemongrass (*Cymbopogon citratus).* Maximum essential oil yield from Ginger and Lemongrass (0.85% (w/w) and 1.37% (w/w), respectively) were ensued (water to raw material ratio of 8:1, microwave power of 250 W and time of 90 min). The results proved that MAHD is a cost effective method and provide maximum oil yield at shorter extraction period without significant effect on their chemical constituents.

Gholivand *et al.* (2013) analysed volatile components in *Echinophora platyloba* DC utilising microwave distillation and then using headspace single drop microextraction (MD–HS SDME). A single drop of n-heptadecan was utilised to collect the headspace volatile compounds after irradiation. The relative peak areas were used as index to optimise the extraction conditions. The confirmation of chemical composition of the MD–HS–SDME extracts was possible according to their retention indexes and mass spectra.

Ranitha *et al.* (2014) conducted a study on MAHD of Lemongrass essential oil *(Cymbopogon citratus)*. A yield of 1.46% was obtained under optimum conditions (water to plant material ratio of 8:1, microwave power of 250 W and extraction time of 90 min). Composition of essential oils did not affect adversely by utilising microwave exposure since the presence of major constituents with similar quality and quantity was proved by GC-MS analysis in essential oils extracted by both methods.

The results indicated that MAHD method provides a good alternate for the extraction of essential oil from Lemongrass (*Cymbopogon citratus*).

A fast, simple and green sample pretreatment method for the extraction of eight carbamate pesticides in rice was introduced by Song *et al.* (2014). Microwave assisted water steam extraction method was used to extract the carbamate pesticides. They applied the extract immediately on a C18 solid phase extraction cartridge for cleaning and concentration. Compared with traditional methods, the microwave assisted method was found to be effective with less extraction time.

To determine the effect of extraction methods on qualitative and quantitative characteristics of the essential oil of Bakhtiari savory, Memarzadeh *et al.* (2015) used different methods for extraction. For essential oil extraction from the aerial parts of savory, hydro distillation using two Clevenger-type apparatus and traditional steam and water distillation (TSWD), innovative technique steam distillation (SD innov), microwave-assisted steam hydro-diffusion (MSHD 400 and 800 W), and microwave assisted hydro-diffusion (MHD 400 and 800 W) were utilised. The maximum essential oil yields were obtained from hydro distillation, and MSHD 400 W. The highest amounts of oxygenated fractions (thymol and carvacrol) were found from MHD (800 W) method than from the other methods. From the experiments conducted, they revealed that extraction of the essential oil from Bakhtiari savory with microwave-assisted hydro-diffusion (MHD) was found best in respect of energy saving, extraction time, oxygenated fraction and product quality.

Gavahian *et al.* (2015) studied ohmic and microwave assisted hydro distillation (OAHD and MAHD) of essential oils from the aerial parts of peppermint. They compared the results with those of the conventional hydro-distillation (HD). It was observed that MAHD was superior in respect of extraction time and essential oils accumulation rate.

Microwave-assisted simultaneous distillation and dual extraction of essential oil from leaves of *Vaccinium uliginosum* was studied by Chen *et al.* (2015). They separated out chlorogenic acid and hyperoside effectively. Optimum conditions were

found to be 20 ml $g^{-1}$ liquid–solid ratio, 26 min of microwave irradiation time, and 389 W of microwave irradiation power. New method produced higher yields of oil in a short time without significant changes in chemical composition of essential oil than that of conventional methods.

Chen *et al.* (2016) conducted experiments on solvent-free microwave extraction (SFME) of Pomelo peel for essential oils and then on a hot-solvent microwave extraction (HSME) for pectin. For essential oil extraction, SFME was found better to the hydro distillation (HD) method and for pectin extraction HSME was more suitable than acidic solution method in respect of extraction efficiency and yield of targeted component.

Sagarika *et al.* (2016) carried out experiments on microwave assisted extraction of nutmeg mace essential oil, compared it with conventional hydrodistillation and the quality characteristics of the essential oil were determined. Physical quality characteristics such as refractive index, specific gravity, solubility and colour of essential oil were found to be similar in both methods whereas the main chemical constituent myristicin was found to be slightly higher in microwave assisted process than the hydro distillation method.

## 2.6 MICROWAVE STEAM DISTILLATION

Some recent studies have utilised the microwave energy along with steam distillation for extraction of essential oil from various aromatic plant materials. Microwaves can be utilised to facilitate the extraction and steam, thus it can maintain mild conditions and have better extraction.

An advanced steam distillation (SD) method using a microwave oven in the extraction process is microwave steam distillation (MSD). In this advanced technique, only the plant material is exposed to microwave radiation, since essential oil of the aromatic plant has a considerably higher dielectric loss than the surrounding steam. Cartridge in which plant material enclosed, is connected to an electrical steam generator and a cooler situated outside the microwave reactor. Vapour crosses the plant material along with microwave heating. The glandular trichomes are exposed to

thermal stresses and localised high pressures due to microwave exposure. Plant cells distends and starts to rupture because of this high pressure. Essential oil evaporates and carries by the steam flow through the sample. The extraction can be prolonged till no more oil is obtained.

An advancement of the microwave accelerated steam distillation (MASD) is microwave steam distillation (MSD). An innovative process design and operation for MASD of essential oils from lavender flowers was established by Chemat *et al.* (2006). In this process, microwave radiation is only applied on the extraction reactor, which is on the basis of conventional steam distillation principle. The cooling system and the part estimated to collect essences are positioned outside the oven. A packed bed of lavender flowers (*Lavandula angustifolia* Mill., Lamiaceae) was kept on the steam source. Only steam can passes through it unlike hydro-distillation, to avoid the mixing of boiling water with vegetable raw material. They found that extraction of lavender oil with MASD was advantageous than SD in respect of energy saving, time of extraction (10 min: MASD, 90 min: SD), cleanliness and essential oil yield and quality.

Deng *et al.* (2006) worked on combination of microwave distillation (MD) and solid-phase micro extraction (SPME) for the determination of essential oil compounds in traditional Chinese medicine (TCM). They found that MD–SPME was better in terms of extraction time (3 min for MD-SPME, 6 h for SD) and amount of organic solvent. SD detected 26 compounds whereas 49 compounds in TCM were isolated and identified by MD-SPME. The new method has a good precision which is illustrated easily from the values of relative standard deviation (less than 9%). From the study, it was concluded that MD-SPME is a simple and fast method for the determination of essential oil compounds in fresh materials.

Sahraoui *et al.* (2011) conducted a study on microwave steam distillation (MSD) of essential oils from fresh citrus by-products (orange peels). They evaluated the efficacy of MSD and SD. MSD was superior with low extraction time (six min), cleanliness and gives an essential oil with better sensory properties at optimised

power (500 W). A sort of microwave superheating phenomena is caused due to microwave heating of water inside the orange peels. It facilitates the distension of the plant cells which leads to oozing out of the essential oil quickly. Results confirmed the efficiency of this innovative method that saves energy and time.

Farhat *et al.* (2011) studied on microwave steam diffusion (MSDf) apparatus for extraction of essential oils from orange peel (byproducts) and compared it with conventional steam diffusion (SDf). Earth's gravity makes the a mixture of hot crude juice and steam moves naturally downwards into a spiral condenser outside the microwave cavity for orange essential oil extraction. The optimal conditions for the extraction were found at steam mass flow rate of 25 g $min^{-1}$ with microwave power of 200 W. The extraction rate (12 min) was accelerated by the MSDf process due to a rapid increase of temperature compared to conventional steam diffusion (40 min) without changing the volatile composition. Extraction of essential oils from orange peel with MSDf was found to be superior to SDf in respect of energy saving, purity and less waste water.

Song *et al.* (2012) conducted a study on microwave-assisted steam extraction (MASE) for the extraction and determination of paeonol in *Cortex moutan*. The reduced time cost and the avoided use of organic solvents suggested that the used method was effective. In comparison with other extraction methods like dynamic microwave-assisted extraction, ultrasonic extraction, and steam distillation it was found that the MASE was a quick and environment friendly. The combination of MAE and SD shows substantial advantages in extraction procedures.

Kusuma *et al.* (2016) conducted a study on extraction of essential oil from the orange peels (*Citrus auranticum* L.) by microwave steam distillation. The outcomes were contrasted SD in respect of extraction yield, time of extraction, chemical composition, and quality of the essential oils. MSD was found better with good extraction yield and extraction time (140 min for MSD, and 7 h for SD). Scanning electron microscopes (SEM) of orange peel undergone SD and MSD revealed that essential oil glands ruptured rapidly with MSD process than that of SD. Composition

of extracted essential oil, analysed by Gas chromatography–mass spectrometry, was found similar in both method and did not indicate any adverse effect by microwave application. They found MSD is an alternate technique to extract orange essential oil which is used for fish growth promoter.



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# *Materials and methods*

# CHAPTER III

# MATERIALS AND METHODS

This chapter outlines the materials and methods adopted for satisfying the objectives of the studies on microwave steam distillation of lemongrass essential oil. The design and development of a microwave assisted process for distillation of lemongrass essential oil was portrayed. The materials used for fabrication of the various components and the instrumentation employed for measurement of parameters were explained. The process of evaluation and optimisation of process parameters for extraction of lemongrass essential oil with maximum oil yield, minimum temperature and energy consumption and the methods for determining the physical and chemical properties of essential oil were detailed.

## 3.1 DEVELOPMENT OF MICROWAVE STEAM DISTILLATION SYSTEM FOR LEMONGRASS ESSENTIAL OIL

The design of a small capacity microwave steam distillation unit for essential oil was conceptualised, and further fabricated based on a thorough review of works carried out on microwave steam distillation. The developed experimental system is shown in Figure 3.1. and Plate 3.1.

The developed microwave steam distillation system composed of the following main components:

1) Microwave reactor
2) Steam generator
3) Cartridge
4) Extraction unit
5) Supporting stand
6) Energy meter
7) Temperature sensor and controller

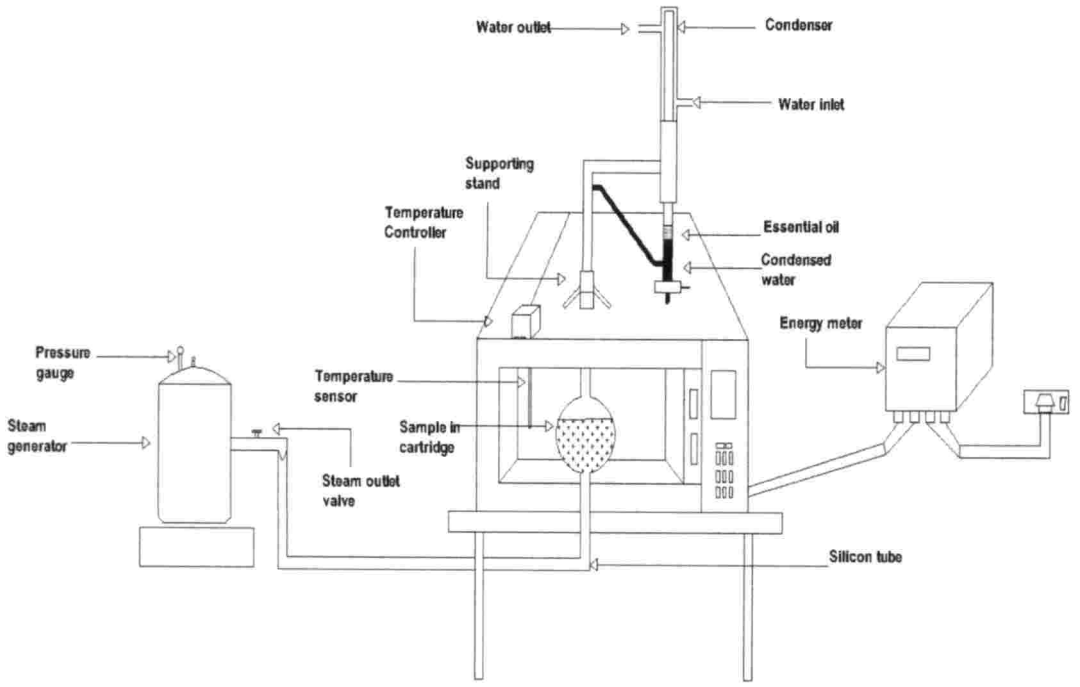


Figure 3.1. Schematic diagram of Microwave Steam Distillation

Plate 3.1. Microwave steam distillation



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### 3.1.1 Microwave Reactor

An advanced steam distillation technique utilising microwave oven for the extraction process is microwave steam distillation (MSD). Hence major requirement for the microwave steam distillation process is a microwave reactor. Commercially available microwave ovens could be effectively utilised for this purpose. The selection of microwave oven should be based on the power consumption. For laboratory scale experiments, ovens with power delivery of 700 W was chosen (Chemat *et al.*, 2006; Sahraoui *et al.*, 2011; Farhat *et al.*, 2011; Kusuma *et al.*, 2016).

Accordingly, a microwave oven (Model: LG MH2044DB) with following specifications was used to serve as the microwave source.

| | |
|---|---|
| Power consumption | 230 V/50 Hz, 700 W (Microwave Output) |
| | 980 W (Grill Input) |
| | 2000 W (Combination Input, Max) |
| Microwave frequency | 2450 MHz |
| Outside dimensions | 260 mm(h) x 455 mm(w) x 340 mm(D) |
| Oven cavity dimensions | 185 mm(h) x 275 mm(w) x 250 mm(D) |
| Oven capacity | 20 litres |

The selected microwave oven has a control panel where time, power levels and action indicators are displayed. The microwave oven is equipped with five power levels such as 140 W, 280 W, 420 W, 560 W and 700 W for giving maximum flexibility and control over microwave application. The process of oil extraction was performed at power levels of 280 W, 420 W and 560 W. The time for extracting the oil can be set on the control panel.

### 3.1.2 Steam Generator

The steam generator (10 L capacity pressure vessel) placed outside the microwave oven produced steam from water by using LPG as heating medium. Steam outlet from steam generator was connected to the cartridge containing

lemongrass via silicon tube (Plate 3.2.). The flow of steam to cartridge was controlled by employing a regulatory valve. Pressure of steam generated was measured using pressure gauge (Micro EN837-1; 0-3.5 kg $cm^{-2}$ or 0-50 psi) (Plate 3.3.) attached to the lid of pressure vessel.

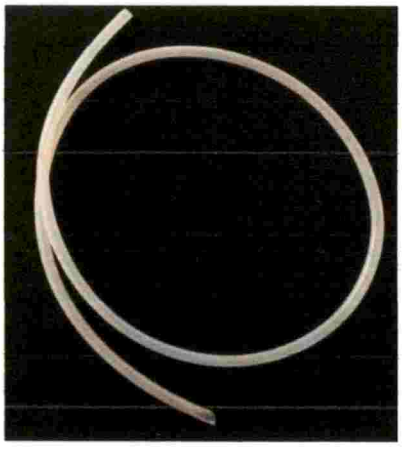

Plate 3.2. Silicon tube

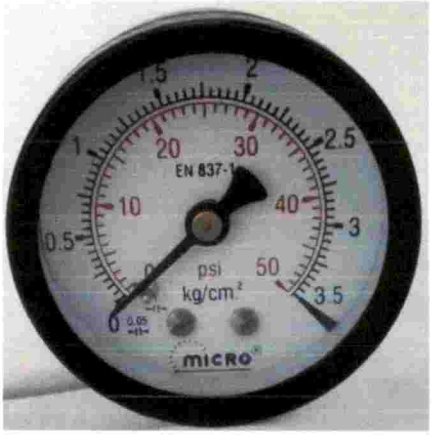


Plate 3.3. Pressure gauge

Plate 3.4. Cartridge



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### 3.1.3 Cartridge

The cartridge made up of microwavable glass was designed according to the internal dimensions of microwave oven cavity for the ease in inserting and removing while loading and unloading the plant material. It has an outside diameter of 100 mm and volume of 80 ml. Cartridge has opening at top and bottom. Top neck has 25 mm length, with 32 mm outside diameter opening. The end of the top neck has conical ground glass joint (25 mm) which can accept any similarly-sized tapered fittings. Bottom stem has 65 mm length, with 11 mm outside diameter opening (Plate 3.4.).

Silicon tube carrying the steam from steam generator, was connected to the bottom stem of cartridge through a hole drilled on the bottom (13 mm diameter) of the microwave cavity.

### 3.1.4 Extraction Unit

Extraction unit, placed outside the microwave oven, consists of clevenger apparatus and condenser. Clevenger apparatus which comprises of 10ml graduated receiver with stopcock, is a tool for essential oil extraction using steam. Condenser which act as a cooling system, condense the distillate continuously and thus separates the essential oil (Plate 3.5.).

A hole was drilled on the top (½ inch diameter) of the microwave cavity in order to fix the condenser into the cartridge which was positioned in the cavity. The cartridge and condenser was connected by means of a glass tube (12 mm x 100 mm) and two silicon rubber corks. A part of glass tube is projected upwards and remaining part is inside the oven cavity. The glass tube acts as carrier of both water and essential oil vapours.

### 3.1.5 Supporting Stand

The supporting stand is placed outside the oven for supporting the extraction unit. The stand made up of a stainless steel ring and three supporting legs. The circular ring has an outer diameter of 63 mm and inner diameter of 60.5 mm. The

height of the circular ring is 70 mm. The three legs each of length 54 mm are welded to the circular ring. A rubber cork was inserted into the circular ring to keep the distillation unit stable. The inner and outer diameters of the cork are 21 mm and 60 mm, respectively with a cork length of 28 mm. The supporting stand is shown in Plate 3.6.

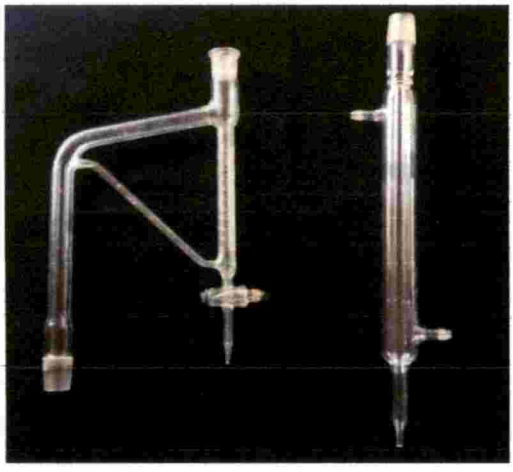

Plate 3.5. Extraction unit

Plate 3.6. Supporting stand

Plate 3.7. Energy meter

Plate 3.8. Temperature sensor and controller

### 3.1.6 Energy Meter

A single phase electronic energy meter (SPEM 01; 240V; 50 Hz) was connected to the microwave reactor to measure the energy consumed during the distillation process (Plate 3.7.). The energy consumed for microwave steam

distillation and for steam distillation without microwave were measured for comparison of the energy efficiency.

### 3.1.7 Temperature Sensor and Controller

A temperature sensor was inserted into the microwave cavity to measure the temperature of extraction. A controller (SELEC TC203AX) connected to the sensor for reading the temperature, was placed outside the microwave oven (Plate 3.8.).

To support the whole system developed, a stand (0.43 x 0.298 x 1.05 m) was fabricated out of mild steel hollow section. The top of the stand is covered by stainless steel plate on which a hole is drilled in order to connect the steam tube with cartridge.

## 3.2 EXPERIMENTAL DESIGN

The process parameters which would influence the essential oil yield, energy consumption and temperature of extraction were chosen as independent variables on the basis of thorough review of literature and the preliminary studies conducted. The physical quality characteristics which are characteristics of these parameters were taken as dependent variables.

### 3.2.1 Independent Variables

a) Bulk density (g $cm^{-3}$):

1) $A_1$ : 0.375
2) $A_2$ : 0.625
3) $A_3$ : 0.875

b) Microwave power (W):

1) $P_1$ : 280
2) $P_2$ : 420
3) $P_3$ : 560

c) Time of extraction (min):

1) $t_1$ : 30
2) $t_2$ : 40

3) $t_3$ : 50

### 3.2.2 Dependent Variables

**Microwave steam distillation system output parameters:**

a) Essential oil yield

b) Energy consumption

c) Temperature of extraction

**Physical quality characteristics of lemongrass essential oil:**

a) Colour

b) Refractive index

c) Specific gravity

d) Solubility

## 3.3 EXPERIMENTAL PROCEDURE

Fresh lemongrass (*Cymbopogon flexuosus*) (Plate 3.9.) collected from Aromatic and Medicinal Plants Research Station, Odakkali, Ernakulum District, Kerala and stored at 2°C till the commencement of experiment was used to evaluate microwave steam distillation process in the developed system. The detailed procedure for microwave steam distillation of lemongrass essential oil is detailed below.

### 3.3.1 Extraction of Essential Oil

The steam generator used was filled half with water, closed and heated by LPG. The lemongrass cut into small size in required quantity as per the experimental design, was filled in the cartridge of the system (Plate 3.10.). The cartridge was placed inside the microwave reactor. One end of clevenger apparatus is connected to the cartridge via glass tube and the other end to the condenser. The microwave power level and time of exposure was set in the control panel of microwave reactor for various treatment conditions. Steam from steam generator regulated by the steam control valve, was allowed to pass through the cartridge via silicon tube. Microwaves at preset power level, heat the plant material for the set time interval. Essential oil in lemongrass gets vaporised and passes out of the microwave cavity along with steam,

through the distillation stem into the condenser. These vapors get condensed and fall into the stem of the clevenger apparatus where the oil and water get separated due to density difference. Oil sets collected as the top layer since it is lighter than water. After completion of the process the water was drained off by opening the valve and the oil was collected. Anhydrous sodium sulphate was added to essential oil for removal of moisture, and then stored in amber coloured glass bottles at 2°C (Plate 3.11.) for further analysis.


Plate 3.9. Lemongrass (*Cymbopogen flexuous*)

Plate 3.10. Sample

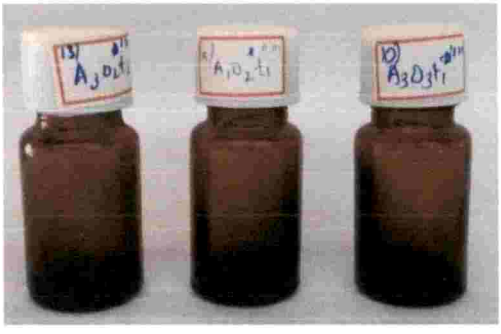

Plate 3.11. Amber coloured glass bottles

Steam distillation was also performed without microwave power for a vigorous comparison with microwave steam distillation process. The experiment was continued until complete extraction of essential oil from plant material was obtained.

The results of this extraction process was explained with that of microwave steam distillation.

## 3.4 DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS OF ESSENTIAL OIL

### 3.4.1 Specific Gravity

Specific gravity was determined by dividing the weight of one ml essential oil by the weight of one ml distilled water. Weights were calculated utilising a balance with an accuracy of 0.001g (Gopika and Ghuman, 2014).

### 3.4.2 Refractive Index

The refractive index of the lemongrass essential oil was determined using reflected system of Newton's rings.The refractive index of a transparent liquid was determined by the method of reflected system of Newton's rings. Newton's rings apparatus, Sodium vapour lamp, and Vernier microscope were used for the experiment (Plate 3.12.). The Newton's rings apparatus consists of an optically plane glass plate P and a convex lens L of large focal length, placed on the glass plate. Above the lens, another glass plate G is arranged at 45° to the horizontal. When the lens is placed over the glass plate, the space between the lens and glass plate contain air.

If $D_m$ and $D_{m+k}$ be the diameters of the $m^{th}$ and $(m+k)^{th}$ dark rings respectively, then,

$$D_m^2 = 4mR\lambda$$

$$D_{m+k}^2 = 4(m+k)R\lambda$$

$$D_{m+k}^2 - D_m^2 = 4kR\lambda$$

where $\lambda$ :the wavelength of the light utilised and R :the radius of curvature of the lens. With a thin film of the transparent liquid between the lens and the glass plate, if $D_m'$ and $D_{m+k}'$ are the diameters of the $m^{th}$ and $m+k^{th}$ rings,

$$D_{m+k}'^2 - D_m'^2 = 4KR\lambda/n$$

where n: refractive index of the liquid

From the two equations,

$$n = D_{m+k}^2 - D_m^2 / D_{m+k}'^2 - D_m'^2 \ldots. (3.1)$$

Light from a sodium vapour lamp (S) is rendered parallel by a short focus convex lens. The parallel rays fall on the glass plate (G), inclined at 45° to the horizontal, gets reflected and then fall normally on the convex lens (L) kept above the glass plate (P). A system of bright and dark concentric circular rings are detected via a microscope (M), arranged vertically above the glass plate (G) as shown in Plate 3.13. The microscope is properly focused so that alternate bright and dark circular rings are seen clearly (Plate 3.14.). Make sure that there are about 25 clear rings on either side of the centre by using fine adjustment screw of the microscope. The microscope is moved towards the left, starting from the centre of the fringe system, so that the cross-wire is tangential to the $m^{th}$ (say 20th) dark ring. The microscope reading on the horizontal scale was taken. Then, by working the fine adjustment screw, the microscope was moved towards the right. The cross-wire was adjusted to be tangential to the 18th, 16th etc. dark ring, up to the second dark ring on the left and the corresponding readings were taken. Then the cross-wire was made tangential to the second dark ring on the right side. Readings were taken corresponding to the 2nd, 4th .... 20th dark ring, as before. The difference between the readings on the left and right of each ring gives the diameter D of the respective ring. Hence $(D_{m+k}^2 - D_m^2)$ was calculated.

A drop of liquid was placed on the plane glass plate and the lens was placed over it. The lens and the glass plate were pressed together so that a thin film of liquid without any air bubble is formed between them. The experiment was repeated as before and $D_m'$ and $D_{m+k}'$ were measured. The refractive index of the liquid was then calculated.

Refractive index (n) = Mean of $(D_{m+k}^2 - D_m^2)$ / Mean of $(D_{m+k}'^2 - D_m'^2)$ .... (3.2)

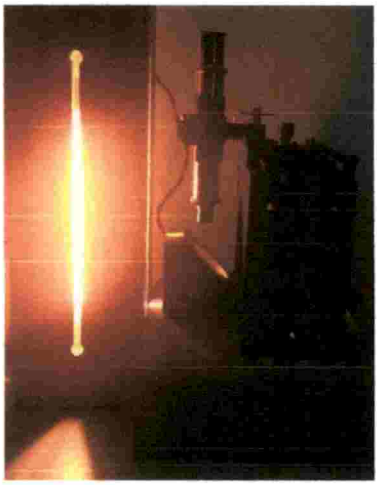

Plate 3.12. Newton's ring apparatus

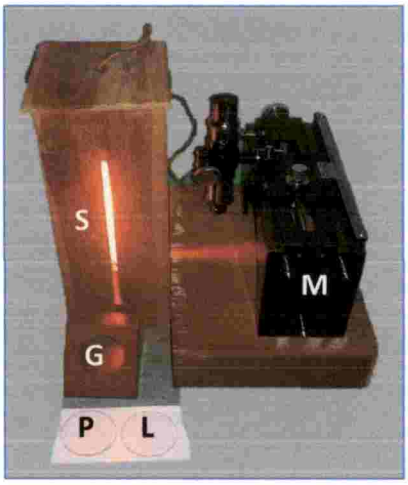


Plate 3.13. Newton's ring experiment set up

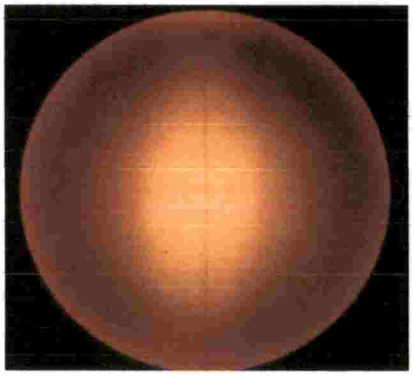

Plate 3.14. Circular rings

### 3.4.3 Solubility

The solubility of lemongrass essential oil was determined based on the procedure suggested by Food Chemical Codex (FCC, 1996). One ml sample of lemongrass essential oil was transferred into a calibrated 10 ml glass stoppered cylinder graduated in 0.1 ml divisions. The oil was then diluted with 0.1 ml of 85 per cent (v/v) ethanol repeatedly each time. The temperature was maintained at 25° C, and the contents mixed thoroughly after each addition of alcohol. The dilution procedure was continued till a clear mixture observed. The volume of alcohol (V) utilised to obtain a completely clear solution was noted. Once the clear solution was

obtained, the dilution process was continued, but with 0.5 ml 85 per cent ethanol til the volume of alcohol added was 20 times to the volume added earlier. The solution was shaken well each time with 0.5 ml ethanol until no turbidity was observed. The results were recorded as ''one volume of essential oil soluble in V volumes or more of 85% ethanol".

### 3.4.4 Colour

The colour of the lemongrass essential oil was found using a Hunter lab colour flex meter (Hunter Association laboratory, Inc., Reston, Virgina, USA; model: HunterLab's ColourFlex EZ) (Plate 3.15.).

Plate 3.15. Hunterlab colourflex meter

The Hunter lab's colour flex spectro calorimeter consists of measurement (sample) port, opaque cover and display unit. This colour flex meter operates on the theory of focusing the light and measuring energy reflected from the sample across the entire visible spectrum. For matching a sequence of colour across the visible spectrum, primary lights are required and describes the colour by mathematical model called as Hunter model. It reads the colour of sample in respect of L*, a* and b* values where, luminance (L) forms the vertical axis, which denotes whiteness to darkness. Chromatic portion of the solids is designated by: redness a (+), greenness a (-), yellowness b (+), and blueness b (-). A transparent glass cup filled with sample was placed over the port of the instrument and an opaque cover which act as a light

trap to exclude the interference of external light was placed over the cup. Before actual measurements colour was calibrated by fixing the definite colours like white and black tiles. After calibration, the sample was placed over the port and values of 'L*', 'a*' and 'b*' were recorded.

## 3.5 STATISTICAL ANALYSIS

Response surface methodology (RSM) was chosen for the design of experimental combinations. This method is based on the multivariate non-linear model which is widely used for optimisation process. It is helpful to study the interactions of the various parameters that affect the process. The main advantage of RSM is reducing the number of experimental runs needed for providing sufficient information about statistically acceptable results (Montgomery, 2001). The independent variables considered were bulk density ($X_1$), microwave power ($X_2$) and time of extraction ($X_3$). The three levels of the process variables were coded as -1, 0 and +1. The values of independent variables at three levels were shown in Table 3.1.

Table 3.1 Values of independent variables at three levels of Box–Behnken design

| **Independent variable** | **Symbol** | | **Level** | |
|---|---|---|---|---|
| | **Coded** | **Uncoded** | **Coded** | **Uncoded** |
| Bulk density (g $cm^{-3}$) | $X_1$ | A | -1 | 0.375 |
| | | | 0 | 0.625 |
| | | | +1 | 0.875 |
| Microwave power (W) | $X_2$ | P | -1 | 280 |
| | | | 0 | 420 |
| | | | +1 | 560 |
| Time of extraction (min) | $X_3$ | T | -1 | 30 |
| | | | 0 | 40 |
| | | | +1 | 50 |

Table 3.2. Experimental design used for extraction of MSD of lemongrass essential oil

| Standard Order | Run | Coded variables | | | Un-coded variables | | |
|---|---|---|---|---|---|---|---|
| | | Bulk density ($g\ cm^{-3}$) | Microwave power (W) | Time of extraction (Min) | Bulk density ($g\ cm^{-3}$) | Microwave power (W) | Time of extraction (Min) |
| 1 | 4 | -1 | -1 | 0 | 0.375 | 280 | 40 |
| 2 | 5 | 1 | -1 | 0 | 0.875 | 280 | 40 |
| 3 | 9 | -1 | 1 | 0 | 0.375 | 560 | 40 |
| 4 | 2 | 1 | 1 | 0 | 0.875 | 560 | 40 |
| 5 | 7 | -1 | 0 | -1 | 0.375 | 420 | 30 |
| 6 | 17 | 1 | 0 | -1 | 0.875 | 420 | 30 |
| 7 | 8 | -1 | 0 | 1 | 0.375 | 420 | 50 |
| 8 | 16 | 1 | 0 | 1 | 0.875 | 420 | 50 |
| 9 | 1 | 0 | -1 | -1 | 0.625 | 280 | 30 |
| 10 | 11 | 0 | 1 | -1 | 0.625 | 560 | 30 |
| 11 | 3 | 0 | -1 | 1 | 0.625 | 280 | 50 |
| 12 | 14 | 0 | 1 | 1 | 0.625 | 560 | 50 |
| 13 | 6 | 0 | 0 | 0 | 0.625 | 280 | 40 |
| 14 | 10 | 0 | 0 | 0 | 0.625 | 280 | 40 |
| 15 | 13 | 0 | 0 | 0 | 0.625 | 280 | 40 |
| 16 | 15 | 0 | 0 | 0 | 0.625 | 280 | 40 |
| 17 | 12 | 0 | 0 | 0 | 0.625 | 280 | 40 |

The experiments were designed using Design Expert Software, Version 7.7.0 (State-Ease, Minneapolis, MN). The same software was used for statistical analysis of

experimental data. According to Box-Behnken design for three independent factors, the total experiments to be conducted are found to be seventeen. Seventeen experiments were performed with three variables as shown in Table 3.2.

## 3.6 SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy (SEM) was used to investigate the morphological features of the lemongrass (Firdaus *et al.*, 2016). SEM can produce a largely magnified image by using electrons. Scanning electron microscopy of cross-sections of lemongrass samples before and after extraction (MSD and SD) of essential oil were carried out using Hitachi SU6600 Variable Pressure Field Emission Scanning Electron Microscope (FESEM) at SEM centre, NIT, Calicut (Plate 3.16.). The samples were first coated with gold since it is non-conducting, by E 1010 ion sputter coating unit (Plate 3.17.) and then examined under SEM. Electron photomicrographs were taken at 10 kV, using an electronic gun (Tungsten Schottky emission electron source) at desired magnifications.

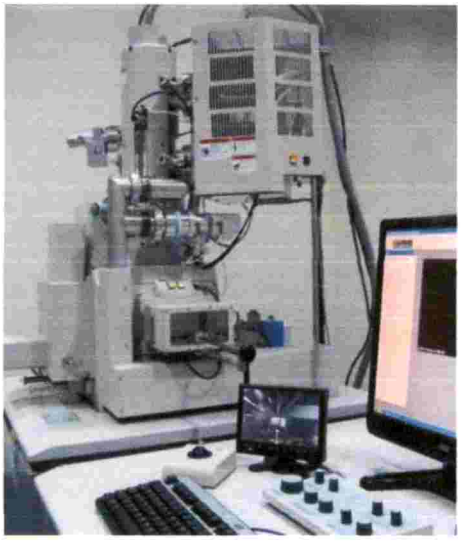

Plate 3.16. Hitachi SU6600 Variable Pressure Field Emission Scanning Electron Microscope (FESEM)

Plate 3.17. Ion sputter coating unit

The main features of SU 6600-FESEM were: Resolution: 1.2 nm/30 kV, 3.0 nm/1 kV, Probe current: 1pA~200nA, Specimen chamber pressure: $10^{-4}$ Pa (high vacuum), 10-300Pa (low vacuum), Specimen Size: Max 150 mm dia. × 40 mm H, Magnification: 500,000 x.

## 3.7 DETERMINATION OF CHEMICAL CONSTITUENTS

The analysis of volatile oil components in the lemongrass essential oil extracted through microwave steam distillation and steam distillation process was carried out using Gas Chromatographic (GC) technique (Model: SHIMADZU GC-17A) (Plate 3.18.). The main aroma compound of the lemongrass essential oil is citral. For determining the quality of the extracted oil in international market, the presence of citral and its amount is taken as a parameter. A Thermo TR-1 with 30 m in length, 0.25 mm inner diameter, and 0.25 μm film thickness column was used for the experiments. A flame injection detector with an operating temperature of 280°C and an injector with temperature of 250°C were used. Nitrogen acts as gas carrier at a flow rate of one ml/min and the split ratio was 1:30. The maximum temperature that can be attained in the equipment was 350°C. The procedure for finding the chemical constituent was adopted from Tyagi and Malik (2010) and Adukwu *et al.* (2012). Citral standard was obtained from Sigma-Aldrich. The standard solution was first injected and the chromatograph of the standard was obtained. Then one μl sample was injected and its chromatograph was recorded following the same procedure. The chromatographs were then analysed for citral content.

Plate 3.18. Gas Chromatography

# Results and discussion

# CHAPTER IV

# RESULTS AND DISCUSSION

This chapter deals with the results on development of a microwave steam distillation system and evaluation of the system developed towards extraction of lemongrass essential oil. The outcomes of the various experiments conducted to optimise the process parameters were discussed in detail and the effect of process variables on physical and chemical characteristics of the microwave steam distilled lemongrass oil in comparison with conventional steam distilled oil were enunciated and explained.

## 4.1 DEVELOPMENT OF MICROWAVE STEAM DISTILLATION SYSTEM FOR LEMONGRASS ESSENTIAL OIL

A microwave steam distillation system for extracting lemongrass essential oil was developed which composed of microwave reactor, steam generator, cartridge, extraction unit, supporting stand, energy meter and temperature sensor and controller as shown in Figure 3.1.

The microwave reactor selected provides a maximum microwave power of 700 W and is supported on the stand fabricated. Steam outlet from steam generator was connected to the cartridge containing lemongrass via silicon tube. Pressure of steam generated was measured using pressure gauge. The extraction unit which mainly comprises of clevenger apparatus and condenser was supported on a stand fabricated for the purpose. The energy consumed for extracting the lemongrass essential oil in microwave steam distillation was measured with the help of a single phase induction type energy meter and the temperature of the extraction was determined using temperature sensor and controller. The oil extracted by microwaves along with steam produced in a steam generator crosses the sample inside the cartridge of the reactor. The plant materials respond differently to the action of microwaves because the heating is through kinetic effects and is a volumetric process. Hence, the process parameters leading to the efficient extraction and quality of oil needs to be standardised.

## 4.2 STANDARDISATION OF THE PROCESS PARAMETERS OF THE MICROWAVE STEAM DISTILLATION SYSTEM

As per the experimental design a series of experiments were performed to evaluate the system developed towards extraction of lemongrass essential oil and to optimise the process parameters. The three levels of bulk densities (0.375, 0.625 and 0.875 g $cm^{-3}$), microwave power (280, 420 and 560 W) and time of extraction (30, 40 and 50 min) was employed for the experiments as input variables.The experiments were performed as per the methodology described in Chapter III under the section 3.3. The process of microwave steam distillation for the extraction of lemongrass essential oil was then standardised. The results of the experiments conducted towards the microwave steam distillation with mean values of essential oil yield, energy consumption and temperature of extraction are tabulated in Table 4.1.

The parameters were optimised through the optimisation process using Box-Behnken method of response surface methodology. Seventeen experimental data were used in the design to optimise the parameters as per response surface methodology. Design Expert (Trial version 7.0.0, STAT-EASE Inc.), a statistical software was used to analyse the experimental data, for analysis of variance (ANOVA), regression coefficient calculations and for graphical analysis (response surfaces) of the experimental data.

In order to relate the independent process variables, a second order quadratic model was used. In the second order polynomial equation, the coefficient of each term was determined using a multiple regression analysis in Design Expert software. Regression coefficients were obtained when experimental data was fitted to the selected models. Analysis of variance (ANOVA) was employed for statistical significance of the terms in the regression equation and thus to determine the adequacy of the quadratic model. To examine the impact of each of the coefficients and to understand the mutual interactions between test variables 'p' values were used. The corresponding coefficient is more significant when 'p' values are smaller in magnitude

($p < 0.05$). The adequacy of regression model was checked by $R^2$, Adjusted $R^2$, Adequate Precision and Fisher's F-test (Montgomery, 2001).

Table 4.1. Effect of process variables towards extraction of lemongrass essential oil

| Sl. No. | Sample | Essential oil yield (ml) | Energy consumption (kWh) | Temperature of extraction (°C) |
|---|---|---|---|---|
| 1. | $A_1 P_1 t_2$ | 0.25 | 1.25 | 56 |
| 2. | $A_3 P_1 t_2$ | 0.40 | 1.25 | 55 |
| 3. | $A_1 P_3 t_2$ | 0.30 | 1.45 | 71 |
| 4. | $A_3 P_3 t_2$ | 0.45 | 1.45 | 68 |
| 5. | $A_1 P_2 t_1$ | 0.40 | 1.10 | 67 |
| 6. | $A_3 P_2 t_1$ | 0.40 | 1.10 | 61 |
| 7. | $A_1 P_2 t_3$ | 0.25 | 1.70 | 65 |
| 8. | $A_3 P_2 t_3$ | 0.50 | 1.70 | 64 |
| 9. | $A_2 P_1 t_1$ | 0.35 | 1.10 | 58 |
| 10. | $A_2 P_3 t_1$ | 0.45 | 1.10 | 73 |
| 11. | $A_2 P_1 t_3$ | 0.35 | 1.60 | 56 |
| 12. | $A_2 P_3 t_3$ | 0.40 | 1.80 | 75 |
| 13. | $A_2 P_2 t_2$ | 0.30 | 1.35 | 65 |
| 14. | $A_2 P_2 t_2$ | 0.35 | 1.38 | 66 |
| 15. | $A_2 P_2 t_2$ | 0.30 | 1.35 | 66 |
| 16. | $A_2 P_2 t_2$ | 0.35 | 1.38 | 65 |
| 17. | $A_2 P_2 t_2$ | 0.35 | 1.35 | 66 |
| 18. | C | 0.30 | 4.68 | 80 |

Adjusted $R^2$ is a measure of the amount of variation around the mean, adjusted for the number of terms in the model. As the number of terms in the model increases,

the adjusted $R^2$ decreases if those additional terms do not increase value to the model. Adequate precision relates the range of predicted values at design points to the normal prediction error. The F-value at probability (p) of 0.1 to 0.01 provided the importance of all polynomial terms statistically. A complete second order quadratic model was utilised to fit the data. $R^2$, Adjusted $R^2$, predicted $R^2$ and Fischer F-test were used for testing of adequacy of the model (Haber and Runyon, 1977). During the explanation of variation in behavior the smaller the value of $R^2$, the less importance the dependent variables in the model have. Partial differentiation of the process parameters was done to optimise the model with respect to each parameter. The resulting function is solved by equating the equation to zero. Statistical calculation was also carried out using the regression coefficients to create three-dimensional plots for the regression models.

## 4.3 EFFECT OF PROCESS PARAMETERS ON OUTPUT CHARACTERISTICS OF MICROWAVE STEAM DISTILLATION SYSTEM

### 4.3.1 Essential Oil Yield

The essential oil yield of lemongrass obtained in various combinations of experiments are shown in Table 4.1. The total yield of oil varied from 0.25 to 0.5 ml. The maximum oil yield was obtained for a bulk density of 0.875g $cm^{-3}$, microwave power of 420 W and extraction time of 50 min.

A second order non-linear regression equation was utilised to relate between dependent and independent variables using the experimental values. Following regression model was obtained to predict the yield (ml) of lemongrass essential oil.

$$\text{Total yield of essential oil} = 0.33 + 0.081A + 0.031B + 0.015C + 0.005\,AB + 0.037\,AC - 0.012\,BC - 0.025\,A^2 + 0.023\,B^2 + 0.035\,C^2 \quad \ldots..4.1$$

Where A: Bulk density (g $cm^{-3}$)

B: Microwave power (W)

C: Time of extraction (min)

It is evident from Equation (4.1) that the total yield of oil was in positive correlation with bulk density, microwave power and time of extraction.

The ANOVA table for the response "essential oil yield" is shown in Appendix A (Table A.1). From Table A.1, it is inferred that the values of R- Squared, Adj R-Squared and Pred R-Squared for the total yield of oil were 93.86, 85.97 and 56.32% respectively. Table A.1 shows that, process parameters had a significant effect on total yield of oil at one per cent ($p<0.001$) level of significance.

The coefficient of determination ($R^2$) of the regression model for oil yield was 93.86% which implies that the model could account 93.86% variability in data. The Pred R Squared (56.32%) is in reasonable agreement with the Adj - R Squared (85.97%). Lack of fit was insignificant and F-value suggested that the model was significant at one per cent and five per cent level of significance. The adequate precision (12.043) value for total yield of oil indicates that the model can be used to predict the response within the design space. Therefore, second order model was adequate in describing the total yield of MSD essential oil.

The relationship between bulk density, microwave power, and time of extraction on total yield of essential oil is illustrated by plotting 3D graphs representing the response surface generated by the model (Equation. 4.1). The 3D responses were shown in Figure 4.1.

From Figure 4.1., it can be inferred that bulk density has significant effect on essential oil yield. An increase in yield of essential oil was observed with an increase in the bulk density. Microwave incident per particle lowered at a fixed power with increment in bulk density that caused in less dielectric heating and as a result effect of microwave radiation was decreased. For the enhanced bulk density, long extraction time may be necessary to achieve the similar level of extraction (Thakker *et al.*, 2016).

Figure 4.1 shows that microwave power has a significant effect on total yield of essential oil. In general, the extraction was improved by raising the microwave power from 280 W to 420 W. At a low microwave power (280 W) the essential oil yield was found to be less. This trend might be due to the temperature being not enough to burst open the oil glands. With increase in microwave power, total yield of essential oil increased to a maximum of 0.5 ml at a microwave power of 420 W. A decline in

the yield of essential oil happened with increment in microwave power beyond 420 W. As per the graphs, the essential oil yield increased initially when microwave power increased. This occurs due to the rapid heat production inside lemongrass with the absorption of microwave energy and the subsequent formation of a higher pressure gradient inside the plant material when subjected to increase in microwave power levels (Abdurahman *et al.*, 2013).

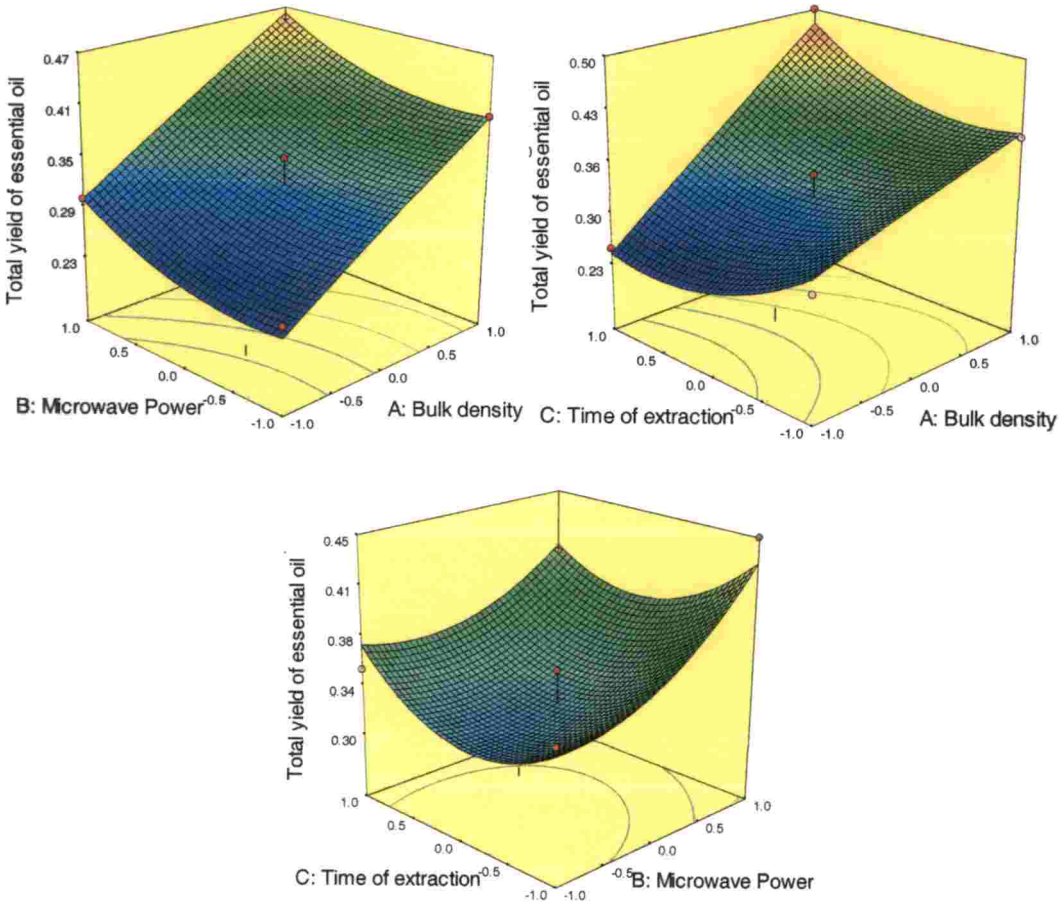


Figure 4.1. Effect of process parameters on total yield of essential oil

Microwave steam distillation provided yield of 0.3 to 0.5% within 30 to 50 min when most of the oil is extracted. Increase in extraction time leads to an increase in the yield of essential oil up to a period of 50 min. But, further increase in extraction time did not improve the extraction performance. Sahraoui *et al.* (2011) reported similar

observations for MSD. The extraction was comparatively slow at the beginning but gradually improved with time. The plant material when exposed to the heat result in breaking of plant cells and the essential oil is released to the environment. At the same time prolonged extraction time produced over heating of the plant material which leads to the vaporisation of the volatile component in the essential oil (Chemat *et al.*, 2006; Vian *et al.*, 2008; Gavahian *et al.*, 2015).

MSD provided an essential oil yield within 30 min of extraction time whereas SD provided this yield after 180 min. When compared with steam distillation without microwave power, the total yield of essential oil obtained in both the process were almost similar (Song *et al.*, 2012; Kusuma *et al.*, 2016). In both process total yield of 0.3-0.4% was observed.

### 4.3.2 Total Energy Consumption

Total energy consumption of lemongrass essential oil extraction obtained in various combinations of experiments are shown in Table 4.1. The energy consumption varied between 1.1 and 1.8 kWh. The least energy consumption was obtained for a time of 30 min of extraction.

A second order non-linear regression equation described the relation between dependent and independent variables. Following regression model was obtained to predict the energy consumption for microwave steam distillation of lemongrass essential oil.

$$\text{Total energy consumption} = 1.36 + 0.005\,A + 0.075\,B + 0.30\,C + 0.003\,AB + 0.002\,AC + 0.050\,BC - 6.000E\text{-}003\,A^2 - 6.000E\text{-}003\,B^2 + 0.044C^2 \quad \ldots\ldots(4.2)$$

Where A: Bulk density (g $cm^{-3}$)

B: Microwave power (W)

C: Time of extraction (min)

It is clear from Equation (4.2) that the energy consumption was in positive correlation with bulk density, microwave power and time of extraction.

The ANOVA table for the response "Total energy consumption" is shown in Appendix A (Table A.2.). From Table A.2, it may be inferred that the values of R-Squared, Adj R-Squared and Pred R-Squared for the total energy consumption were 99.23, 98.24 and 89.65%, respectively. The coefficient of determination ($R^2$) of the regression model for total energy consumption was 99.23% which implies that the model could account 99.23% variability in data. The Pred R-Squared (89.65%) is in reasonable agreement with the Adj R-Squared (98.24%). The adequate precision (33.18) value for total energy consumption indicates that the model can be used to predict the response within the design space. Therefore, second order model is adequate in describing the total energy consumption of MSD essential oil.

The relationship between total energy consumption and independent variables are illustrated by plotting 3D graphs representing the response surface generated by the model (Equation. 4.2). The 3D responses were shown in Figure 4.2.

It may be perceived from the Figure 4.2. that total energy consumption increases with increase in microwave power and time of extraction. The total energy consumption varied between 1.1 kWh and 1.8 kWh for microwave steam distillation. Energy consumption for steam distillation without microwave power was found to be 4.68 kWh. Bulk density has less significant effect on total energy consumption than that of other independent variables.

By comparing with steam distillation without microwave power, the total energy consumed for extracting lemongrass essential oil through microwave steam distillation was found to be very less. Therefore, it could be inferred that microwave steam distillation was superior in terms of energy savings.

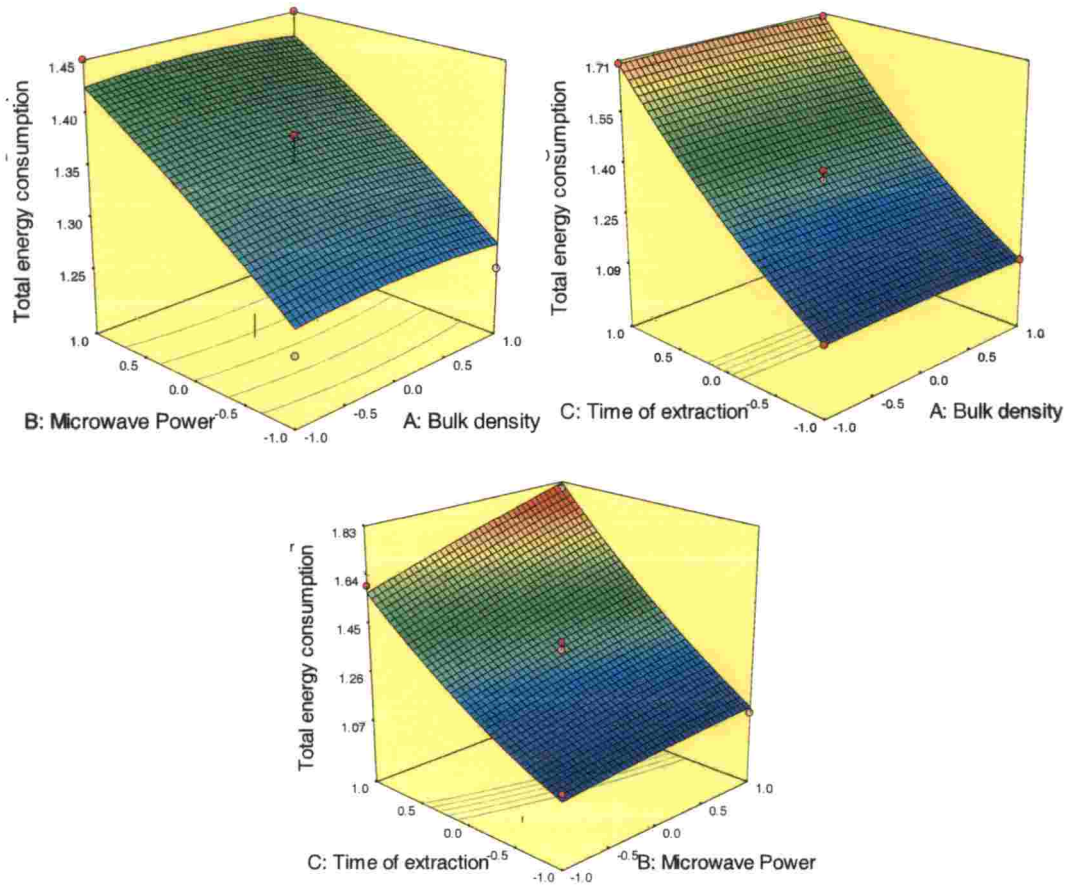


Figure 4.2. Effect of process parameters on total energy consumption

### 4.3.3 Temperature of Extraction

The temperature of extraction measured in various combinations of experiments are shown in Table 4.1 which ranges from 55 to 75°C. The minimum temperature of 55°C was obtained for a bulk density of 0.875 g $cm^{-3}$, microwave power of 280 W and extraction time of 40 min. The maximum temperature was found for a bulk density of 0.625 g $cm^{-3}$, microwave power of 560 W and extraction time of 50 min. The second order non-linear regression equation was used to relate the dependent and independent variables using the experimental values. To predict the temperature of extraction of lemongrass essential oil following regression model was obtained.

$$\text{Temperature of extraction} = 65.60 - 1.37\,A + 7.75\,B + 0.13\,C - 0.50\,AB + 1.25\,AC + 1.00\,BC - 2.18\,A^2 - 0.93\,B^2 + 0.83C^2 \quad \ldots\ldots(4.3)$$

Where A: Bulk density (g $cm^{-3}$)

B: Microwave power (W)

C: Time of extraction (min)

It can be understood from the Equation (4.3) that the temperature of extraction was in positive correlation with microwave power and time of extraction and in negative correlation with bulk density.

The ANOVA table for the response "Temperature of extraction" is given in Appendix A (Table A.3). From Table A.3, it can be concluded that the values of $R^2$, Adj $R^2$ and Pred $R^2$ for temperature of extraction were 98.71, 97.06 and 82.62%, respectively. The coefficient of determination ($R^2$) of the regression model for total time of extraction was 98.71% which implies that the model could account 98.71% variability in data. The $R^2$-pred (82.62%) is in reasonable agreement with the $R^2$-adj (97.06%). Lack of fit was insignificant and F-value suggested that the model was significant at one per cent and five per cent level of significance. The adequate precision (26.825) value for total time of extraction indicates that the model can be utilised to predict the response within the design space as it is greater than four (Montgomery, 2001). Therefore, second order model was adequate in describing the temperature of extraction of microwave steam distillation of essential oil. Table A.3 shows that, process parameters had a significant effect on temperature of extraction at one per cent ($p<0.001$) level of significance with F- calculated value (59.68).

The relationships of bulk density, microwave power and time of extraction with that of the response 'temperature of extraction' are illustrated by plotting 3D surface graphs generated by the model (Equation. 4.3). The 3D responses were shown in Figure. 4.3.

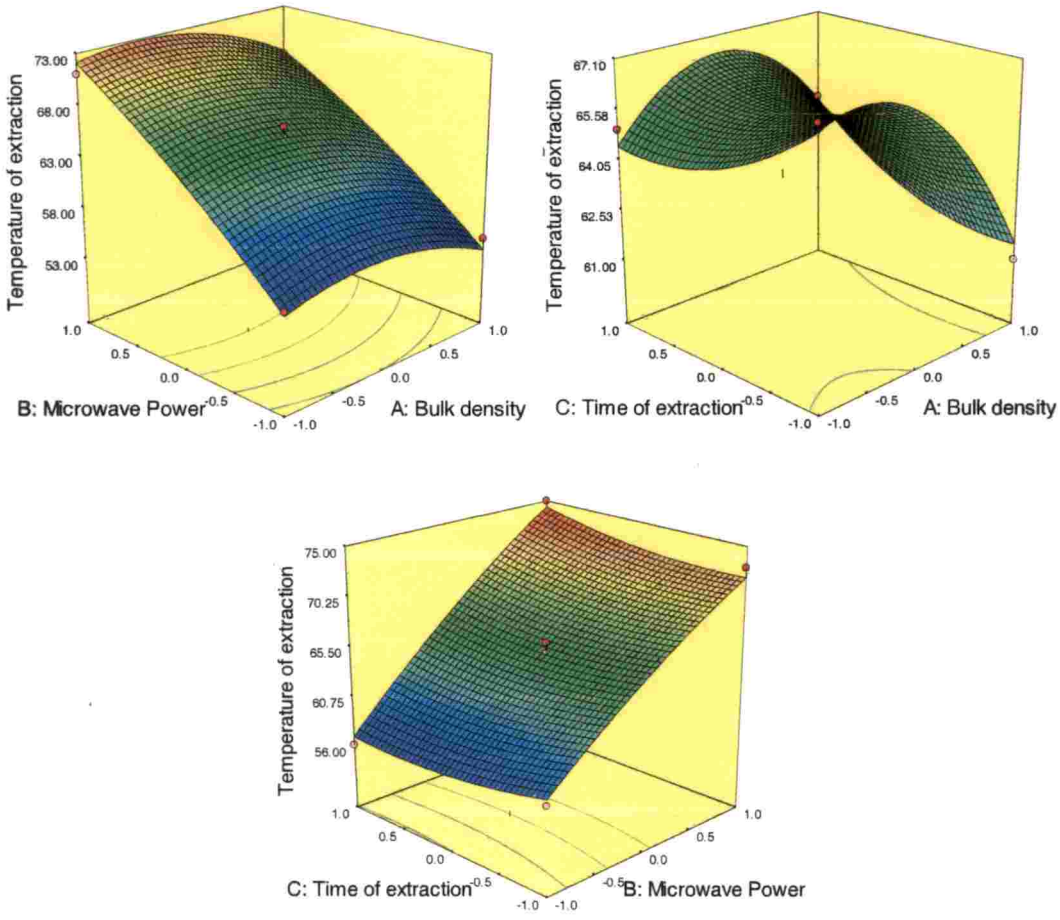


Figure 4.3. Effect of process parameters on temperature of extraction

It can be clearly observed from Figure 4.3. that increase in microwave power increases temperature of extraction. This might be due to the enhancement of heat generation with increase in microwave power. When compared with steam distillation without microwave power, the maximum temperature for microwave steam distillation was observed to be 75°C. Figure 4.3. shows that increment in time of extraction enhances temperature of extraction due to prolonged exposure of plant material to microwave irradiation. It can be concluded from the Equation 4.3. that bulk density had negative correlation on temperature of extraction. This was also supported by surface plot in Figure. 4.3. from which it can be depicted that raise in bulk density leads to reduction in temperature of extraction.

## 4.4 DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS OF THE LEMONGRASS ESSENTIAL OIL

The physical quality characteristics of the lemongrass essential oil extracted using steam distillation (SD) method without microwave power and microwave steam distillation (MSD) method are listed in Table 4.2.

Table 4.2. Physical quality characteristics of lemongrass essential oil

| Sl. No. | Sample | Specific gravity | Refractive Index | Solubility (v/v) | L* | a* | b* |
|---|---|---|---|---|---|---|---|
| 1. | $A_1 P_1 t_2$ | 0.96 | 1.42 | 1.82 | 23.40 | 2.59 | 15.06 |
| 2. | $A_3 P_1 t_2$ | 0.89 | 1.46 | 2.02 | 18.10 | 2.10 | 15.07 |
| 3. | $A_1 P_3 t_2$ | 0.95 | 1.48 | 2.22 | 22.10 | 2.29 | 12.56 |
| 4. | $A_3 P_3 t_2$ | 0.95 | 1.45 | 2.52 | 27.30 | 2.09 | 12.58 |
| 5. | $A_1 P_2 t_1$ | 0.89 | 1.50 | 2.02 | 19.20 | 2.43 | 12.43 |
| 6. | $A_3 P_2 t_1$ | 0.94 | 1.49 | 2.52 | 25.20 | 2.12 | 15.98 |
| 7. | $A_1 P_2 t_3$ | 0.93 | 1.41 | 2.02 | 20.30 | 2.49 | 8.20 |
| 8. | $A_3 P_2 t_3$ | 0.90 | 1.47 | 3.02 | 16.20 | 2.15 | 14.25 |
| 9. | $A_2 P_1 t_1$ | 0.87 | 1.49 | 2.02 | 23.70 | 2.50 | 12.32 |
| 10. | $A_2 P_3 t_1$ | 0.95 | 1.40 | 3.02 | 29.95 | 2.00 | 8.71 |
| 11. | $A_2 P_1 t_3$ | 0.91 | 1.42 | 2.52 | 19.77 | 2.50 | 12.42 |
| 12. | $A_2 P_3 t_3$ | 0.87 | 1.48 | 2.22 | 20.48 | 2.46 | 12.80 |
| 13. | $A_2 P_2 t_2$ | 0.93 | 1.52 | 2.02 | 25.15 | 2.38 | 8.51 |
| 14. | $A_2 P_2 t_2$ | 0.92 | 1.54 | 1.92 | 24.04 | 2.79 | 11.82 |
| 15. | $A_2 P_2 t_2$ | 0.93 | 1.51 | 2.22 | 21.88 | 2.75 | 12.36 |
| 16. | $A_2 P_2 t_2$ | 0.92 | 1.52 | 2.22 | 22.38 | 2.71 | 12.07 |
| 17. | $A_2 P_2 t_2$ | 0.93 | 1.54 | 2.02 | 23.93 | 2.73 | 12.32 |
| 18. | C | 0.84 | 1.45 | 2.00 | 16.5 | 1.12 | 6.35 |

### 4.4.1 Specific Gravity

The specific gravity of lemongrass essential oil obtained in various experiments were tabulated in Table 4.2. The values of specific gravity varied between 0.875 and 0.965.

The second order non-linear regression equation best fitted the data infer the changes in specific gravity of essential oil. It was evident from the regression equation values that are tabulated in Table 4.3., that the microwave power had a positive effect, whereas the bulk density and time had a negative effect on specific gravity of essential oil.

The ANOVA table for the response "specific gravity" is given in Appendix B (Table B.1). From Table B.1., it could be inferred that the values of $R^2$, $R^2$-adj and $R^2$-pred for the specific gravity were 89.87, 76.85 and 34.23%, respectively. The coefficient of determination ($R^2$) of the regression model for refractive index was 89.87% which implies that the model could account 89.87% variability in data. Lack of fit was insignificant and F-value suggested that the model was significant at one per cent and five per cent level of significance. The adequate precision (9.209) value for specific gravity indicates that the model can be used to predict the response within the design space. An adequate precision of greater than 4 is a prerequisite for reliable prediction using mathematical models (Montgomery, 2001). Hence, the present quadaratic second order model was best suitable for the determination of changes in specific gravity by the process parameters. Table B.1. shows that, process parameters had a significant effect on the specific gravity at one per cent ($p<0.001$) level of significance.

The relationship between bulk density, microwave power and time of extraction on specific gravity are illustrated by plotting 3D graphs representing the response surface generated by the model. The 3D responses were shown in Figure 4.4.

Figure 4.4. illustrates that microwave power had a significant effect on specific gravity of oil, whereas time and bulk density has an insignificant effect. From Figure 4.4. it may be revealed that specific gravity increases with increase in microwave

power. No significant difference in specific gravity values were observed between the MSD essential oil and steam distilled oil (Mgudu *et al.*, 2012).

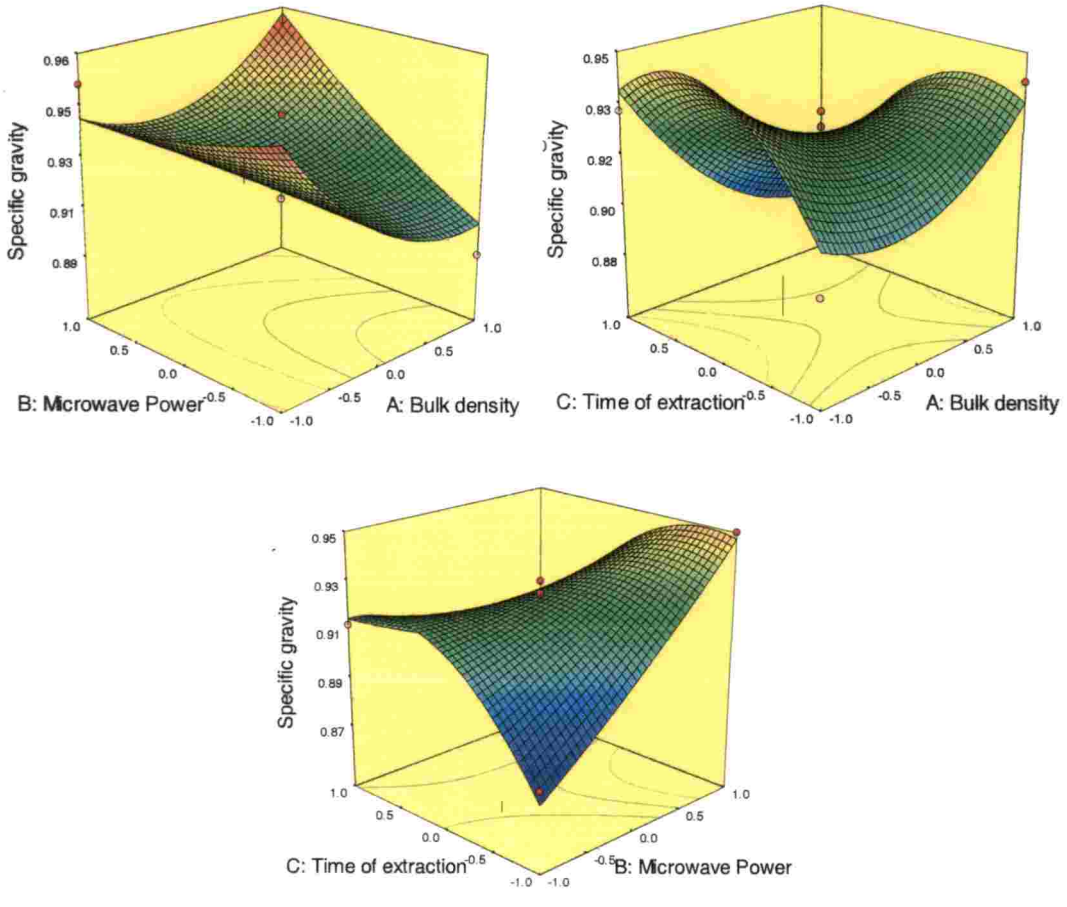


Figure 4.4. Effect of process parameters on specific gravity of essential oil

### 4.4.2 Refractive Index

The values of refractive index of lemongrass essential oil obtained in various experiments are shown in Table 4.2. The values of refractive index varied from 1.404 to 1.540.

A second order non-linear regression equation was used to relate the dependent and independent variables. The regression equation terms tabulated in Table 4.3. illustrates that the refractive index of essential oil is not very much effected by the input variables.

The ANOVA table for the response "refractive index" is given in Appendix B (Table B.2). From Table B.2, it is inferred that the values of $R^2$ and $R^2$-adj for the refractive index were 88.71 and 74.19%, respectively. The coefficient of determination ($R^2$) of the regression model for refractive index was 88.71% which implies that the model could account for 88.71% variability in data. Lack of fit was insignificant and F-value suggested that the model was significant at one per cent and five per cent level of significance. The adequate precision (7.453) value for refractive index indicates that the model can be utilised to predict the response within the design space. Table B.2. shows that, the process parameters had a significant effect on refractive index at one per cent ($p<0.001$) level of significance.

The relationship between bulk density, microwave power and time of extraction on the response 'refractive index' are illustrated by plotting 3D graphs representing the response surface generated by the model. The 3D responses were shown in Figure 4.5.

The time of extraction was found to have negative effect on the refractive index of the essential oil. The microwave power and bulk density have significant effect on the refractive index of the oil. As the microwave power increases, the refractive index of oil has got decreased slightly. This might be due to the raise in temperature which increases speed of light in the medium resulting in lower refractive index values, as the ratio of speed of light in vacuum to the speed of light in medium is refractive index (Anon., 2014). But when compared with the refractive index of steam distilled oil, the refractive index of MSD essential oil also falls within the range. Therefore, the refractive index was found to be similar for essential oil obtained in both the processes (Ma *et al.*, 2012).

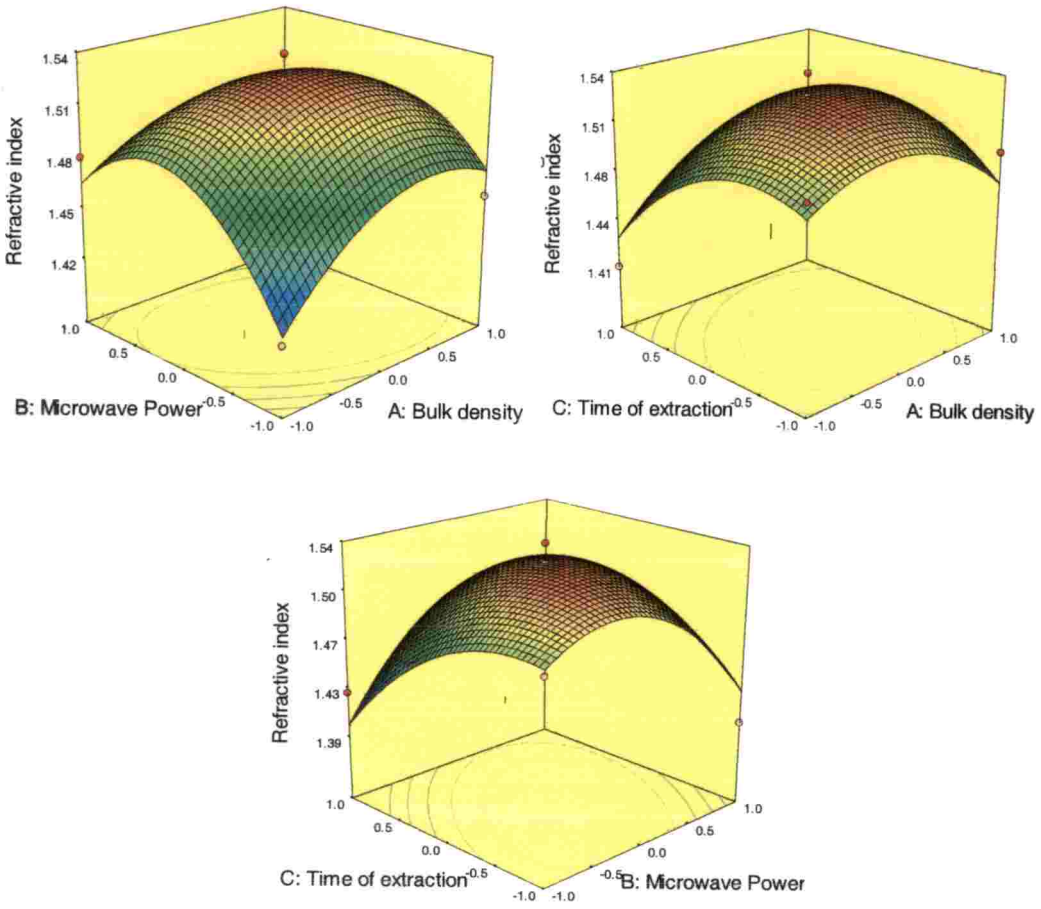


Figure 4.5. Effect of process parameters on refractive index of essential oil

### 4.4.3 Solubility

The solubility of essential oil obtained in various experiments in 85% ethanol were tabulated in Table 4.2. The values of solubility varied between 1.82 and 3.02 v/v. The solubility values obtained in MSD lemongrass essential oil were in close relation with SD lemongrass essential oil.

To correlate the dependent and independent variables, a second order non-linear regression model was generated from the data. It is evident from the regression coefficient of model tabulated in Table 4.3. that the input variables have insignificant effect on solubility of essential oil.

The ANOVA table for the response "solubility" is given in Appendix B (Table B.3). From Table B.3, it is inferred that the values of R- Squared and Adj R-Squared for the solubility were 86.03 and 68.07%, respectively. The coefficient of determination ($R^2$) of the regression model for solubility was 86.03% which implies that the model could account 86.03% variability in data. Lack of fit was insignificant and F-value suggested that the model was significant at one per cent and five per cent level of significance. The adequate precision (7.958) value for solubility indicates that the model can be utilised to predict the response within the design space.

Table B.3. shows that, the process parameters had a significant effect on the solubility at one per cent ($p<0.001$) level of significance.

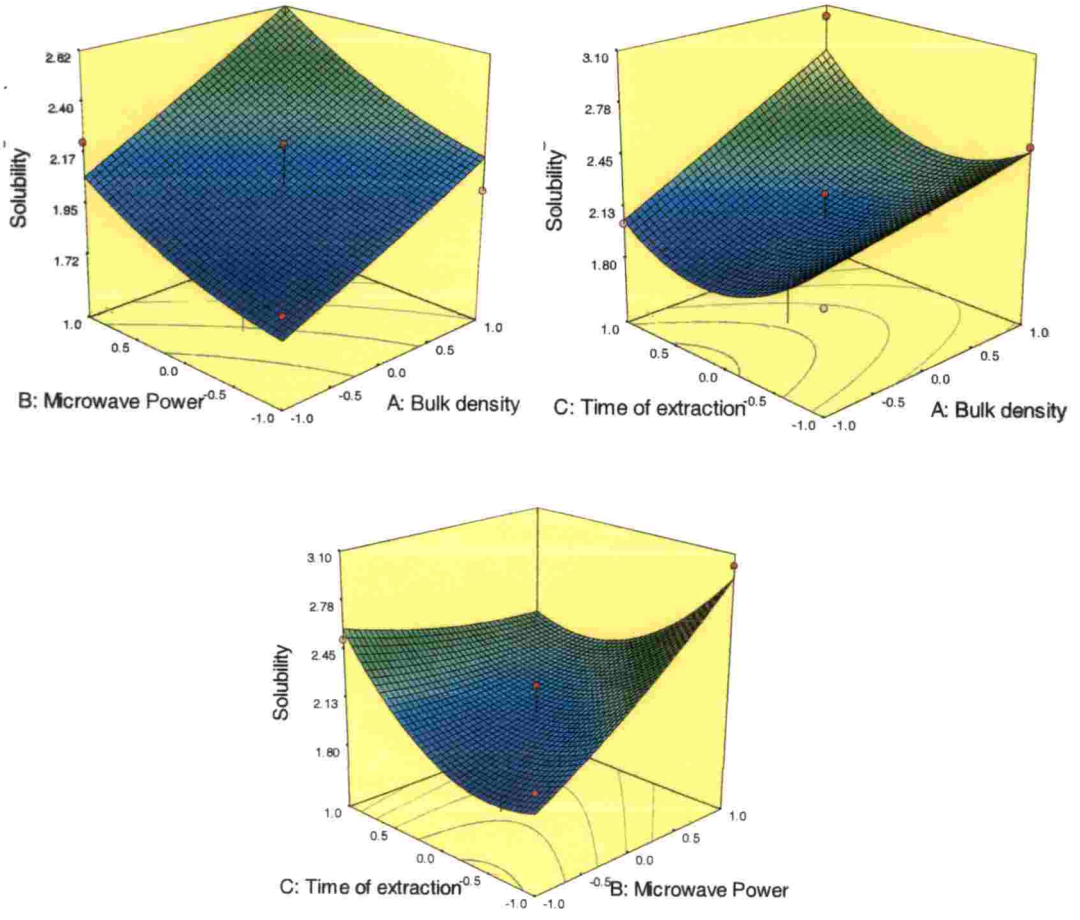


Figure 4.6. Effect of process parameters on solubility of essential oil

The variation of bulk density, microwave power and time of extraction with that of solubility is shown by plotting 3D graphs representing the response surface generated by the model. The 3D responses were shown in Figure 4.6.

From Figure 4.6. it may be perceived that independent variables has positive effect on the solubility of essential oil. With increase in microwave power solubility increased. However, there is no significant difference between the solubility values of oil obtained by SD and MSD processes.

**4.4.4 Colour**

The colour of lemongrass essential oil is determined by using Hunter lab colour flex meter as explained in section 3.4.4. The values of L*, a* and b* obtained for various samples are given in Table 4.2.

The ANOVA tables for the responses L*, a* and b* are shown in Appendix B (Table B.4., B.5., and B.6.). A second order non-linear regression equation described the relation between dependent and independent variables. It is evident from the regression coefficients for the model tabulated in Table 4.3. that the colour of oil is not effected by the input variables.

From Table B.4., it could be inferred that the values of $R^2$ and $R^2$-adj for the 'L (whiteness / darkness)' were 93.85 and 85.94%, respectively. The coefficient of determination ($R^2$) of the regression model for L* was 93.85%. From Table B.5., it could be inferred that the values of $R^2$ and $R^2$-adj for the 'a (redness /greenness)' were 87.39 and 71.17%, respectively. The coefficient of determination ($R^2$) of the regression model for 'a*' was 87.39%. It can be inferred from Table B.6. that the values of $R^2$ and $R^2$-adj for the 'b* (yellowness / blueness)' were 82.89 and 60.89%, respectively. The coefficient of determination ($R^2$) of the regression model for 'b*' was 82.89 %.

For all the colour values, lack of fit was found to be insignificant and F-value suggested that the models were significant at one per cent and five per cent level of significance. Therefore, second order models were adequate in describing the L*, a* and b* values of MSD essential oil.

Tables B.4, B.5 and B.6 shows that, the process parameters had a significant effect on the colour values at one per cent ($p<0.001$) level of significance

The effect of bulk density, microwave power and time of extraction on the colour of the essential oil is illustrated by plotting 3D graphs representing the response surface generated by the model. The 3D responses for L*, a* and b* were shown in Figure. 4.7., 4.8. and 4.9.

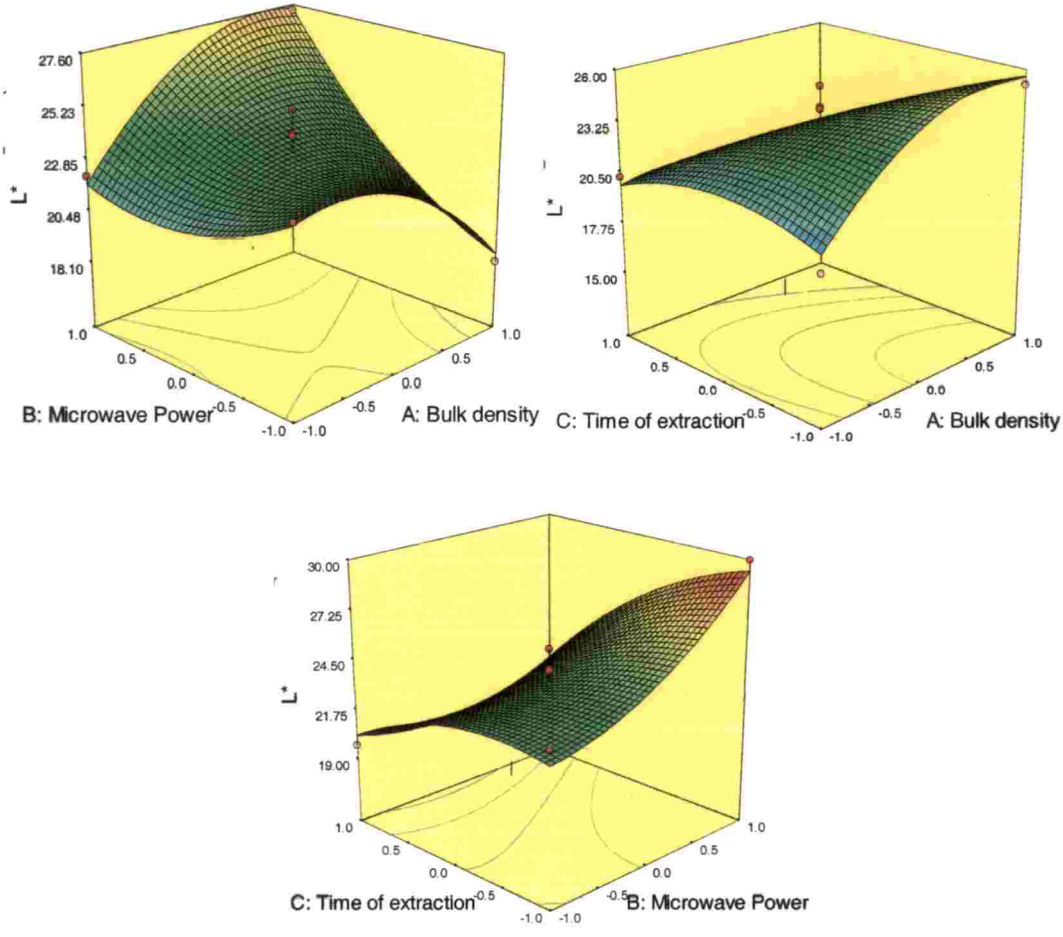


Figure 4.7. Effect of process parameters on L*

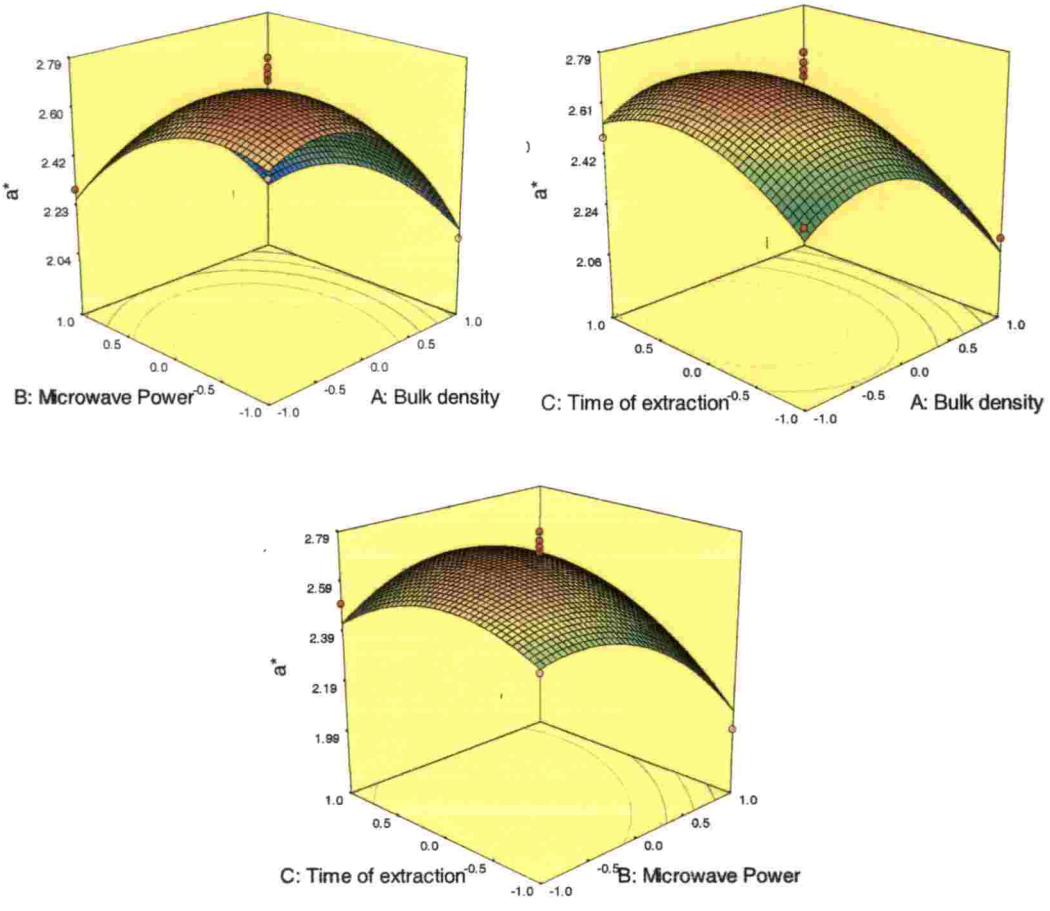


Figure 4.8. Effect of process parameters on a*

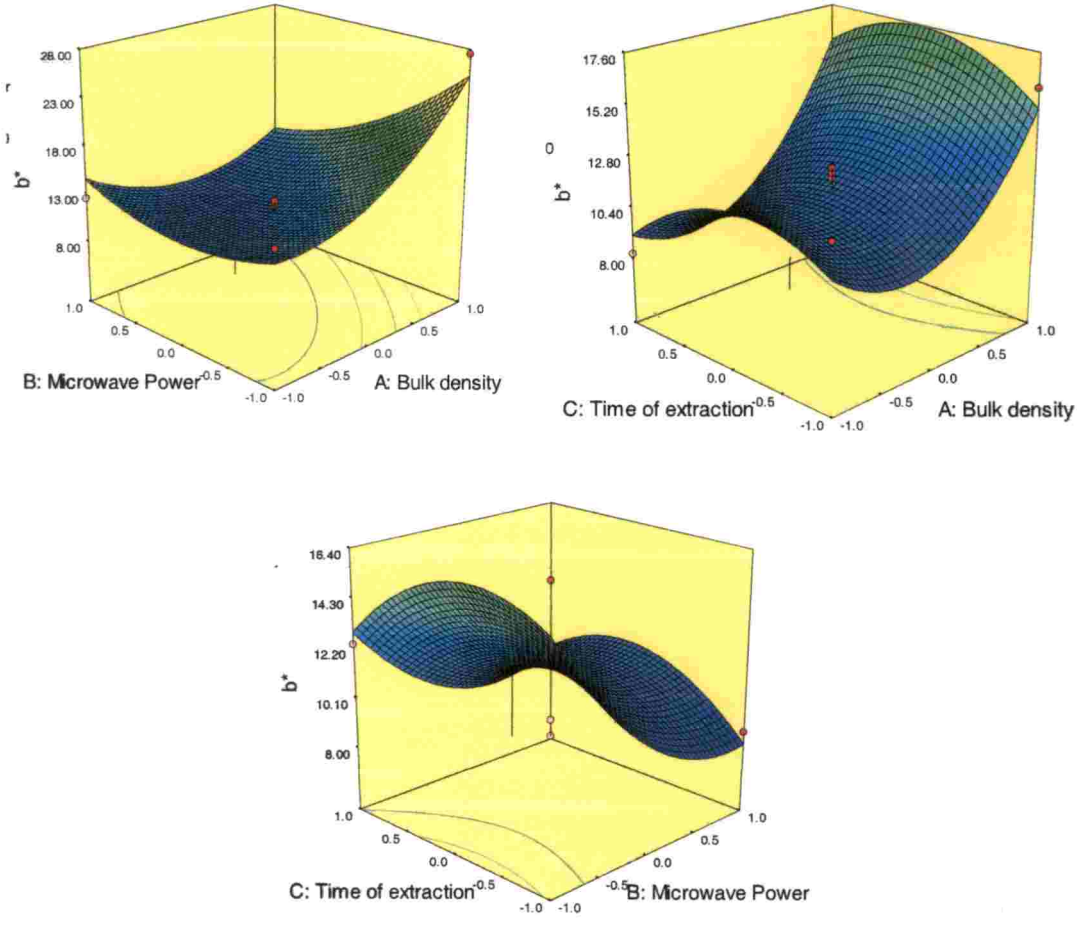


Figure 4.9. Effect of process parameters b*

From Figure 4.8. and 4.9. it may be concluded that the process parameters had a significant effect on the a* and b* value of essential oil. Increase in microwave power leads to a slight increase in these values which indicates that the oil has attained yellow and reddish colour. This might be due to the reason that with increment in microwave power, the colour of oil changes to dark yellow or even black because of the presence of suspended materials (Baron and Villa, 2014).

Further it may be inferred from the surface plots 4.7. that the process parameters have insignificant effect on the 'L*' value. This indicates that a clear oil is obtained by MSD process.

Table 4.3. Regression coefficients of physical quality characteristics of lemongrass essential oil

| Physical quality characteristics | Constant | A | B | C | AB | BC | AC | $A^2$ | $B^2$ | $C^2$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Specific gravity | 0.923 | -0.00747 | 0.010843 | -0.00576 | 0.017342 | -0.01952 | -0.02791 | 0.015656 | 0.003326 | -0.02343 |
| Refractive index | 1.527 | 0.007531 | 0.002434 | -0.0124 | -0.0175 | 0.017438 | 0.035506 | -0.02828 | -0.04622 | -0.03128 |
| Solubility | 2.08 | 0.25 | 0.2 | 0.025 | 0.025 | 0.125 | -0.325 | 0.0075 | 0.0575 | 0.3075 |
| L* | 23.476 | 0.225 | 1.8575 | -2.6625 | 2.625 | -2.525 | -1.385 | -2.0005 | 1.2495 | -1.2505 |
| a* | 2.6715 | -0.16781 | -0.10781 | 0.069375 | 0.069375 | -0.0075 | 0.11625 | -0.23731 | -0.16856 | -0.13669 |
| b* | 11.41792 | 2.757031 | -2.58125 | -0.22161 | -3.10573 | 0.623958 | 0.997396 | 3.330625 | 2.178021 | -2.03396 |

A: Bulk density (g $cm^{-3}$) B: Microwave power (W) C: Time of extraction (min)

### 4.4.5. Desirability

Desirability analysis was performed by employing the Design expert software. Desirability ranges from zero to one for any given response. A zero indicates that one or more responses fall outside desirable limits and a value of one represents the ideal case (Maran *et al.*, 2013).

From the desirability analysis, the optimum operating conditions for extraction of lemongrass essential oil were found to be: bulk density of 0.375 g $cm^{-3}$; microwave power of 420 W; and time of extraction of 30 min. The yield of lemongrass essential oil, total energy consumption and temperature of extraction at this optimum process parameter levels for microwave steam distillation process were found to be 0.40 ml, 1.10 kWh and 67°C respectively whereas the same were found to be 0.30 ml, 4.68 kWh and 80°C respectively for steam distillation process without microwave power. The time of extraction for SD process was 180 min.

Table 4.4. Optimal level obtained from desirability analysis

| Sl. No | Response | Units | Desirability | Optimal level | Low level | High level |
|---|---|---|---|---|---|---|
| 1 | Total yield of essential oil | ml | Maximise | 0.40 | 0.25 | 0.50 |
| 2 | Total energy consumption | kWh | Minimise | 1.10 | 1.10 | 1.80 |
| 3 | Temperature of extraction | Celsius | Minimise | 67 | 55 | 75 |
| 4 | Specific gravity | | Range | 0.894 | 0.874 | 0.965 |
| 5 | Refractive index | | Range | 1.49 | 1.403 | 1.54 |
| 6 | Solubility | V/V | Range | 2.02 | 1.82 | 3.02 |
| 7 | L* | | Range | 19.2 | 16.2 | 29.95 |
| 8 | a* | | Maximise | 2.43 | 1.99 | 2.78 |
| 9 | b* | | Maximise | 12.43 | 8.20 | 15.07 |

These results clearly indicate that the microwave steam distillation process resulted in a very rapid extraction process with considerable saving in energy. The saving in time and energy of the process was found to be 83.33 and 76.49%, respectively. The desirability of the optimisation was found to be 0.785. As the desirability value is close to one, the optimised values could be considered ideal.

## 4.5 SCANNING ELECTRON MICROSCOPY

The SEM images of lemongrass leaf before and after steam distillation were analysed. Figure 4.10 depicts the micrograph of the fresh leaf of lemongrass (before extraction). Images of the lemongrass which was subjected to a 3 h steam distillation (Figure 4.11) and 30 min microwave steam distillation (Figure 4.12) are also shown for comparison.

The SEM image of fresh leaves indicate the presence of intact oil bearing bundles (Figure 4.10.). On the other hand the other images shows that these glands were broken and the oil had been extracted. The images indicated apparent physical changes in the lemongrass by both these methods. While SD requires 180 min to rupture the glands MSD destroyed the glands in 30 min. This implies that microwaves cause the glandular walls to crumble more quickly and effectively.

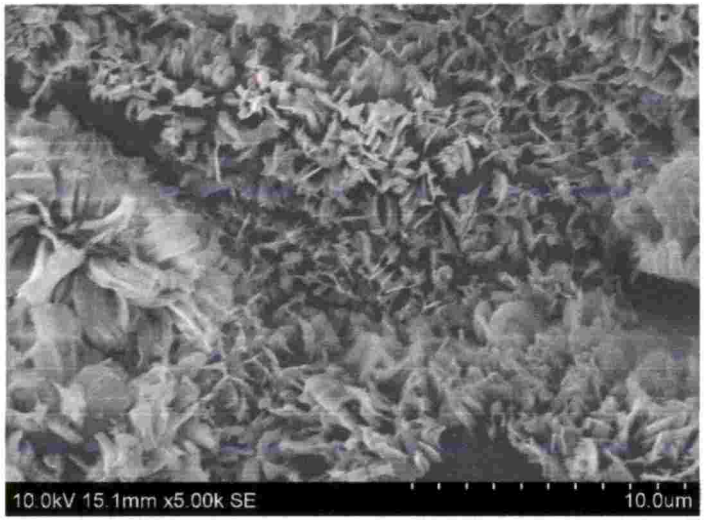


Figure 4.10. SEM micrograph of lemongrass before extraction

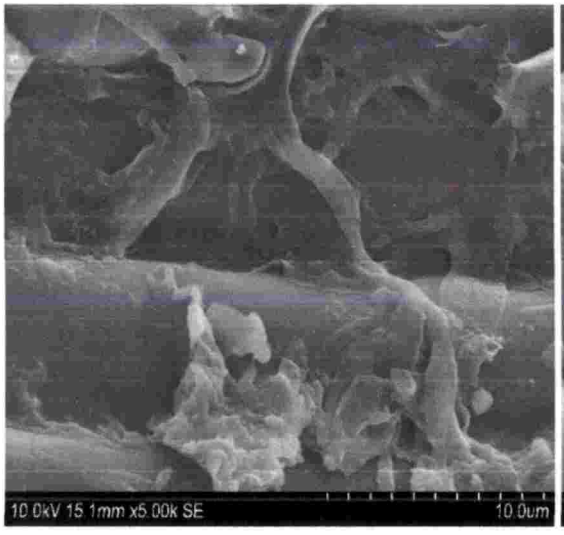


Figure 4.11. SEM micrograph of lemongrass after steam distillation without microwave power

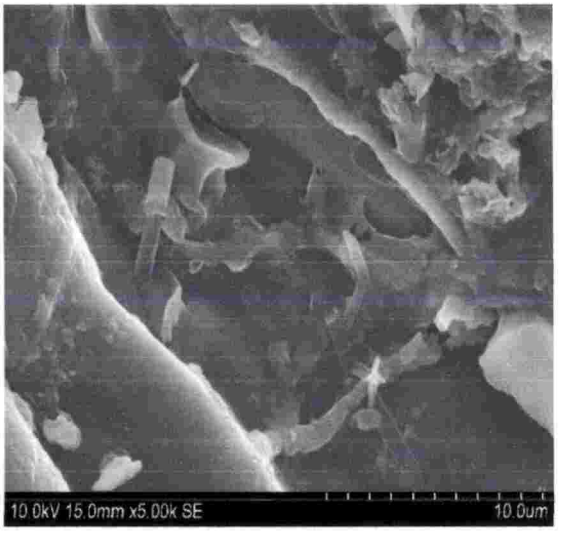


Figure 4.12. SEM micrograph of lemongrass after MSD

The gland undergone SD (Figure 4.11) was found to be wrinkled while that of MSD (Figure 4.12) was found not. Difference in the rate of heat transfer in both extraction techniques resulted in such changes. Irradiation, conduction and convection are three modes of heat transfer inside the sample utilised by MSD. Thus with MSD, heat is produced fast from inside the glands and from the outside. In SD, heat transfer could happen via conduction and convection only. For MSD, through microwave heating, the pressure build-up within the glands exceeded the expansion capacity of the glands and hence a faster rupture of the cell walls was observed, which was not occur with SD (Golmakani and Rezaei, 2008; Kusuma *et al.*, 2016).

## 4.6 DETERMINATION OF CHEMICAL CONSTITUENTS

Gas Chromatographic technique was carried out for the analysis of volatile oil components in the lemongrass essential oil extracted through steam distillation without microwave power and microwave steam distillation process. Citral (geranial and neral), the major aroma compound of the lemongrass essential oil was assessed. The presence of citral was analysed by comparing with the chromatograph of citral standard.

The gas chromatograph for citral standard, lemongrass essential oil extracted through steam distillation without microwave power and via microwave steam distillation are shown in Figure 4.13., Figure 4.14. and Figure 4.15. respectively. The results of gas chromatography analysis are given in tabulated form in Appendix C (Table C.1., C.2. and C.3.). The combination of the neral and geranial isomers - Citral is the chief compound to verify the quality of lemongrass essential oil. The major compound citral can be identified easily as it has the highest peak.

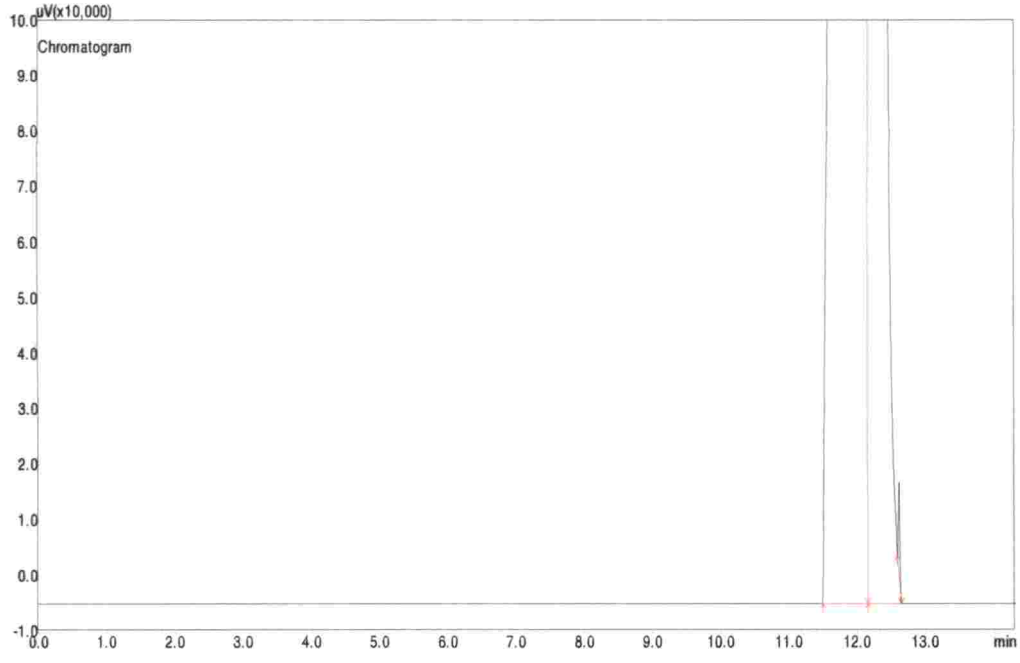


Figure 4.13. Gas chromatograph of citral standard

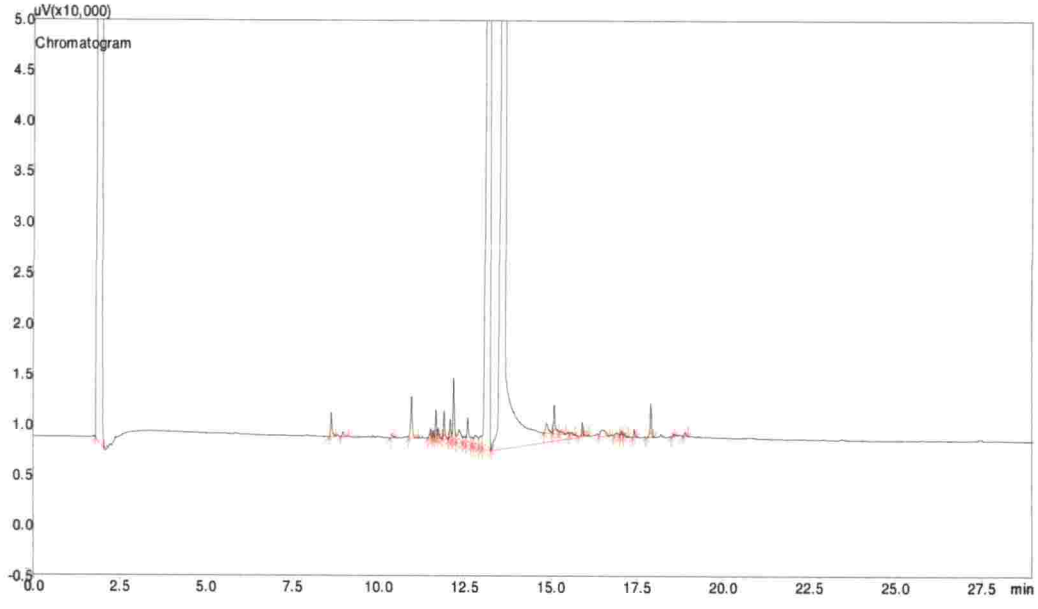


Figure 4.14. Gas chromatograph of steam distilled lemongrass essential oil without microwave power

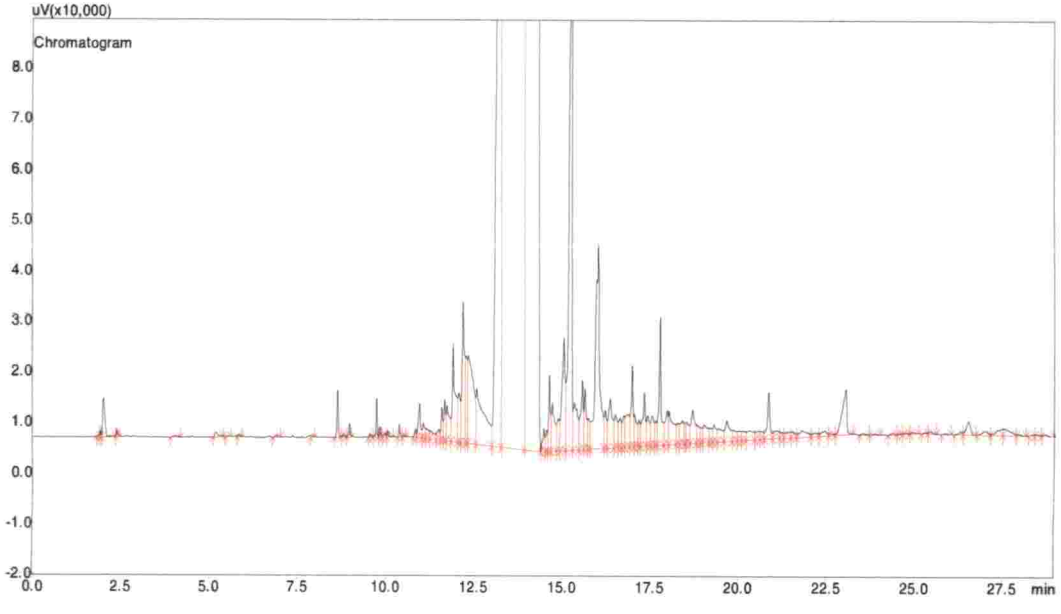


Figure 4.15. Gas chromatograph of microwave steam distilled lemongrass essential oil

From the results, it was observed that high concentration of key compound 'citral' is present in the essential oil isolated by MSD than that of SD essential oil. From the Figure 4.14. and 4.15., it can be concluded that the peak of citral in the chromatograph of MSD lemongrass oil at optimised conditions (peak height : 915353.1) is higher compared to the peak of citral in the chromatograph of SD lemongrass oil (peak height :175417.4). By comparing the chromatographs obtained, the oil compositions revealed that higher amounts of other aromatic volatile chemical constituents are contained in lemongrass essential oil extracted through MSD than that of oil extracted via steam distillation.

The microwave radiation effect that can penetrate into biological materials and creates heat by interaction with polar molecules, water leads to this difference. This heat cause damage into the cells spread and facilitates the release of active compounds from its glands and ultimately improves the efficiency of the extraction process. Also, this variation might be due to the degradation of citral at high temperatures employed in SD process. The citral content of MSD sample was slightly higher due to low power density and lesser extraction time because of which the oil was not exposed to high temperatures for a long time resulting in lower degradation of thermally liable citral (Ranitha *et al.*, 2014; Gavahian *et al.*, 2015; Sagarika *et al.*, 2016). It may be concluded that MSD greatly accelerated the extraction process, but without causing significant affect in the volatile oil composition and is obviously superior to SD in terms of the quality of the oil extracted.

# *Summary and conclusion*

# CHAPTER V

# SUMMARY AND CONCLUSION

Essential oils are aromatic oily liquids distilled from different parts of aromatic plants. These volatile oils represent the typical flavour and aroma of a particular plant from which they are obtained. They are low-volume, very high value products and contain a complex mix of components. These oils are used principally in perfumery and food flavourings.

There are various separation techniques for essential oils such as hydro distillation and steam distillation on the basis of distinct principles. Among the isolation procedures, steam distillation method is extensively used for commercial scale production. The major shortcomings of the conventional methods are low yields, long extraction time, high energy consumption, high cost, thermal and hydrolytic degradation and environmental pollution.

Modern technologies which developed gradually conquered the inadequacy of traditional methods, and enhanced the extraction efficiency. Such an innovative isolation technique for essential oil is microwave steam distillation. Microwaves can be used to mediate the extraction and steam and thus we can maintain mild conditions and have superior extraction. In this process microwave radiation is only applied on the extraction reactor, which is the basis of steam distillation principle. In microwave steam distillation process, while the plant material is subjected to microwave radiation, steam generated outside permeate through the plant material, evaporates and carries the essential oil towards the condenser where it is separated and collected. Since the plant material respond differently to the action of microwaves, the process parameters needs to be optimised for each biomaterial extracted using microwave steam distillation process.

In this study a microwave steam distillation system for extracting lemongrass essential oil was developed. The developed microwave steam distillation system composed of a microwave reactor, steam generator, cartridge, extraction unit,

supporting stand, energy meter and temperature sensor and controller. The microwave reactor selected provides a maximum microwave power of 700 W and is supported on a stand. Steam outlet from steam generator was connected to the cartridge containing lemongrass via silicon tube. Pressure of steam generated was measured using pressure gauge. The extraction unit mainly comprises of a clevenger apparatus and condenser. A supporting stand was fabricated for supporting the extraction system. The energy consumed for extraction in microwave steam distillation was measured using a single phase induction type energy meter and temperature of the extraction was determined using a temperature sensor and controller.

In order to evaluate the developed system towards extraction of lemongrass essential oil, the process parameters which would influence the essential oil yield, energy consumption and temperature of extraction were chosen as independent variables after thorough review of literature and the preliminary studies. The physical quality characteristics like specific gravity, refractive index, solubility and colour of essential oil were taken as dependent variables. Three levels of bulk densities (0.375, 0.625 and 0.875 g $cm^{-3}$), microwave power (280, 420 and 560 W) and time of extraction (30, 40 and 50 min) were taken as process parameters for the experiments.

The steam generator used was filled half with water, closed and heated by LPG. The desirable amount of lemongrass cut into small size, as per the experimental design, was filled in the cartridge which was placed inside the microwave reactor. One end of clevenger apparatus was connected to the cartridge via glass tube and the other end to the condenser. The microwave power level and time of exposure was set in the control panel of microwave reactor for various treatment conditions. Steam from steam generator regulated by the steam control valve, was allowed to pass through the cartridge via silicon tube. Microwaves at preset power level, heat the plant material for the set time interval. Essential oil in lemongrass gets vaporised and passes out of the microwave cavity along with steam, through the distillation stem into the condenser. These vapors get condensed and fall into the stem of the clevenger apparatus where the oil and water got separated due to density difference. Oil which is lighter than water

gets collected as the top layer. After completion of the process the water was drained off by opening the valve and the oil. The collected essential oil was then dehydrated with anhydrous sodium sulphate and stored at 2°C in amber coloured glass bottles for further analysis. Steam distillation was also performed without microwave power for a rigorous comparison with microwave steam distillation process. The physical quality characteristics of essential oil obtained by both the processes were compared.

For optimisation of the process parameters and to verify the sufficiency of the experimental design, the second order non-linear regression equation was fixed between dependent and independent variables. Analysis of variance (ANOVA) for the final predictive equation was carried out using the statistical software Design Expert (Trial version 7.0.0, STAT-EASE Inc.). Response Surface Methodology (RSM) was adopted and Box-Behnken design of three variables and three levels, each with three centre point combinations was used. The response surface equation was optimised for the response variables using the above software.

The chemical constituents of the essential oil extracted through steam distillation (SD) and optimised microwave steam distillation process (MSD) were obtained using Gas chromatography. Scanning electron microscopy was carried out for the supplemental chemical analysis of the lemongrass before and after extraction.

The results showed that an increase in essential oil yield was observed with increase in the bulk density and extraction time. The extraction was improved by increasing the microwave power from 280 W to 420 W. Total energy consumption and temperature of extraction enhance with increase in microwave power and time of extraction. The process parameters were found to have insignificant effect on the physical quality characteristics of the oil extracted through MSD and SD.

Microwave steam distillation resulted in an essential oil yield of 0.3-0.5%, with an extraction time of 30 min and energy consumption of 1.10 kWh. The time taken for extracting the lemongrass essential oil in steam distillation process was about 3 h with an energy consumption of 4.68 kWh. This indicates that the microwave steam distillation resulted in rapid extraction with about 83.33 per cent and 76.49 per cent

saving in time and energy respectively, when compared to steam distillation without microwave power.

The physical quality characteristics of essential oil was found to be similar in both the process. Gas Chromatography analysis of the essential oils brought out the fact that the usage of microwave radiation did not adversely affect the composition and the main aromatic chemical compound Citral was slightly higher in essential oil extracted through microwave steam distillation compared with steam distillation method. The Scanning Electron Microscopy (SEM) of lemongrass undergone microwave steam distillation and steam distillation provided evidences to a rapid rupture of essential oil glands with microwave steam distillation.

The optimised operating conditions of bulk density, microwave power and time of extraction for lemongrass essential oil in microwave steam distillation were found to be of 0.375 g $cm^{-3}$, 420 W and 30 min respectively. From the study it was concluded that microwave steam distillation could be considered as an extraction technique that results in the rapid production of high quality essential oil at shorter extraction period with minimum energy consumption.

The following are suggestions for future research work on the microwave steam distillation process:

1. Microwave steam distillation process could be compared with a conventional steam distillation unit
2. The heat loss through the silicon tube should be minimised through appropriate system
3. A provision for measurement of steam flow rate could be incorporated

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# *Appendices*

## APPENDIX A

## ANOVA tables for output charcteristics of developed microwave steam distillation system for lemongrass essential oil

Table A.1. ANOVA table for total yield of essential oil

| Source | Sum of squares | Degrees of freedom | Mean square | F value | P-value Prob>F | |
|---|---|---|---|---|---|---|
| Model | 0.075 | 9 | 8.282E-003 | 11.89 | 0.0018 | significant |
| A-Bulk density | 0.053 | 1 | 0.053 | 75.83 | < 0.0001 | |
| B-Microwave Power | 7.813E-003 | 1 | 7.813E-003 | 11.22 | 0.0123 | |
| C-Time of exposure | 6.513E-003 | 1 | 0.063 | 11.59 | <0.0001 | |
| AB | 0.000 | 1 | 0.000 | 0.000 | 1.000 | |
| AC | 5.625E-003 | 1 | 5.625E-003 | 8.08 | 0.0250 | |
| BC | 6.250E-004 | 1 | 6.250E-004 | 0.90 | 0.3750 | |
| $A^2$ | 2.632E-005 | 1 | 2.632E-005 | 0.038 | 0.8514 | |
| $B^2$ | 2.132E-003 | 1 | 2.132E-003 | 3.06 | 0.1237 | |
| $C^2$ | 5.158E-003 | 1 | 5.158E-003 | 7.41 | 0.0297 | |
| Residual | 4.875E-003 | 7 | 6.964E-004 | | | |
| Lack of Fit | 1.875E-003 | 3 | 6.250E-004 | 0.83 | 0.5413 | not significant |
| Pure Error | 3.000E-003 | 4 | 7.500E-004 | | | |
| Cor Total | 0.079 | 16 | | | | |

R-Squared : 0.9386 Pred R-Squared: 0.5632 Adj R-Squared: 0.8597 Adeq Precision: 12.043

Table A.2. ANOVA Table for total energy consumption

| Source | Sum of squares | Degrees of freedom | Mean square | F value | P-value Prob>F | |
|---|---|---|---|---|---|---|
| Model | 0.783273 | 9 | 0.08703 | 100.1994 | < 0.0001 | significant |
| A-Bulk density | 0 | 1 | 0 | 0 | 1.0000 | |
| B-Microwave Power | 0.045 | 1 | 0.045 | 51.80921 | 0.0002 | |
| C-Time of exposure | 0.72 | 1 | 0.72 | 828.9474 | < 0.0001 | |
| AB | 0 | 1 | 0 | 0 | 1.0000 | |
| AC | 0 | 1 | 0 | 0 | 1.0000 | |
| BC | 0.01 | 1 | 0.01 | 11.51316 | 0.0116 | |
| $A^2$ | 0.000152 | 1 | 0.000152 | 0.174515 | 0.6886 | |
| $B^2$ | 0.000152 | 1 | 0.000152 | 0.174515 | 0.6886 | |
| $C^2$ | 0.008152 | 1 | 0.008152 | 9.385042 | 0.0182 | |
| Residual | 0.00608 | 7 | 0.000869 | | | |
| Lack of Fit | 0.005 | 3 | 0.001667 | 6.17284 | 0.0555 | not significant |
| Pure Error | 0.00108 | 4 | 0.00027 | | | |
| Cor Total | 0.789353 | 16 | | | | |

R-Squared : 0.9923 Pred R-Squared: 0.9824 Adj R-Squared: 0.8965 Adeq Precision: 33.181

Table A.3. ANOVA Table for temperature of extraction

| Source | Sum of Squares | df | Mean square | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|
| Model | 533.2853 | 9 | 59.25392 | 59.68021 | < 0.0001 | significant |
| A-Bulk density | 15.125 | 1 | 15.125 | 15.23381 | 0.0059 | |
| B-Microwave Power | 480.5 | 1 | 480.5 | 483.9568 | < 0.0001 | |
| C-Time of exposure | 0.125 | 1 | 0.125 | 0.125899 | 0.7332 | |
| AB | 1 | 1 | 1 | 1.007194 | 0.3490 | |
| AC | 6.25 | 1 | 6.25 | 6.294964 | 0.0405 | |
| BC | 4 | 1 | 4 | 4.028777 | 0.0847 | |
| $A^2$ | 19.91842 | 1 | 19.91842 | 20.06172 | 0.0029 | |
| $B^2$ | 3.602632 | 1 | 3.602632 | 3.62855 | 0.0985 | |
| $C^2$ | 2.865789 | 1 | 2.865789 | 2.886407 | 0.1331 | |
| Residual | 6.95 | 7 | 0.992857 | | | |
| Lack of Fit | 5.75 | 3 | 1.916667 | 6.388889 | 0.0526 | not significant |
| Pure Error | 1.2 | 4 | 0.3 | | | |
| Cor Total | 540.2353 | 16 | | | | |

R-Squared : 0.9871 Pred R-Squared: 0.8262 Adj R-Squared: 0.9706 Adeq Precision: 26.825

## APPENDIX B

### ANOVA tables for physical quality charcteristics of microwave steam distilled lemongrass essential oil

Table B.1. ANOVA table for specific gravity of lemongrass essential oil

| Source | Sum of Squares | df | Mean square | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|
| Model | 0.010722 | 9 | 0.001191 | 6.900852 | 0.0093 | significant |
| A-Bulk density | 0.000446 | 1 | 0.000446 | 2.584211 | 0.1520 | |
| B-Microwave Power | 0.000941 | 1 | 0.000941 | 5.448223 | 0.0523 | |
| C-Time of exposure | 0.000265 | 1 | 0.000265 | 1.536221 | 0.2551 | |
| AB | 0.001203 | 1 | 0.001203 | 6.968472 | 0.0334 | |
| AC | 0.001525 | 1 | 0.001525 | 8.831788 | 0.0208 | |
| BC | 0.003116 | 1 | 0.003116 | 18.05213 | 0.0038 | |
| $A^2$ | 0.001032 | 1 | 0.001032 | 5.978503 | 0.0444 | |
| $B^2$ | 4.66E-05 | 1 | 4.66E-05 | 0.269851 | 0.6194 | |
| $C^2$ | 0.002312 | 1 | 0.002312 | 13.39336 | 0.0081 | |
| Residual | 0.001208 | 7 | 0.000173 | | | |
| Lack of Fit | 0.000978 | 3 | 0.000326 | 5.672053 | 0.0634 | not significant |
| Pure Error | 0.00023 | 4 | 5.75E-05 | | | |
| Cor Total | 0.01193 | 16 | | | | |

R-Squared : 0.8987 Adj R-Squared: 0.7685 Adeq Precision: 9.209

Table B.2. ANOVA table for refractive index of lemongrass essential oil

| Source | Sum of Squares | df | Mean square | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|
| Model | 0.027508 | 9 | 0.003056 | 6.110668 | 0.0131 | significant |
| A-Bulk density | 0.000454 | 1 | 0.000454 | 0.907178 | 0.3726 | |
| B-Microwave Power | 4.74E-05 | 1 | 4.74E-05 | 0.094784 | 0.7671 | |
| C-Time of exposure | 0.001231 | 1 | 0.001231 | 2.460485 | 0.1607 | |
| AB | 0.001225 | 1 | 0.001225 | 2.449089 | 0.1616 | |
| AC | 0.001216 | 1 | 0.001216 | 2.431627 | 0.1629 | |
| BC | 0.005043 | 1 | 0.005043 | 10.0818 | 0.0156 | |
| $A^2$ | 0.003367 | 1 | 0.003367 | 6.731411 | 0.0357 | |
| $B^2$ | 0.008996 | 1 | 0.008996 | 17.98457 | 0.0038 | |
| $C^2$ | 0.004121 | 1 | 0.004121 | 8.238722 | 0.0240 | |
| Residual | 0.003501 | 7 | 0.0005 | | | |
| Lack of Fit | 0.002821 | 3 | 0.00094 | 5.531964 | 0.0660 | not significant |
| Pure Error | 0.00068 | 4 | 0.00017 | | | |
| Cor Total | 0.03101 | 16 | | | | |

R-Squared : 0.8871 Adj R-Squared: 0.7419 Adeq Precision: 7.453

Table B.3. ANOVA table for solubility of lemongrass essential oil

| Source | Sum of Squares | df | Mean square | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|
| Model | 1.736824 | 9 | 0.19298 | 4.790293 | 0.0255 | significant |
| A-Bulk density | 0.5 | 1 | 0.5 | 12.41135 | 0.0097 | |
| B-Microwave Power | 0.32 | 1 | 0.32 | 7.943262 | 0.0258 | |
| C-Time of exposure | 0.005 | 1 | 0.005 | 0.124113 | 0.7350 | |
| AB | 0.0025 | 1 | 0.0025 | 0.062057 | 0.8104 | |
| AC | 0.0625 | 1 | 0.0625 | 1.551418 | 0.2530 | |
| BC | 0.4225 | 1 | 0.4225 | 10.48759 | 0.0143 | |
| $A^2$ | 0.000237 | 1 | 0.000237 | 0.005879 | 0.9410 | |
| $B^2$ | 0.013921 | 1 | 0.013921 | 0.345558 | 0.5751 | |
| $C^2$ | 0.398132 | 1 | 0.398132 | 9.882699 | 0.0163 | |
| Residual | 0.282 | 7 | 0.040286 | | | |
| Lack of Fit | 0.21 | 3 | 0.07 | 3.888889 | 0.1113 | not significant |
| Pure Error | 0.072 | 4 | 0.018 | | | |
| Cor Total | 2.018824 | 16 | | | | |

R-Squared : 0.8603 Adj R-Squared: 0.6807 Adeq Precision: 7.9576

Table B.4. ANOVA table for L* of lemongrass essential oil

| Source | Sum of Squares | df | Mean square | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|
| Model | 174.9072 | 9 | 19.43413 | 11.86391 | 0.0018 | significant |
| A-Bulk density | 0.405 | 1 | 0.405 | 0.247239 | 0.6343 | |
| B-Microwave Power | 27.60245 | 1 | 27.60245 | 16.8504 | 0.0045 | |
| C-Time of exposure | 56.71125 | 1 | 56.71125 | 34.62038 | 0.0006 | |
| AB | 27.5625 | 1 | 27.5625 | 16.82601 | 0.0046 | |
| AC | 25.5025 | 1 | 25.5025 | 15.56845 | 0.0056 | |
| BC | 7.6729 | 1 | 7.6729 | 4.684057 | 0.0672 | |
| $A^2$ | 16.85053 | 1 | 16.85053 | 10.2867 | 0.0149 | |
| $B^2$ | 6.573685 | 1 | 6.573685 | 4.013022 | 0.0852 | |
| $C^2$ | 6.584212 | 1 | 6.584212 | 4.019448 | 0.0850 | |
| Residual | 11.46662 | 7 | 1.638089 | | | |
| Lack of Fit | 4.3917 | 3 | 1.4639 | 0.827656 | 0.5436 | not significant |
| Pure Error | 7.07492 | 4 | 1.76873 | | | |
| Cor Total | 186.3738 | 16 | | | | |

R-Squared : 0.9385 Adj R-Squared: 0.8594 Adeq Precision: 14.3818

Table B.5. ANOVA Table for a* of lemongrass essential oil

| Source | Sum of Squares | df | Mean square | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|
| Model | 0.913233 | 9 | 0.10147 | 5.388414 | 0.0185 | significant |
| A-Bulk density | 0.225288 | 1 | 0.225288 | 11.96356 | 0.0106 | |
| B-Microwave Power | 0.092988 | 1 | 0.092988 | 4.937989 | 0.0617 | |
| C-Time of exposure | 0.038503 | 1 | 0.038503 | 2.044645 | 0.1958 | |
| AB | 0.019252 | 1 | 0.019252 | 1.022322 | 0.3456 | |
| AC | 0.000225 | 1 | 0.000225 | 0.011948 | 0.9160 | |
| BC | 0.054056 | 1 | 0.054056 | 2.870568 | 0.1340 | |
| $A^2$ | 0.237125 | 1 | 0.237125 | 12.59214 | 0.0094 | |
| $B^2$ | 0.119635 | 1 | 0.119635 | 6.35302 | 0.0398 | |
| $C^2$ | 0.078667 | 1 | 0.078667 | 4.177494 | 0.0803 | |
| Residual | 0.131818 | 7 | 0.018831 | | | |
| Lack of Fit | 0.025461 | 3 | 0.008487 | 0.319187 | 0.8124 | not significant |
| Pure Error | 0.106358 | 4 | 0.026589 | | | |
| Cor Total | 1.045051 | 16 | | | | |

R-Squared : 0.8738 Adj R-Squared: 0.7117 Adeq Precision: 5.8160

Table B.6. ANOVA Table for b* of lemongrass essential oil

| Source | Sum of Squares | df | Mean square | F value | p-value Prob > F | |
|---|---|---|---|---|---|---|
| Model | 241.3603 | 9 | 26.81781 | 3.767647 | 0.0471 | significant |
| A-Bulk density | 60.80977 | 1 | 60.80977 | 8.543194 | 0.0222 | |
| B-Microwave Power | 53.30281 | 1 | 53.30281 | 7.488538 | 0.0291 | |
| C-Time of exposure | 0.392904 | 1 | 0.392904 | 0.055199 | 0.8210 | |
| AB | 38.58221 | 1 | 38.58221 | 5.420434 | 0.0528 | |
| AC | 1.557296 | 1 | 1.557296 | 0.218785 | 0.6542 | |
| BC | 3.979194 | 1 | 3.979194 | 0.559039 | 0.4790 | |
| $A^2$ | 46.70763 | 1 | 46.70763 | 6.561978 | 0.0375 | |
| $B^2$ | 19.97379 | 1 | 19.97379 | 2.806127 | 0.1378 | |
| $C^2$ | 17.41889 | 1 | 17.41889 | 2.447188 | 0.1617 | |
| Residual | 49.82544 | 7 | 7.11792 | | | |
| Lack of Fit | 39.09063 | 3 | 13.03021 | 4.855313 | 0.0805 | not significant |
| Pure Error | 10.73481 | 4 | 2.683701 | | | |
| Cor Total | 291.1857 | 16 | | | | |

R-Squared : 0.8288 Adj R-Squared: 0.6088 Adeq Precision: 8.3889

## APPENDIX C

Table C.1. Result of Gas Chromatography of Citral standard

| Peak | Ret.time | Area | Height |
|---|---|---|---|
| 1 | 12.104 | 14741640 | 767370.7 |
| 2 | 12.391 | 11628863 | 918666.7 |
| 3 | 12.618 | 23997 | 17185.1 |

Table C.2. Result of Gas Chromatography of steam distilled lemongrass essential oil

| Peak | Ret.time | Area | Height |
|---|---|---|---|
| 1 | 1.926 | 7901801 | 1112991 |
| 2 | 8.623 | 6998.9 | 2412.8 |
| 3 | 8.965 | 1796.9 | 465.6 |
| 4 | 10.379 | 1306.2 | 443.2 |
| 5 | 10.947 | 14080.8 | 4127.6 |
| 6 | 11.493 | 2821.4 | 987.5 |
| 7 | 11.578 | 1944.5 | 761.7 |
| 8 | 11.654 | 6547.7 | 2830.2 |
| 9 | 11.725 | 2624.2 | 1053.2 |
| 10 | 11.89 | 8146.5 | 2813.8 |
| 11 | 12.069 | 6361 | 2149.5 |
| 12 | 12.169 | 18022.9 | 6344.1 |
| 13 | 12.327 | 10434.1 | 1315.4 |
| 14 | 12.492 | 4278.1 | 786.1 |
| 15 | 12.577 | 11307.7 | 2664 |

| | | | |
|---|---|---|---|
| **16** | 12.7 | 4003.9 | 859.1 |
| **17** | 12.8 | 9073.3 | 1149.7 |
| **18** | 12.955 | 7229.3 | 1214 |
| **19** | 13.211 | 898423 | 159335.1 |
| **20** | 13.653 | 1302665 | 175417.4 |
| **21** | 14.868 | 5408.9 | 974.2 |
| **22** | 15.083 | 10026 | 2865.8 |
| **23** | 15.25 | 2221.1 | 298.3 |
| **24** | 15.55 | 1699.8 | 283.8 |
| **25** | 15.899 | 3270.2 | 1327.9 |
| **26** | 16.491 | 7342.5 | 666.1 |
| **27** | 16.893 | 2783.5 | 354.8 |
| **28** | 17.039 | 2028.6 | 639.2 |
| **29** | 17.122 | 1032.5 | 330.3 |
| **30** | 17.411 | 2182.5 | 831.9 |
| **31** | 17.89 | 9228.6 | 3358.5 |
| **32** | 18.548 | 1100.4 | 300.5 |
| **33** | 18.885 | 1342.6 | 380 |

Table C.3. Result of Gas Chromatography of microwave steam distilled lemongrass essential oil

| Peak | Ret.time | Area | Height |
|---|---|---|---|
| **1** | 1.858 | 1552.7 | 403.4 |
| **2** | 1.907 | 2137.6 | 1230.2 |
| **3** | 1.997 | 35567.4 | 7607.5 |
| **4** | 2.379 | 3471.8 | 1569.9 |

| | | | |
|---|---|---|---|
| **5** | 4.033 | 1825.1 | 257.5 |
| **6** | 5.185 | 6760.7 | 964.6 |
| **7** | 5.508 | 1074.4 | 275.4 |
| **8** | 5.824 | 1433.1 | 437.9 |
| **9** | 6.89 | 2190.1 | 555.9 |
| **10** | 7.888 | 1229.4 | 338.1 |
| **11** | 8.634 | 23838.9 | 9251.9 |
| **12** | 8.81 | 1991.4 | 501.5 |
| **13** | 8.974 | 8063.3 | 2680.8 |
| **14** | 9.552 | 2090 | 582.6 |
| **15** | 9.742 | 18375.5 | 7563 |
| **16** | 9.847 | 5359.4 | 2120.7 |
| **17** | 9.987 | 3239.6 | 792 |
| **18** | 10.058 | 4091.9 | 1292.3 |
| **19** | 10.383 | 6713.8 | 2496.2 |
| **20** | 10.488 | 1084.4 | 243.8 |
| **21** | 10.836 | 4644.3 | 1640.3 |
| **22** | 10.951 | 27945.2 | 6691.9 |
| **23** | 11.062 | 12986.7 | 2952.8 |
| **24** | 11.14 | 13308.5 | 2004.7 |
| **25** | 11.268 | 14106.5 | 1671.7 |
| **26** | 11.498 | 10961.4 | 2185 |
| **27** | 11.583 | 20753.8 | 6413.6 |
| **28** | 11.661 | 28392.9 | 7912.8 |
| **29** | 11.73 | 46361.2 | 6969.6 |

| | | | |
|---|---|---|---|
| **30** | 11.901 | 114670.5 | 18915.4 |
| **31** | 12.071 | 48185.4 | 9703.3 |
| **32** | 12.182 | 139061.2 | 27758.1 |
| **33** | 12.265 | 60489.4 | 17188.9 |
| **34** | 12.332 | 196420.9 | 17272.6 |
| **35** | 12.579 | 177017 | 10877.8 |
| **36** | 13.25 | 1502258 | 176823.1 |
| **37** | 13.875 | 11924637 | 467480.5 |
| **38** | 14.322 | 15970012 | 915353.1 |
| **39** | 14.481 | 19481.8 | 4492.8 |
| **40** | 14.566 | 14160.7 | 4171.3 |
| **41** | 14.633 | 49571.2 | 14907.3 |
| **42** | 14.722 | 62239.2 | 9397.7 |
| **43** | 14.886 | 35413.1 | 6332.8 |
| **44** | 15.042 | 135924.8 | 22195.1 |
| **45** | 15.259 | 634180.2 | 118883 |
| **46** | 15.348 | 81900.9 | 9339.9 |
| **47** | 15.569 | 73320.4 | 13531.9 |
| **48** | 15.641 | 54631.6 | 11764.8 |
| **49** | 15.745 | 24202.6 | 6078.3 |
| **50** | 16.022 | 350633 | 40334.9 |
| **51** | 16.225 | 36513.4 | 7464 |
| **52** | 16.363 | 79349.8 | 9720.8 |
| **53** | 16.512 | 41814.1 | 6544.2 |
| **54** | 16.645 | 31981.7 | 5781.2 |

| | | | |
|---|---|---|---|
| **55** | 16.749 | 30325.9 | 6170.8 |
| **56** | 16.876 | 53942.7 | 6373.2 |
| **57** | 16.99 | 65983.4 | 15889.1 |
| **58** | 17.056 | 35963.5 | 6347.4 |
| **59** | 17.188 | 21523.3 | 5192.9 |
| **60** | 17.329 | 60546.2 | 10564 |
| **61** | 17.429 | 35906.1 | 5928 |
| **62** | 17.552 | 38191.3 | 5893.2 |
| **63** | 17.659 | 18081.3 | 4838.9 |
| **64** | 17.779 | 106379.4 | 25083 |
| **65** | 17.979 | 42438.8 | 6762.5 |
| **66** | 18.041 | 60885.5 | 6508.9 |
| **67** | 18.281 | 21648.6 | 4184 |
| **68** | 18.416 | 33865.5 | 4566.8 |
| **69** | 18.491 | 13374 | 4156.8 |
| **70** | 18.54 | 25518.4 | 4207.3 |
| **71** | 18.7 | 52067 | 6542 |
| **72** | 18.831 | 20929.9 | 3580.4 |
| **73** | 18.943 | 12605.3 | 3089.9 |
| **74** | 19.028 | 33123.6 | 3520.5 |
| **75** | 19.19 | 16535.5 | 2822.6 |
| **76** | 19.314 | 23329.7 | 3451.6 |
| **77** | 19.449 | 27124 | 2598.6 |
| **78** | 19.674 | 38991.1 | 4064.5 |
| **79** | 19.851 | 18473.4 | 2181.4 |

| | | | |
|---|---|---|---|
| **80** | 20.038 | 13171.5 | 1891.9 |
| **81** | 20.138 | 13561.3 | 1789.8 |
| **82** | 20.315 | 24866.3 | 1816.8 |
| **83** | 20.578 | 13893.4 | 1647.2 |
| **84** | 20.68 | 11335 | 1536.7 |
| **85** | 20.866 | 48813.8 | 9069.1 |
| **86** | 21.072 | 17049.8 | 1561.2 |
| **87** | 21.215 | 7947 | 1252.3 |
| **88** | 21.396 | 12711.6 | 1161.4 |
| **89** | 21.545 | 10126.2 | 1205.4 |
| **90** | 21.809 | 20817.8 | 1283.9 |
| **91** | 22.175 | 7977.2 | 675.7 |
| **92** | 22.447 | 11482.3 | 997.1 |
| **93** | 22.59 | 3609.1 | 427.6 |
| **94** | 23.05 | 80764.3 | 8821.7 |
| **95** | 23.537 | 2098.4 | 271 |
| **96** | 23.876 | 1768.1 | 228.2 |
| **97** | 24.371 | 2492.3 | 367.1 |
| **98** | 24.599 | 1011.3 | 174.8 |
| **99** | 24.753 | 2274 | 281.6 |
| **100** | 24.957 | 2811.6 | 352.5 |
| **101** | 25.275 | 1759.7 | 205 |
| **102** | 25.434 | 3231.4 | 409.7 |
| **103** | 25.872 | 1446.8 | 178.1 |
| **104** | 26.29 | 4434.7 | 497 |

| | | | |
|---|---|---|---|
| **105** | 26.521 | 22056.7 | 2561.8 |
| **106** | 26.996 | 9267.5 | 837.9 |
| **107** | 27.475 | 15801.4 | 1237.1 |
| **108** | 27.525 | 19203 | 1279.4 |
| **109** | 27.955 | 2528.3 | 272.1 |
| **110** | 28.37 | 1334.7 | 204.4 |
| **111** | 28.454 | 2706.2 | 293.3 |
| **112** | 28.684 | 3378.6 | 327 |

# STUDIES ON MICROWAVE STEAM DISTILLATION PROCESS FOR EXTRACTION OF LEMONGRASS ESSENTIAL OIL

By

CLAUDIA K.L.

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ABSTRACT OF THESIS

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In

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(Agricultural Processing and Food Engineering)

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## ABSTRACT

Essential oils are concentrated aromatic oily liquids distilled from different parts of aromatic plants. Conventionally steam distillation has been widely used for extraction. Modern technologies have been continuously developed to conquer the inadequacies of conventional methods. Microwave steam distillation is based on the interaction between water in the plant material and microwaves generated by the energy source. In this process, the steam generated outside accelerates evaporating and carrying of the essential oil, from the plant material, towards the condenser. In this study a microwave steam distillation system for extracting lemongrass essential oil was developed which composed of a microwave reactor, steam generator, cartridge, extraction unit, supporting stand, energy meter and temperature sensor and controller. In order to evaluate the developed system towards extraction of lemongrass essential oil, the effect of process parameters which would influence the essential oil yield, energy consumption and temperature of extraction such as bulk densities of 0.375, 0.675 and 0.875 g $cm^{-3}$, microwave powers of 280, 420 and 560 W and soaking times of 30, 40 and 50 min were studied. The physical quality characteristics such as specific gravity, refractive index, solubility and colour of essential oil were analysed. The optimised operating conditions of bulk density, microwave power and time of extraction for lemongrass essential oil in microwave steam distillation were found to be of 0.375 g $cm^{-3}$, 420 W and 30 min respectively. Scanning Electron Micrographs of lemongrass provided evidences to sudden rupture of essential oil glands with microwave steam distillation process. Gas Chromatographs of essential oil extracted through microwave steam distillation process showed higher percentage of Citral than that of steam distillation process. From the study it was concluded that microwave steam distillation could be considered as an extraction technique that results in the rapid production of high quality essential oil at shorter extraction period with minimum energy consumption.