OPTIMIZATION OF MICROWAVE ASSISTED PROCESS FOR EXTRACTION OF NUTMEG SEED ESSENTIAL OIL

By

SAFEENA P. A.

SRAVAN RAJ

SREELATHA E.

STENY SIMON

THEERTHA M. S.

PROJECT REPORT

Submitted in partial fulfillment of the

Requirement for the degree

Bachelor of Technology

In

Food Engineering

Faculty of Food Engineering and Technology

KERALA AGRICULTURAL UNIVERSITY

Department of Food & Agricultural Process Engineering

KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY

TAVANUR - 679 573,

KERALA, INDIA

2017

DECLARATION

We hereby declare that the thesis entitled “OPTIMIZATION OF MICROWAVE ASSISTED PROCESS FOR EXTRACTION OF NUTMEG SEED ESSENTIAL OIL” is a bonafide record of research work done by us during the course of research and that the thesis has not previously formed the basis for the award of any degree, diploma, associateship, fellowship or other similar title of any other University or Society.

Safeena P A

Theertha M S

Sravan Raj

Sreelatha E

Steny Simon (2013-06-021)

Place: Tavanur
Date: 27/02/2017

CERTIFICATE

Certified that this project report entitled "OPTIMISATION OF MICROWAVE ASSISTED PROCESS FOR EXTRACTION OF NUTMEG SEED ESSENTIAL OIL " is a record of project work done jointly by Safeena P.A. (2013-06-017), Theertha M S (2013-06-018), Sravan Raj (2013-06-019), Sreelatha E (2013-06-020), Steny Simon (2013-06-021) under my guidance and supervision and that it has not previously formed the basis for the award of any degree, diploma, associateship ,fellowship to them.

Place: Tavanur

Date: 27/02/2017

Dr. Prince M.V.

Professor &

Head i/c

Dept. of FAP

K.C.A.E.T, Tavanur

ACKNOWLEDGEMENT

With profound and reverence, we would like to express my sincere gratitude and thanks to Dr. Prince M.V.,Professor &Head& project guide, Department of FAPE, KCAET, for his valuable guidance, profound suggestions, constant backing, encouragement and advice throughout the project work.

We are immensely thankful to Dr. Santhi Mary Mathew, Professor, Department of FAPE, KCAET, Tavanur for her constant support, encouragement and guidance that helped us to complete our Project successfully.

With deep sense of gratitude and due respect, we acknowledge our gratitude to
Dr.Hajilal. M S, Dean, KCAET, Tavanur.

We are immensely thankful to Dr.Sudheer K P, Associate Professor, Department of FAPE, KCAET, and Tavanur for his constant support, encouragement and guidance that helped us to complete our Project successfully. We also extend our sincere thanks and gratitude to Dr.Rajesh G.K., Associate Professor, and Department of FAPE for his support and encouragement.

We are extremely thankful to Smt. Sreeja R, Assistant Professor, Department of FAPE , for her constant help, support, valuable suggestions and guidance that helped in the completion of our Project successfully.

We would also like to thank Lab Assistants Mr. Radhakrishnan, Mr. Sreenish, and Mr. Suhail for their constant support, valuable assistance throughout the Project.

Also I thankfully remember the services of Mr. Lenin, Technician, KCAET, Tavanur for their immense help. We would also like to thank our parents for their constant support throughout the Project.

We also extend our sincere thanks to all the faculties of Library, KCAET, and Tavanur for their help and support.

Last but not the least, we thank our sincere friends for their ever willing help, support and cooperation that helped us to complete the Project successfully.

Above all, I bow my head to God Almighty who had been a part of our efforts, invigorating, enlightening and making me confident and optimistic.

Safeena P .A.

Theertha M S

Sravan Raj

Sreelatha E.

Steny Simon

DEDICATED TO ALL

FOOD ENGINEERS

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF PLATES

LIST OF SYMBOLS AND ABBREVIATIONS

^oC	:	Degree Celsius
%	:	Percentage
&	:	And
/	:	Per
D	:	Diameter
et al.	:	and others
etc.	:	Etcetera
GHz	:	Giga Hertz
g	:	Gram
$g.l^{-1}$	:	gram per litre
$g.mg^{-1}.day-^{1}$	:	gram per milli gram per day
H	:	Hour
Hz	:	Hertz
i.e.	:	that is
I.U	:	International Unit
K.C.A.E.T	:	Kelappaji College of Agricultural Engineering and Technology
Kcal	:	kilo calorie
kWh	:	kilo Watt hour
Mg	:	Milli gram
MHz	:	Mega Hertz
min	:	Minute (s)
Ml	:	Milli Litre
Mm	:	Milli Meter
MT	:	Metric Tonne
RDA	:	Recommended Dietary Allowance
RF	:	Radio Frequency
S	:	Second (s)
Sl.	:	Serial
T	:	Tonne
V	:	Volt
W	:	Watt
Wb	:	Wet basis
$W.g^{-1}$	:	Watt per gram
KWh	:	Kilo watt hour
Fig	:	figure
F &APE	:	Food & agricultural process engineering
No.	:	Number

INTRODUCTION

CHAPTER 1

INTRODUCTION

Spices which are obtained from plant or vegetable products or mixtures of both are used in whole or ground form for cooking, mainly for providing flavour, aroma and pungency to food. These low volume high value products are known to have disease inhibiting and health promoting properties.They have been in use since ancient times for their carminative, anti-inflammatory anti-flatulent properties. The active principles in the spices may help in smooth digestion through increasing intestinal system function and stimulating excessive secretion of gastro-intestinal enzymes inside the stomach. Spices are heterogeneous collections of a wide variety of volatile and non-volatile basic dietary additives.The oil of the plant known as “Essential oil” are the volatile components distilled from the aromatic plant materials which possess characteristic flavour and taste. The essential oil consists of fragrances which are oily in nature and represent the essence or active constituents of plants. They are called volatile or ethereal oils as they evaporate when exposed to atmosphere at ordinary temperatures. Demand and price of essential oils and herbal products are increasing constantly in national and international markets due to strong pro-consumer movement.

Essential oils are generally extracted by distillation. Distillation may be defined as separation of components from a mixture of two or more liquids by virtue of difference in their vapour pressure. There are three systems of distillation- hydro, hydro-steam and steam distillation.Other processes include solvent extraction, expression, absolute oil extraction, resin tapping, and cold pressing. But these methods carry the disadvantages mainly concerned with the quality of final product such as loss of some volatile notes, low extraction efficiency and degradation of unsaturated ester compounds through thermal or hydrolytic effects (Ferhat *et al.,* 2006).

Microwave energy could be used effectively to mediate the extraction of essential oil in place of steam or water heating in order to introduce its inherent advantages. As in the case of microwave heating of food materials, the internal heating of the already present water within the plant material by the microwaves leads to the rupture of the glands and odoriferous receptacles freeing the essential oil which is then evaporated by the in-situ water of the plant material. The water then evaporated could then be passed through a condenser outside the microwave cavity where it is condensed. 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 efficiency, less extraction time and increased yield with quality of the extracted oil superior to that of other conventional methods due to the mild conditions (Lucchesi *et al*., 2004). Besides, microwave extraction may be classified as a green technology and is energy efficient. The process control is also easy. Since microwave heating is a volumetric process in which heating is through kinetic effects, biomaterials respond differently with the microwave energy.

Myristica fragrans is commonly known as “nutmeg”, it produces two spices: mace and nutmeg. Nutmeg is the seed kernel inside the fruit and mace is the red lacy covering (aril) on the kernel. M. fragrans belongs to Myristicaceae family in the order Magnoliales which contains about 150 genera and more than 3000 species. Myristica species are natives of Moluccas, indigenous to India, Indonesia and Sri Lanka and now cultivated in many tropical countries of both hemispheres as well as in South Africa (Pal *et al.*, 2011). *M. fragrans* is a spreading aromatic evergreen tree usually growing to about 5 to13 m high. Fruit is composed of three parts: the pericarp or husk, the mace and the seed. Seeds (nutmegs) are broadly ovoid (2 to 3 cm long), firm, fleshy, whitish and transversed by red-brown veins. When fresh, the aril (mace) is bright scarlet becoming more horny, brittle and with a yellowish-brown color when dried.

Nutmeg is popular as a spice and also possesses various therapeutic properties. Nutmeg has a characteristic pleasant fragrance and a slightly warm taste. It is used to flavor many kinds of baked foods, confections, puddings, meats, sausages, saucers, vegetables and beverages. It is also used as components of curry powder, teas and soft drinks or mixed in milk and alcohol (Olaleye *et al.,* 2006). For a long time, *M. fragrans* has been used as a folklore medicine for treating diarrhea, mouth sores and insomnia (Somani and Singhai, 2008). Since the Middle Ages, nutmeg has been used as a stomachic, stimulant, carminative as well as for intestinal catarrh and colic, to stimulate appetite, to control flatulence and has a reputation as an emmenagogue and abortifacient (Min *et al*., 2011). The essential oil of nutmeg is used externally for rheumatism and possesses analgesic and antiinflammatory properties (Santos et al., 1997; Olajide *et al*., 1999).

Some recently published studies have utilized the microwave energy for extraction of essential oil from various spices. Since microwaves heat the biomaterial through kinetic effects and are a volumetric heating process, the plant materials respond differently to the action of microwaves. Therefore, the process parameters leading to the efficient extraction and quality oil needs to be optimized for each material extracted using microwave assisted technology. In this context, it may be noted that such optimization studies pertaining to nutmeg seed oil has not been found reported.

Considering the above facts a study was undertaken on **“Optimization of microwave assisted process for extraction of nutmeg seed essential oil”** with the following objectives:

- Evaluation of the developed microwave assisted extraction system towards extraction of nutmeg seed essential oil and optimization of the process parameters.
- Characterization of the microwave assisted extracted nutmeg seed essential oil in comparison with hydro distilled oil.

REVIEW OF LITERATURE

CHAPTER 2

REVIEW OF LITERATURE

This chapter deals with the review of research work reported on the scenario of nutmeg and benefits of nutmeg usage as a food ingredient. A review on application of microwave technology in extraction of essential oil and dielectric properties of oil has also being stated.

NUTMEG

Nutmeg (*Myristica fragrans* Houtt.,) is one of the most important spices with high economic value. It is an evergreen aromatic tree cultivated in many tropical countries. The two spices nutmeg and mace are called twins because they form the both parts of the fruit "*Myristicafragrans*". The more or less spherical fruits up to 8–15 cm in diameter and containing numerous large seeds, usually hang singly on long stalks beneath the foliage (Keay, 1989). Used as traditional flavorings for sweets including – custards, cakes, desserts and other palatable dishes especially fish, spinach, pasta and quiche. These are used in beverages like coca cola as a flavoring agent. They also contain hallucinogens, and can be fatally toxic if used in a large quantity like eating an entire nutmeg, however consumption of small quantities normally used in cooking are considered safe (Nelson, 2013).

Global Scenario

Native of Indonesia, nutmeg tree grows there abundantly and is now cultivated in West Indies, India, Phillipines, Srilanka, Tropical America and Pacific Islands (Verghese, 2000). In India, the plant is grown in certain pockets of Kerala, Tamil Nadu, Karnataka, Goa, Maharashtra, North East India and Andamans. But the major producer and supplier of nutmeg is Indonesia, which contributes around 80 per cent of world nutmeg (Krishnamoorthy, 2000).

On a global scale, the annual growth rate in spices consumption is estimated at around 10 per cent. India exports its spices to more than 120 countries in the world. World trade in spices has shown a consistently upward trend over the past 25 years. Spices like nutmeg can be used for intensive agriculture under mixed farming systems along with other horticultural crops. Efforts to increase the sale value of broken seeds of nutmeg can be carried out by processing nutmeg into essential oils, nutmeg's oleoresin, and nutmeg butter.

National Scenario

The area and production of nutmeg in India during 2014-15 is 19,000 hectares and 12,780 MT, respectively.Value added products including spice oils, oleoresins, mint products, curry powder/paste/condiments, and spice powders contributed around 58 per cent in value towards the total export earnings by spices from India. Nutmeg oil is one of the major spice oils which is getting exported and USA is the major importer of spice extracts followed by Germany, UK, South Korea and China. Guinea and Sri Lanka are the two major competing countries with India in production and export of nutmeg and mace (IISR, 2013).

State Scenario

Nutmeg cultivation in Kerala is majorly concentrated in the Thrissur, Ernakulam and Kottayam districts. The climatic conditions of Kerala suit nutmeg cultivation, and it is grown in homesteads in the State as an intercrop. Different varieties of nutmeg are available in Kerala, with plants varying in growth patterns, shape and size of the nuts, the quality and quantity of the mace etc. Trees possessing over 10,000 fruits per year regularly, with nuts weighing 10 g each and mace 1 g/fruit can be selected as mother trees (Bavappa and Ruettimann, 1981). Male or female nutmeg trees can exist. When seeds are planted, around 50 per cent of the plants will be male and they will bear only less quantities of fruits. Moreover, there is no reliable way of identifying whether the plant is male or female before it starts flowering. So planting seeds for establishing a nutmeg plantation is a risky task for farmers. Hence farmers should opt for vegetative propagated plants using budding or grafting techniques. At the same time, it should be noticed that male trees are needed for pollination. An ideal plantation should have male-female trees in the ratio of 1:10 (Rethinam and Edison, 1991). Among the total production of nutmeg in India, Kerala accounts for 97.15 per cent of the area and 98 per cent of the total production (SBI, 2015).

Benefits of Nutmeg

Nutmeg has been used for thousands of years for various purposes. Joseph and Rachael (2003) reported that nutmeg has a variety of therapeutic properties and can also be used in a wide range of recipes. It is also used for insomnia, anxiety, calming muscle spasms, vomiting, nausea, indigestion, diarrhea, gout, joint pain, lowering blood pressure level, male infertility, impotence, improving concentration, lowering cholesterol, increasing blood circulation, toothaches etc.

Adding nutmeg seed to your diet offers a number of nutritional benefits, including increased fiber and mineral intake, but consuming too much might cause harmful side effects. Use nutmeg seed in your cooking as a source of dietary fiber - a nutrient important for lifelong health. Fiber helps control both your blood cholesterol and your blood sugar levels, and following a fiber-rich diet helps reduce your risk of chronic illnesses, including Type 2 diabetes and cardiovascular disease.

Fiber also softens your stool, so you're less likely to suffer from constipation, and fights other digestive disorders, such as diverticular disease. Each 2-tablespoon serving of ground nutmeg seed provides you with 2.9 grams of dietary fiber - 8 percent of the daily recommended intake for men and 11 percent for women, as set by the Institute of Medicine.

Nutmeg seed also offers health benefits by boosting your mineral intake, particularly copper and manganese. Both minerals help keep your skeleton strong and heathy. Manganese also helps you synthesize sex hormones, while copper boosts your immune system. A serving of ground nutmeg seed contains 0.41 milligram of manganese. 23 and 18 percent of the recommended daily manganese intakes for women and men, respectively as well as 144 micrograms of copper, or 16 percent of the recommended daily intake.

Nutmeg seeds also house chemicals that might combat cancer growth. One study, published in the "Journal of the Medical Association of Thailand" in 2007, found that unknown compounds in nutmeg were able to fight the growth of leukemia cells in test-tube studies. An additional test-tube study, published in the May 2005 issue of "Toxicology Letters," found that nutmeg promoted brain cancer cell death. As of September 2013, it's not yet known exactly how well nutmeg seed fights cancer development, but it might offer some anti-cancer benefits. Myristica oil, the natural oil found in nutmeg, can cause adverse reactions if consumed in large amounts. If you develop symptoms of Myristica oil poisoning which can include digestive upset, flushed skin, chest pain, confusion and hallucinations seek immediate medical attention (Sylvie Tremblay, 2015).

Antimicrobial Effect

Latha *et al.* (2005) stated that the essential oils obtained from nutmeg seeds are used in tonics. Also, they inhibit the growth of *Listeria monocytogenes* by abolishing the production of the bacterial extracellular protein, listeriolysin and the bacterial enzyme phospholipase. Nutmeg extract showed mild antibacterial activity against pathogenic staphylococci. Gupta *et al.* (2013) conducted a study on antioxidant and antimicrobial potential of nutmeg. Here, extracts of nutmeg were evaluated for antimicrobial activity against gram positive (*B. subtilis and S. aureus*), gram negative (*P. putida and P. aeruginosa*) bacteria and pathogenic fungi (*A. fumigatus, A. niger and A. flavus*) using disc diffusion method. Also their minimum inhibitory concentrations (MIC) were determined. It was found that the extracts of nutmeg used in the present study possess substantial antimicrobial activity against the tested microorganism. Acetone extract of nutmeg has shown vigorous antimicrobial activity than all other extracts of nutmeg used in the study. High antioxidant and antimicrobial activity could be due to the presence of α-pinene, β-pinene, myrcene, 1, 8-cineole, carvacrol, terpinen-4-ol, eugenol and isoeugenol. This study strongly maintained the ethno-pharmacological importance of the nutmeg. Also, it was found that the antioxidant and antimicrobial activity possessed by nutmeg could be helpful preventing or to reduce the progress of various oxidative stress-related diseases and infections by subtle pathogenic microorganisms.

Escherichia coli O157:H7 is an enterohemorrhagic serotype of the bacteria *Escherichia coli* and a cause of illness, typically through consumption of contaminated raw food including raw milk. Infection with this type of pathogenic bacteria may lead to hemorrhagic diarrhea, and also cause kidney failure. Takikawa *et al.* (2002) studied antimicrobial activity of nutmeg against *Escherichia coli* O157:H7. Various spices (5 g each) were homogenized at 25ºC for 10 min with 5 ml of 70 per cent ethyl alcohol, and the supernatant solutions obtained by centrifugation were employed as spice extracts. When the E. coli strains were incubated with each spice extract at concentrations of 0.01 per cent and 0.l per cent, a noteworthy difference was observed between the O157 and non-pathogenic strains towards their tolerance to nutmeg. The populations of the non-pathogenic strains could not be reduced, but those of the O157 strains were remarkably reduced.

Cytotoxic, Anticancer and Chemo Protective Effects

It is generally accepted that components that induce Phase I or Phase II drug metabolizing enzymes can protect the body against chemical carcinogenesis or damage, especially during the starting phase. Morita *et al.* (2003) studied hepatoprotective effect of myristicin from nutmeg on lipopolysaccharide/d-galactosamine-induced liver injury and concluded that essential oils could be utilized to protect body organs against carcinogenesis. Similarly, nutmeg showed a powerful hepatoprotective activity against liver damage caused by certain chemicals. The protective activity was correlated with myristicin, a major constituent. Recently it was found that myristicin induces cytotoxicity in human neuroblastoma SK-N-SH cells by an apoptotic mechanism i.e. eliminating unhealthy and unnecessary cells (Lee *et al.*, 2005).

Nutritional and Health Benefits

Burdock (1995) stated that specific gravity of nutmeg seed oil at 25°C falls within the range of 0.880 to 0.930, refractive index at 20°C varies between 1.4740 and 1.4880 and solubility is 1:3 in 90 per cent ethanol.

About 30-55 per cent of the nutmeg seed consists of oils and 45-60 per cent consists of solid matter including cellulose matter. Two types of nutmeg oil are present:

- Essential oil
- Fixed oil

The essential oil also called as volatile oil account for 5-15 per cent and fixed oil account for 24-40 per cent (Forrest and Heacock, 1972; Abdullah *et al.*, 2010). The relative percentages of the different components will vary depending on the geographical origin of the nutmeg.

Table 2.1 Nutriional value per 100 g of seed spice. (Agbogidi and Azagbaekwe, 2013).

Principle	Nutrient Value	Percentage of RDA
Energy	475 Kcal	24
Carbohydrates	50.50 g	39
Protein	6.71 g	12
Total Fat	32.38 g	162
Cholesterol	0 mg	0

Dietary Fiber	20.2 g	54
Vitamins		
Folates	76 µg	19
Niacin	1.350 mg	8
Pyridoxine	0.160 mg	12
Riboflavin	0.448 mg	34
Thiamin	0.312 mg	26
Vitamin-A	800 IU	27
Vitamin C	21 mg	35
Electrolytes		
Sodium	80 mg	5
Potassium	463 mg	10
Minerals		
Calcium	252 mg	25
Copper	2.467 mg	274
Iron	13.90 mg	174
Magnesium	163 mg	41
Manganese	1.500 mg	65
Phosphorus	110 mg	30
Zinc	2.15 mg	20

The major components of essential oil and their relative percentages are as follows:

Table 2.2 Major components of essential oil of nutmeg seed. (Takikawa *et al*., 2002).

Sl.No	Component	Percentage (per cent)
1.	Sabinene or Camphene	50
2.	α-Pinene	20
3.	Dipentene	8
4.	d-Linalool	6
5.	d-Borneol	6
6.	i-Terpineol	6
7.	Geraniol	6
8.	Myristicin	4

9.	Safrole	0.6
10.	Eugenol	2
11.	IsoEugenol	2

Health benefits

Nutmeg helps in getting rid of flatulence, diarrhea, and improves appetite as well. Nutmeg is helpful in clearing up the congestion resulting from cold and thus, is widely used in cough syrups. It is even helpful in aroma therapy (Gill, 1992; Iwu, 1993). One of the main properties of nutmeg is that it is helpful in stimulating the brain. It provides relief from stress and fuels mental activities as well. It can even boost concentration and assimilation rate as it is supposed to improve blood circulation to brain. However, make sure to take in a very little amount, as too much can cause delirium (Hallstrom and Thuvander, 1997). Nutmeg proves to be an excellent tonic for the cardiovascular system. It increases the blood circulation and stimulates the heart functions (Balick and Paul, 2000). Nutmeg oil is a great liver tonic, as it can remove the toxins therein. It is helpful in treating kidney infections and dissolves kidney stones also (Kasahara *et al*., 2005). Research shows that nutmeg can beat insomnia. It boosts the level of serotonin, which helps induce relaxation in turn (Pandey, 2005).

Nutmeg oil is helpful in treating bad breath (Barceloux, 2009). It is also antiseptic in nature and helps cure toothaches as well gum problems. It is because of this property that the oil is even used in many kinds of toothpaste (Duke, 1994; Osemene *et al*., 2013).

Medicinal use

The nutmeg oil is used as a local massage to reduce muscular pain and rheumatic pain of joints (Pamplona-Roger, 1999). Freshly prepared decoction with honey has been used to relief of nausea, gastritis and indigestion ailment (Dorman *et al.*, 2000). Nutmeg cures diarrhea, rheumatic pains; powdered seeds or decoction of the seeds are used in the treatment of diarrhea, carminative and rheumatism (Sofowora, 1993; Okoegwale and Omofezi, 2001). Since ancient times, nutmeg and its oil were being used in Chinese and Indian traditional medicine for illness related to the nervous and digestive systems. The compounds in these spices such as myristicin and elemicin are soothing as well as stimulant properties on brain (Maikhubu, 2006).

The powdered seed is added as flavouring agent to conceal the unpleasant taste/odor of several herbal preparations. The decoction of the nutmeg is used for the treatment of flatulence, nausea and vomiting. The oil of the nutmeg is rubbed over the stomach to relieve pain. Charred nutmeg is an excellent remedy for fevers and chills and the dose is generally half to one gram taken twice a day. Grated nutmeg mixed with Vaseline is applied externally to cure piles (Agbogidi and Azagbaekwe, 2013).

MICROWAVES

Datta and Anantheswaran (2000) stated that microwaves are the electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz with a corresponding wavelength ranging from 1 m to 1 mm. Domestic microwave appliances operate generally at a frequency of 2450 MHz, while industrial microwave systems operate at a frequency of 915 MHz and 2450 GHz. Microwaves are coherent and polarized in contrast to visible waves (apart from lasers). They obey the laws of optics and can be transmitted, absorbed or reflected depending on the type of material.

Guan *et al.,* (2011) described the other advantages of microwave food processing such as reduction in processing time, operational cost, product uniformity, ease of operation, low maintenance, very less change of flavor and nutritional change of food and protection from the surface browning and crusting due to heating from inside.

Chandrasekaran *et al.,* (2013) and Jermann *et al.,* (2015) reported that microwaves found vast applications in the field of food processing such as cooking, drying, pasteurization and preservation of food materials. Contrary to conventional thermal processing techniques heat is generated volumetrically throughout the product at faster rates. Solid and pumpable foods can be processed by means of microwaves effectively. This includes fluids containing large particles.

MICROWAVE HEATING

Kowalski (2013) studied changes of antioxidant activity and formation of 5-hydroxymethylfurfural (HMF) in honey during thermal and microwave processing. In this study, four types of honey (honeydew, lime, acacia and buckwheat) were analyzed. Honey samples were subjected to conventional heating in a water bath (WB) at 90°C up to 60 min or to the action of a microwave field (MW) with constant power of $1.26 W.g^{-1}$ of the sample up to 6 min. Changes in the antioxidant capacity of honeys were measured as a percentage of free radical scavenging ability. Changes in the total polyphenols content (TPC) (equivalents of gallic acid mg/100 g of honey) were also determined. Formation of HMF in honey treated with a microwave field was faster in comparison with the conventional process. It was found that the effect of a microwave field, although it greatly accelerates the formation of HMF, is suitable for honey processing because of the much shorter operation time.

Maria *et al.,* (2014) studied *L. monocytogenes* inactivation kinetics under microwave and conventional thermal processing in kiwi fruit puree. It was revealed that the level of microwave power applied had a considerable influence on the *L. monocytogenes*inactivation rate. The higher the microwave power level, the faster the inactivation. The inactivation of *L. monocytogenes*under microwave heating at 900 W ($D_{60°C}$ = 17.35 s) and 1000 W ($D_{60°C}$ = 17.04 s) happened faster than in a conventional thermal process ($D_{60°C}$ = 37.45 s). Consequently, microwave heating showed greater effectiveness for *L. monocytogenes*inactivation than conventional heating.

Maria *et al.,* (2015) performed a comparative study between microwave and conventional heat processing of kiwi fruit puree. In this study, the impact of microwave (1000 W – 340 s) and conventional heat (97ºC – 30 s) pasteurization and storage (4, 10, 22ºC for up to 63 days) on total and individual carotenoids and chlorophylls in kiwifruit puree was evaluated. Bio accessibility of carotenoids, before and after pasteurization and storage, was also studied. Microwaves and conventional heating led to noticeable changes in the chlorophyll (42–100 per cent losses) and carotenoid (62–91 per cent losses) content. First and second order kinetics properly explained the degradation of total carotenoids and chlorophylls over time, respectively. Pasteurized samples showed significantly ($p < 0.05$) enhanced stability of these pigments, with microwaves (k = 0.007 – 0.031 100 $g.mg^{-1}.day^{-1}$ at 4 to 22ºC) promoting chlorophyll stability to a greater extent than conventional heating (k = 0.0015–0.034 100 $g.mg^{-1}.day^{-1}$ at 4 to 22ºC). Bio accessibility of carotenoids remained ($p < 0.05$) unaffected by processing and storage. These results stressed that the pigment composition of microwaved kiwifruit was more similar to that of the fresh fruit and better preserved during storage.

Saritha *et al.,* (2015) conducted a study on influence of microwave energy on pectic principles of mango peel and concluded that microwave energy has been used for heating purposes which generates heat energy within short time periods. Also microwave energy can be exploited where rapid increase in temperature is desired. Maximum pectin yield could be obtained with a shorter heating period as compared to the conventional method of extractions which were reported earlier. Higher methoxyl content and viscosity were observed in the mango peel pectin extracted at 660 and 1000 W for 20 min indicating better gelling characteristics of the pectin. Yield of pectin was found to be maximum from the mango peel exposed to microwave energy of 1000 W for 20 min. Methoxyl content, viscosity and galacturonic acid decreased at 25 min of extraction at all microwave energy levels. Pectin extracted at the optimum conditions contained galacturonic acid, methoxyl content and viscosity of 57.2 g/100 g, 8.2 g/100 g and 98.2 mPa s, respectively.

*L. monocytogenes*is a pathogen of great concern in minimally processed because of its all-over presence and psychrotrophic nature, with a particular ability to multiply at low temperatures, low water activity levels, acidic pH, and also allowing it to reach levels high enough to cause human diseases. Microwave heating is one of the novel thermal technologies that can be used as an alternative in order to achieve or possibly enhance tomato juice shelf life, quality and nutrient content.Stratakos*et al.* (2015) evaluated the effect of an industrial scale continuous flow microwave volumetric heating system in comparison to conventional commercial scale pasteurization for the processing of tomato juice in terms of physicochemical properties, microbial characteristics and antioxidant capacity. Physicochemical and colour characteristics of juices were very similar between technologies and during storage. Both conventional and microwave pasteurization inactivated microorganisms and kept them in low levels throughout storage. Juice processed with the microwave system showed an increased cytoprotective effect against H_2O_2 induced oxidation in Caco-2 cells. Organoleptic analysis revealed that the two tomato juices were very similar. The continuous microwave volumetric heating system appears to be a viable alternative to conventional pasteurization.

Principle of Microwave Heating

Datta *et al.,* (2000) stated that microwave heating is caused due to the ability of the materials to absorb microwave energy and convert it into heat. Microwave heating of food materials mainly occurs due to dipolar rotation and ionic polarization mechanisms. The presence of moisture or water causes dielectric heating due to the dipolar nature of water. When an oscillating electric field is incident on the food materials, the water molecules which are permanently polarized dipolar molecules try to realign in the direction of the electric field. Due to the high frequency of 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.

Fan *et al.,* (2013) concluded that the effects of microwave heating on the ordered structures in starch granules were remarkably similar to the effects of rapid heating in an oil bath. The rate of heating determined the differences in the proportions of amorphous starch, double helices and V-type single helices, while the electromagnetic effects of microwave heating did not have a significant impact on the ordered structures in starch granules.

Bakibaev *et al.,* (2015) performed an experiment on polymerization of lactic acid using microwave and conventional heating and found that the process of obtaining polylactic acid (PLA) by microwave irradiation proceeds hundreds of times faster. PLA samples synthesized by this method have the same optical characteristics as the PLA obtained by conventional heating.

Liu and Lanier (2016) studied rapid (microwave) heating rate effects on texture, fat/water holding and microstructure of cooked comminuted meat batters concluding that the rapid heating can produce acceptable properties of water/fat holding and texture (fracture and small strain mechanical properties) in cooked comminuted meat batters (gels) of relatively high fat content.

Dielectric Properties of Food Material

The interest in the dielectric properties of agricultural materials and food products has concentrated primarily to predict heating rates describing the behavior of food materials when subjected to high frequency fields in dielectric heating applications, or so called novel thermal treatments (Venkatesh and Raghavan, 2004; Sosa-Morales *et al.,* 2010).

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

$$\varepsilon_r = \varepsilon_r' - j\varepsilon_r'' \qquad \text{.....(2.1)}$$

where ε_r' and ε_r'' are commonly called the dielectric constant and loss factor, respectively. The real part, the dielectric constant (ε_r'), describes the ability of a material to store energy when it is subjected to an electric field and influences the electric field distribution and the phase of waves travelling through the material. The imaginary part, the loss factor (ε_r''), influences both energy absorption and attenuation, and describes the ability to dissipate energy in response to an applied electric field or various polarization mechanisms, which commonly results in heat generation. The amount of thermal energy converted in the food is proportional to the value of the loss factor (Tang, 2005).

According to Sosa-Morales *et al*., (2009), other properties related to dielectric parameters are penetration depth and electrical conductivity. The penetration depth is usually defined as the depth into a sample where the microwave and RF power has dropped to 1/e (e=2.718) or 36.8 per cent of its transmitted value. The penetration depth is a function of ε_r' and ε_r''.

Llave *et al.*, (2016) observed the dielectric properties of tylose water pastes during microwave thawing and heating. Salt was confirmed as a good additive for increasing the dielectric loss factor; however higher salt addition leads to an increase in the thawing time and non-uniformity through decreased penetration depth.

MICROWAVE ASSISTED OIL EXTRACTION

Handa (2008) stated that the traditional methods of producing essential oils are hydro distillation (water distillation, water and steam distillation and direct steam distillation), expression, extraction with cold fat etc. The choice of a particular process for the extraction of essential oil is generally dictated by the following considerations:

a) Sensitivity of the essential oil to the action of heat and water

b) Volatility of the essential oil

c) Water solubility of the essential oil

Essential oils with high solubility in water and those that are susceptible to damage by heat cannot be steam distilled. Also, the oil must be steam volatile for steam distillation to be feasible. Most of the essential oils in commerce are steam volatile, reasonably stable to heat and practically insoluble in water; hence they are suitable for processing by steam distillation.

Hydro distillation (Method A) differs from steam distillation (Method B) mainly in that the plant material is almost entirely covered with water in the still which is placed on a furnace. An important factor to consider in water distillation is that the water present in the tank must always be enough to last throughout the distillation process, otherwise the plant material may overheat and char. In this method, water is made to boil and the essential oil is carried over to condenser with the steam which is formed. Water-distilled oil is slightly darker in color and has much stronger still notes than oils produced by other methods (Sudeep, 2008).

Though Method A and Method B are most commonly used they possess some disadvantages such as more time consuming for the process of extraction, compounds altering and degradation of compounds that takes place due to high temperatures, low oil yield and high energy consumption for the complete extraction process (Lucchesi *et al.,* 2004; Chen *et al.*, 2016).

Hong *et al.,* (2001) performed an experiment on microwave-assisted extraction of phenolic compounds from grape seeds. The microwave power (300-150 W) and time of extraction (20-200 s) were varied during the optimization process. The polyphenol content of the resulting extracts were measured as mg of tannic acid equivalent per gram of crude extract (mg TAE/g of crude extract), using a Folin-Ciocalteau reagent. The important observation is that when the solvent polarity was changed by the addition of 10 per cent water, the yield increased to 15.2 per cent and the polyphenol content increased to 429 mg TAE/g of crude extract.

Luque-Garcia *et al.,* (2002) proposed a new method for extraction of fat from prefried and fried meat and fish. A drastic reduction of the procedure time (55 min versus 8 h) is achieved with similar reproducibility to that provided by the conventional method. In addition, the proposed method is cleaner than conventional Soxhlet as 75–80 per cent of the extractant is recycled.

Chemat *et al.,* (2006) studied microwave accelerated steam distillation of essential oil (MASD) from lavender flowers. It was revealed that in steam distillation (SD) and MASD the extraction temperature is equal to water boiling temperature at atmospheric pressure (100ºC). To reach this temperature and thus obtain the distillation of the first essential oil droplet, it is necessary to heat only 5 min with MASD against 30 min for SD. As a result an extraction time of 10 min with MASD provides yields comparable to those obtained after 90 min by means of SD, which is one of the reference methods in essential oil extraction. The ultimate yield of essential oil obtained from lavender flowers was 8.86 per cent by MASD and 8.75 per cent by SD. The energy required to perform the two extraction methods are 1.5 kWh for SD and 0.13 kWh for MASD, respectively.

Chemat *et al.,* (2006) stated that extraction time in microwave assisted process was found to decrease with increase in temperature. This decrease could be attributed to the fact that with increase in temperature, the vapour pressure of water present inside the celery seeds increased leading to leaching out and evaporation of volatile oil along with water.

Lucchesi *et al.,* (2007) studied solvent-free microwave extraction (SFME) of cardamom essential oil. The results revealed that microwaves seem to cause the rupture of the cells and the glands more rapidly than in conventional hydro-distillation. When the glands were subjected to more severe thermal stresses and localized high pressures, as in the case of microwave heating, the pressure build-up within the glands could have exceeded their capacity for expansion, and caused their rupture more rapidly than in conventional extraction. Statistical treatment of the results revealed that the selected parameters: extraction time, irradiation power and moisture content of the seeds were significant. The essential oils were analyzed by gas chromatography–mass spectrometry (GC–MS). Essential oils provided by SFME are dominated by the oxygenated fraction which is the more valuable and composed of highly odoriferous aromatic compounds.

Golmakani *et al.,* (2008) compared the microwave-assisted hydro distillation (MAHD) with the traditional hydro distillation (HD) method in the extraction of essential oils from *Thymus vulgaris* L. The results showed that MAHD was superior in terms of saving energy and extraction time (75 min, compared to 4 h in HD). Scanning electron microscopy (SEM) of thyme leaves undergone HD and MAHD provided evidences as to a sudden rupture of essential oil glands with MAHD. The refractive indices, specific gravities and colour of essential oils obtained from thyme aerial parts for both MAHD and HD fall within the ranges specified by Food Chemical Codex (FCC). Gas chromatography–mass spectrometry analysis of the extracted essential oils indicated that the use of microwave irradiation did not adversely influence the composition of the essential oils.

Leslie and Maria (2011) developed a microwave assisted method with the aim of improving the extraction efficiency of Theobromine and Caffeine from cacao. The results showed that the microwave method was more efficient compared with a standard method and the extraction efficiency increased from 15 per cent to 72 per cent in case of

Theobromine and 36 per cent to 153 per cent in case of Caffeine. Also the method was found to be precise, fast and easy.

Desai and Parikh (2012) performed a comparative study on microwave assisted extraction of essential oil from the leaves of *Cymbopogon flexuosus* (Steud.) Wats. (Lemon grass). The effect of various parameters like solid loading, volume of water, rehydration time, extraction time, and power on yield and composition of essential oil was examined. Better quality was obtained for the essential oil extracted by MAE under the conditions of 20 per cent solid loading, 500 ml water, 1 h rehydration time, 45 min extraction time, and 850 W power. Yield of essential oil was found to be the same (1.04 per cent) for HD and MAE. HD required 90 min to treat 50 g of plant material with an energy consumption of 0.75 kWh while MAE was complete in 45 min by treating 100 g of plant material and using 0.6375 kWh. Thus, with reduced energy consumption and carbon footprints, MAE can be considered as a potential green method for extracting essential oil from the leaves of lemongrass. The essential oils extracted either by MAE or HD has almost similar chemical constituents; however, the percentage varies with respect to the technique employed. Citral is the main component found in essential oil extracted by either technique. A higher amount of citral (80.01 per cent) is present in oil extracted by MAE compared to that by HD (72 per cent).

Kiruba *et al.,* (2013) optimized the microwave assisted process for extraction of Phenolic antioxidants from grape seeds (*Vitis vinifera*) which are rich in phytochemicals that have antioxidant properties. The influence of independent variables such as microwave power (100, 150, and 200 W), extraction time (2, 4, and 6 min), and solvent concentration (30 per cent, 45 per cent, and 60 per cent ethanol) and their interactions on total phenols and the antioxidant activity (1,1-diphenyl-2-picrylhydrazyl (DPPH) and ferric ion reducing antioxidant power (FRAP)) were determined; and the microwave-assisted extraction (MAE) process was optimized using a central composite design. The total phenols that were expressed as gallic acid equivalents (GAE), catechin equivalents (CAT), and tannic acid equivalents (TAE) were significantly influenced by the solvent concentration and the time of extraction. The response variables were maximized for 6 min of MAE of grape seed (GS) with 32.6 per cent ethanol at 121 W with a desirability function of 0.947. The predicted extraction yields were 13±0.89, 21.6±1.59 and 15.9±1.32 mg GAE, CAT, and TAE, respectively per gram of GS. The predicted antioxidant activity per gram of dry weight GS was 80.9 per cent for the inhibition of DPPH and 135 μM ascorbic acid equivalents for FRAP test.

Baron and Villa (2014) studied microwave assisted extraction of essential oil and pectin from orange peel in different stages of maturity. The results showed that the essential oil extraction yield was slightly higher using additional water under the best extraction conditions (600 W, 10 min), and the limonene content, determined by GC-MS, was between 90.5 and 97.9 per cent. It was observed that at a power of 200 W, no essential oil was extracted and at microwave power higher than 600 W, the oil turned dark yellow or even black with the presence of suspended solid material. Scanning electron microscopy (SEM) analyses after essential oil extraction showed that the intracellular spaces of the plant tissue increased with time under microwave irradiation. Without solvent, the orange peel was carbonized when the microwave power was higher than 600 W. The content of essential oil decreased with the maturity (0.14 to 0.08 per cent).

With increase in microwave power and decrease in solvent the peels begun to carbonize and further increase in time leads to completely charred and black coloured sample. Also, with increase in solvent the pectin extraction yield also has got enhanced (Kratchanova *et al.*, 2004; Baron and Villa, 2014).

Gopika and Ghuman (2014) developed a microwave assisted extraction method for extraction of essential oil from celery seeds. A domestic microwave oven was modified and Clevenger apparatus attached to it to make it an extraction unit. Effect of various parameters such as soaking time, temperature and power density during MAE was studied. A multivariate study based on a Box-Behnken design was used to evaluate the influence of three major variables (soaking time, temperature and power density) affecting the performance of MAE on celery seed. Oil yield, time of extraction and energy consumption ($MJ.kg^{-1}$ oil) by MAE were determined and compared with those obtained by the traditional hydro-distillation (HD). It was found that microwave assisted process gave approximately same oil yield (1.90 per cent) in less time (93.5 min) and with low energy consumption (58191.78 $MJ.kg^{-1}$ oil). Results revealed that the selected parameters had significant effect on the responses.

Also, the results revealed that the lower yield of oil extracted at 90ºC might be due to the temperature being not enough to burst open the oil glands. Oil yield was also lower at 110ºC because evaporation rate was higher than the condensation rate. Also, soaking time was found to have significant effect on oil yield. Increase in soaking time, leads to increase in pressure inside the seeds till bursting of outer layer took place. This bursting led to release of oil, which increased the oil yield when compared to conventional hydro-distillation process. With increase in soaking time, oil yield decreased to a point of minima at 8 h. With further increase in soaking time, oil yield increased, but to a lesser value than at 4 h.

Avelina *et al.*, (2016) performed an experiment to evaluate the effect of different microwave assisted extraction (MAE) process parameters in the extraction yield of orange peel essential oil. Results demonstrated that particle size, moisture content and its interaction significantly affected ($p < 0.05$) the yield obtained and had an influence on the extraction mechanism. The yield of oil during microwave assisted process is more by 0.9 per cent than oil obtained by hydro distillation process. Besides, the process reduce the processing times. The results demonstrate the significant effect ($p < 0.05$) of initial moisture content and particle size on essential oil yield. In the case of particle size, the decrease in size improved the extraction and this can be related to an increase in the superficial area which promotes a better contact of the sample with the solvent and penetration of microwaves.

High moisture content improves the extraction recovery in most cases, due to microwaves interacting selectively with the free water molecules present in the gland and vascular system, leading to rapid heating and temperature increase, followed by rupture of walls and release of the essential oil into the solvent (Letellier and Budzinski, 1999; Avelina *et al.*, 2016;). But Ferhat *et al.* (2009) stated that during lavender flowers essential oil extraction by microwave steam diffusion did not find difference in the oil yield observing that the only effect in the process was the time needed for essential oil extraction.

COMBINATION TECHNOLOGIES

Though microwave heating was effective for reduction of come-up-times and was less sensitive to food heterogeneities, a major problem associated with microwave heating was localized heat zones related with the variation in dielectric, physical and thermal properties of food components. Microwave heating assisted with conventional heating methods such as vacuum and microwave absorbents was advantageous to diminish localized heat zones in foods.

You *et al.,* (2007) performed an experiment to determine triazines in infant nutrient cereal-based foods by pressurized microwave-assisted extraction (PMAE) coupled with high-performance liquid chromatography and mass spectrometry. The recoveries increased from 66.2 to 88.6 per cent by using PMAE. Compared with atmospheric pressure microwave-assisted extraction (AMAE), ultrasonic extraction (UE) and soxhlet extraction (SE), the proposed method was more efficient, faster and more straightforward and required no additional cleanup steps. When the proposed method was applied to the aged spiked nutrient cereal samples, the results indicated that, although the recoveries of analytes were much lower than those obtained from fresh spiked samples, they were nevertheless satisfactory for the quantitative analysis of practical samples. The highest recoveries were obtained in the time ranging from 8 to 10 min, while low recoveries were obtained when the extraction time is shorter than 8 min and longer than 10 min. On the one hand, the low recoveries at short irradiation time might result from insufficient microwave energy, which can be available to attain the temperature of phase change and hence enable the breaking of the analyte–matrix bonds or might result from the strong adsorption of the analytes on the sample particle surface. Also long extraction times can cause degradation of the thermo liable compounds.

Nguyen *et al.,* (2013) designed and fabricated a continuous flow simultaneous microwave and ohmic combination heater to heat treat particulate foods without leaving solids under processed. The results showed that maximum solid-liquid temperature differences under microwave and ohmic heating were about 8.1 and 8.0ºC, respectively. However, when microwave and ohmic heating techniques were applied simultaneously, there was no significant temperature difference between solid and liquid phases. Energy efficiency of combination heating was higher than microwave heating and a maximum increase in energy conversion of 12.8 per cent was obtained.

Lee *et al.,* (2015) developed a dual cylindrical microwave and ohmic combination heater for minimization of thermal lags in the processing of particulate foods. Results showed that particle size and salt concentration affected temperature variations between solution and particulates in ohmic heating. For microwave heating, the solution temperature lagged behind the particle temperature up to 12.5 g/l salt concentrations, regardless of particle size and mass fraction; however, an opposite tendency was observed in the food mixtures including 20 g/l salt concentration. The maximum temperature differences between particles and solution obtained by individual microwave and ohmic heating were 7.1 ± 1.7 and 11.9 ± 2.9ºC, respectively, while the combination heating exhibited little significant temperature gaps (maximum difference <3.08ºC) at 12.5 g/l salt concentrations.

Samani *et al.* (2015) analyzed the combinative effect of ultrasound and microwave power on *Saccharomyces cerevisiae* in orange juice processing. It was found that conventional heat pasteurization of orange juice sometimes results in an off flavour due to overheating of the juice at the heat-exchange surface. Heating with microwave heats the juice uniformly without changing the taste. Also, it causes the complete inactivation of bacteria and pectin methyl esterase was obtained. There was no adverse effect on juice flavour. Also the appearance of orange juice in the combinative method was better than those of conventional method (57 % vs. 43 %).

Chen *et al.,* (2016) studied a two stage microwave extraction of essential oil and pectin from pomelo peels and stated that microwave can enhance the extraction process by two distinct mechanisms: one attributes to the diffusion across the intact oil gland while the other involves the convection through the broken oil gland. As far as stability of essential oil is concerned the usage of extreme extraction condition, especially high temperature brought negative effects such as thermal degradation of essential oil. Also prolonging extraction time would be helpful to complete extraction of target compounds when microwave power remained low. The percentage of limonene increased with increasing microwave power at low microwave powers of 150 and 300 W, but decreased in high microwave power of 450 W. The findings suggested that microwave extraction at low microwave power may be a promising and effective technique for the extraction of essential oils because of its higher yield and better quality of essential oils when compared with hydro distillation (HD).

Fangyuan *et al.,* (2016) studied cyclodextrin based ultrasonic assisted microwave extraction. They concluded that the presence of cyclodextrin or ethanol significantly increased the extraction efficiency of the analytes. Secondly, ultrasound assisted microwave extraction provided the highest extraction yields demonstrating that ultrasound and microwave are crucial parameters in the extraction efficiency. UAME extracts compounds from herbal matrices in very short periods of time through the synergistic effect of acoustic effects and microwave radiation.

MATERIALS AND METHODS

CHAPTER 3

MATERIALS AND METHODS

This chapter describes the developed system of a microwave assisted process for extraction of nutmeg seed essential oil. The materials used for fabrication of the various components and the instrumentation employed for measurements of parameters were explained. The optimization of process parameters for extraction of nutmeg seed essential oil with maximum oil yield, minimum time and energy consumption and the methods for determining the physical and chemical properties of essential oil were detailed.

3.1 DEVELOPED SYSTEM OF MICROWAVE ASSISTED UNIT FOR EXTRACTION OF ESSENTIAL OIL

Based on a thorough review of works carried out on microwave assisted oil extraction, the design of a small capacity oil extraction unit assisted by microwave was conceptualized, further refined and then fabricated at KCAET ,Tavanur (Nukasani, 2016). The developed experimental system as shown in Fig 3.1 and Plate 3.1 consists of the following main components:

1. Microwave cavity
2. Extraction unit
3. Supporting stand
4. Energy meter

3.1.1 Microwave Cavity

The prime requirement for the microwave assisted extraction process is a microwave source. Commercially available microwave ovens could be effectively utilized for this purpose. The selection of microwave oven should be based on the power consumption. For laboratory scale experiments, ovens with maximum power delivery of 1000 W were generally chosen (Chemat *et al.,* 2006; Lucchesi *et al.,* 2007; Jiao *et al.,* 2012). Accordingly, a microwave oven with (Model: Magicook MW20BC) with following specifications was used to serve as the microwave source.

Power consumption	230 V/50 Hz, 1200 W (Microwave)
	1100 W (Grill)
	2000 W (Convection)
Operation frequency	2450 MHz
Outside dimensions	262 mm(h) x 452 mm(w) x 395 mm(D)
Oven cavity dimensions	195 mm(h) x 315 mm(w) x 325 mm(D)
Oven capacity	20 litres
Cooking uniformity	Turntable system

The oven consists of a control panel where cooking time, power, action indicators and clock time are displayed and controlled. The oil is extracted by micro mode since the temperatures are low at this mode. The power could be increased in steps of 10’s such as P-70, P-80, P-100 etc. in which P-100 indicates that oven utilizes 100 per cent of its rated power i.e. 1200 W for extraction of oil. The time for extracting the oil can be set by pressing the time button on the control panel.

Water outlet

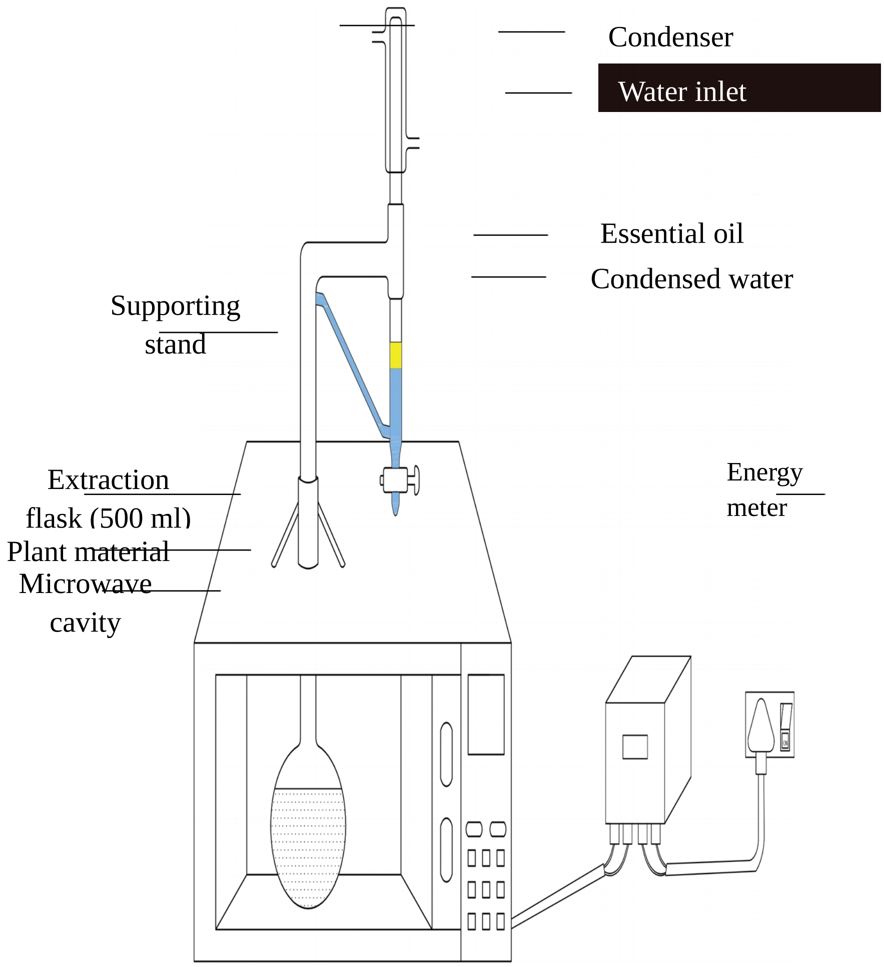


Figure 3.1. Schematic of the microwave assisted extraction unit

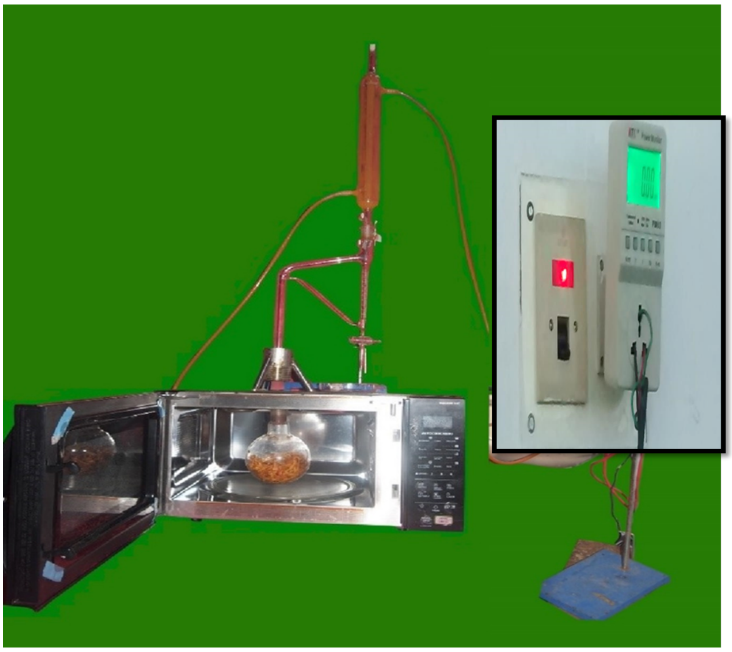


Plate 3.1. Developed microwave assisted extraction unit (Nukasani, 2016).

3.1.2 Extraction Unit

The Extraction unit consists of a Clevenger hydro distillation system in which recycling of distilled water also takes place. Clevenger hydro distillation system consists of a round bottomed flask, Clevenger and a condenser (Plate 3.2). The capacity of the round bottom flask match with the dimension of the microwave cavity because of its ease in inserting and removing the flask while loading and unloading the plant material (Chen *et al.*, 2016). A hole of ½ inch diameter was drilled on the top of the microwave cavity in order to fix the condenser into the round bottom flask placed in the cavity. The round bottomed flask and the condenser was connected by means of a glass tube (1/2 inch diameter) and two rubber corks. Half portion of glass tube is projected upwards and remaining half portion is inside the oven cavity holding the round bottomed flask. The glass tube acts as carrier of vapors, both water and essential oil.

3.1.3 Supporting Stand

The supporting stand is fabricated by using stainless steel material. It is placed outside the oven for supporting the glass extraction unit. The stand comprises of a circular ring and three stainless steel pipes which acts as supporting legs for the ring. The circular ring has an outer diameter of 48 mm and inner diameter of 43 mm. The height of the circular ring is 69 mm. The three pipes each of diameter 12.7 mm and length 80 mm is welded to the circular ring. For keeping the distillation unit straight without tilting a silicon rubber cork was inserted into the circular ring through which the glass stem passes. The inner and outer diameters of the cork are 23 mm and 43 mm, respectively with a cork length of 20 mm. The supporting stand is shown in (Plate 3.3).

3.1.4 Energy Meter

A digital type energy meter was connected to the microwave assisted extraction system to measure the energy consumed during the distillation process. The energy consumed for microwave assisted extraction process at different process levels as per the experimental design and for conventional hydro distillation process were measured for comparison of the energy efficiency of the microwave assisted process.

Plate 3.2. Components of the extraction system

Plate 3.3. Supporting stand

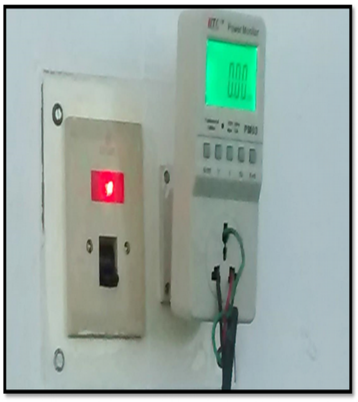


Plate 3.4. Energy meter

3.2 PHYSICAL QUALITY CHARACTERISTICS OF OIL

3.2.1 Refractive Index

The refractive index of a transparent liquid was determined by the method of reflected system of Newton's rings for which Newton's rings apparatus, Sodium vapour lamp, and Vernier microscope were used (Plate 3.5). The Newton's rings apparatus consists of an optically plane glass plate P on which is placed a convex lens L of large focal length. Above the lens, another glass plate G is arranged at 45^0 to the horizontal. The complete set up is shown in Plate 3.6. When the lens is placed over the glass plate, the space between the lens and glass plate contains 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 λ is the wavelength of the light used and R is 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,

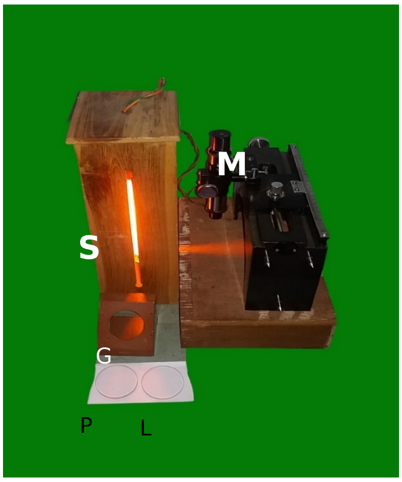
$$n = D_{m+k}^2 - D_m^2 / D_{m+k}'^2 - D_m'^2 \qquad \text{…. (3.1)}$$

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^o to the horizontal, gets reflected and then fall normally on the convex lens L placed over the glass plate P. Systems of bright and dark concentric circular rings are observed through a microscope M, arranged vertically above the glass plate G as shown in Plate 3.7. The microscope is properly focused so that alternate bright anddrk circular the rings are seen clearly. By working its fine adjustment screw of the microscope make sure that there are about 25 clear rings on either side of the centre. Starting from the centre of the fringe system, the microscope is moved towards the left so that the cross-wire is tangential to the m^{th} (say 20^{th}) dark ring. The microscope reading on the horizontal scale was taken. By working the fine adjustment screw, the microscope was moved towards the right. The cross-wire was adjusted to be tangential to the 18^{th}, 16^{th} etc. dark ring is succession 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 2^{nd}, 4^{th}

…. 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 is 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.

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



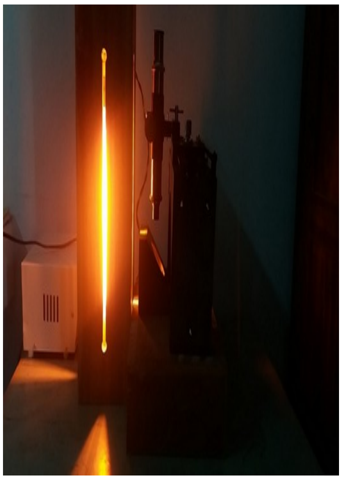


Plate 3.5. Newton's rings apparatus   Plate 3.6. Refractive index experimental set up

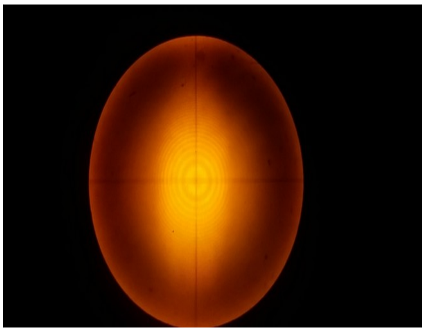


Plate 3.7. Circular rings

3.2.2 Specific Gravity

Specific gravity was calculated by dividing the weight of 1 ml essential oil by the weight of 1 ml distilled water. Weights were measured using a balance with an accuracy of 0.001g (Gopika and Ghuman, 2014).

3.2.3 Solubility Test

The solubility of nutmeg essential oil was determined based on the procedure suggested by Food Chemical Codex (FCC, 1996). One ml sample of nutmeg seed 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 25ºC, and the contents mixed thoroughly after each addition of alcohol. The dilution procedure was continued until a clear mixture was obtained. The volume of alcohol (V) used to obtain a completely clear solution was recorded. Once the clear solution was obtained, the dilution process was continued, but with 0.5 ml 85 per cent ethanol until the volume of alcohol added was 20 times the volume added earlier. The solution was thoroughly shaken each time with 0.5 ml ethanol until no turbidity was observed. The results were expressed as ‘‘one volume of essential oil soluble in V volumes or more of 85 per cent ethanol”.

3.3 EXPERIMENTAL DESIGN

Based on a thorough review of literature and the preliminary studies conducted, the process parameters which would influence the nutmeg seed essential oil yield, extraction time and energy consumption were chosen as independent variables. The physical quality characteristics which are characteristics of these parameters were selected as dependent variables.

3.3.1 Independent Variables

a) Solid : Water ratio (S):

1) S_1 : 2:1

2) S_2 : 4:3

3) S_3 : 1:1

b) Microwave power density (P):

1) P_1 : 7.2 $W.g^{-1}$

2) P_2 : 8.4 $W.g^{-1}$

3) P_3 : 9.6 $W.g^{-1}$

c) Soaking time (T):

1) T_1 : 2 h

2) T_2 : 3 h

3) T_3 : 4 h

3.3.2 Dependent Variables

Microwave assisted extraction system output parameters:

a) Essential oil yield
b) Extraction time
c) Energy consumption

Physical quality characteristics of nutmeg essential oil:

a) Refractive index
b) Specific gravity
c) Solubility

3.4 STATISTICAL ANALYSIS

Unlike conventional empirical optimization, the statistical optimization method can take into account the interaction of variables in generating the process response. Response Surface Methodology (RSM) was adopted in the experimental design as it emphasizes the modeling and analysis of the problem in which response of interest is influenced by several variables and the objective is to optimize this response (Montgomery, 2001). The main advantage of RSM is to reduce number of experimental runs needed to provide sufficient information for statistically acceptable results. Three variables i.e. solid: water ratio, power density and soaking time at three levels were chosen based on preliminary trials conducted prior to final experimentation. A Box–Behnken design of three variables and three levels, each with three centre point combinations, was used (Box and Behnken, 1960). This design was taken as it fulfilled most of the requirements needed for optimization of the microwave assisted process. In the above design the three levels of the process variables were coded as -1, 0, +1 and designed in coded X form (Gopika and Ghuman, 2014). The values of independent variables at three levels were shown in Table 3.1.

According to Box-Behnken design for three independent factors, the total experiments to be conducted are found to be seventeen. Thirteen experiments were performed with three variables and three levels of each variable as shown in Table 3.2.

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

Independent variable	Symbol		Level	
	Coded	**Un-coded**	**Coded**	**Un-coded**
Solid : water ratio	X_1	S	-1	2:1
			0	4:3
			1	1:1
Power density ($W.g^{-1}$)	X_2	P	-1	7.2
			0	8.4
			1	9.6
Soaking time (h)	X_3	T	-1	2
			0	3
			1	4

Table 3.2. Experimental design used for extraction of MAE of nutmeg seed essential oil.

Standard Order	Run	Coded variables			Un-coded variables		
		Solid:Water ratio	Power density ($W.g^{-1}$)	Soaking time (h)	Solid: Water ratio	Power density ($W.g^{-1}$)	Soaking time (h)
1	4	-1	-1	0	2:1	7.2	3
2	5	1	-1	0	1:1	7.2	3
3	14	-1	1	0	2:1	9.6	3

4	7	1	1	0	1:1	9.6	3
5	12	-1	0	-1	2:1	8.4	2
6	17	1	0	-1	1:1	8.4	2
7	8	-1	0	1	2:1	8.4	4
8	10	1	0	1	1:1	8.4	4
9	1	0	-1	-1	4:3	7.2	2
10	2	0	1	-1	4:3	9.6	2
11	15	0	-1	1	4:3	7.2	4
12	13	0	1	1	4:3	9.6	4
13	6	0	0	0	4:3	8.4	3
14	9	0	0	0	4:3	8.4	3
15	3	0	0	0	4:3	8.4	3
16	11	0	0	0	4:3	8.4	3
17	16	0	0	0	4:3	8.4	3

3.5 EXPERIMENTAL PROCEDURE

In order to evaluate the developed system towards extraction of essential oil, fresh nutmeg seed collected from local farmer was used. The detailed procedure for extraction of nutmeg mace essential oil employing the microwave assisted process is detailed below.

3.5.1 Determination of Moisture Content

Moisture content of nutmeg seeds is determined by Dean and Stark Toluene Distillation method as per AOAC (2000).

3.5.2 Extraction of Essential Oil

The desirable amount of nutmeg seed i.e. 100 g is soaked for a various period of time as per the experimental design. The soaked sample is filled in round bottomed flask of the extraction unit. The microwave power level along with time is set in the control panel for various treatment conditions and the set up was switched on. The set power level, the microwaves heat the plant material up to set running time. During this process the vapors of water as well as essential oil in seed gets vaporized and passes out of the microwave cavity through the distillation stem into the condenser. These vapors then passed through the condenser where they gets condensed and falls back into the bottom of the extraction unit where the oil and water gets separated and oil which is lighter than water settles on the top and water which is denser settles on the bottom. After completion of process the oil is collected by means of a stopper provided on the extraction unit. The water is drained off and the essential oil thus collected is dehydrated with anhydrous sodium sulphate and stored at 2ºC in amber coloured glass bottles (Plate 3.9) for further analysis.

Conventional method of extraction i.e. Hydro distillation was performed using Clevenger apparatus as control for comparing the microwave assisted process. In the round bottomed flask of Clevenger apparatus, 50 g of sample and 500 ml of distilled water was taken and the whole set up including (Clevenger tube and condenser) was placed on a heating mantle (Desai and Parikh, 2012). The temperature of the heating mantle was maintained at 100ºC. The experiment was performed until complete extraction of essential oil from plant material is obtained.

Plate 3.8. Dean and Stark apparatus

Plate 3.9. Amber colored glass bottles

3.6 DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS OF NUTMEG SEED ESSENTIAL OIL

3.6.1 Refractive Index

The refractive index of the seed essential oil was determined using reflected system of Newton's rings (Plate 3.6) as explained in section 3.2.1.

3.6.2 Specific Gravity

The specific gravity of the seed essential oil was determined using the procedure explained in section 3.2.2.

3.6.3 Solubility

The solubility of the seed essential oil in 85 per cent (v/v) ethanol was determined using the procedure explained in section 3.2.3.

3.7 DETERMINATION OF CHEMICAL CONSTITUENTS

The main aromatic constituent of the nutmeg oil is myristicin. The presence of myristicin and its quantification is taken as a parameter for determining the quality of the extracted oil in international market. In this study the myristicin content of the essential oil extracted through microwave assisted process and hydro distillation process were determined using Gas chromatography (Shimadzu GC-17A, Japan) (Plate 3.10) with a column (30 m in length x 0.25 mm inner dia. x 0.25 µm film thickness), a flame injection detector with an operating temperature of 280ºC and an injector with a temperature of 250ºC, manual injection and nitrogen as gas carrier. The maximum temperature that can be attained in the equipment was 350ºC. The procedure for finding the myristicin content was adopted from Essam and Maytham (2012) and Ester *et al.* (2013). Myristicin standard was obtained from M/s.Sigma, St. Louis, MO, USA.The standard solution was first injected and the chromatograph of the standard was obtained. Then the sample was injected and its chromatograph was recorded following the same procedure. The injection was made with an initial split ratio of 1:30 with the injection port temperature of 250ºC. The initial temperature was set to 100ºC with an initial 1.0 min hold followed by programmed temperature (increment) at the rate of 15ºC/min to 60ºC followed by holding at the rate of 60ºC/ 5 min to 280ºC. The chromatographs were then analyzed for myristicin content.

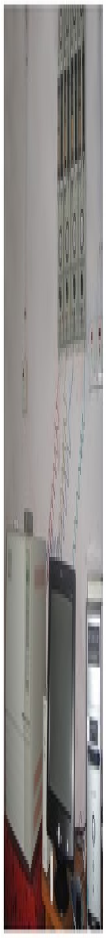


Plate 3.10. Gas chromatograph

RESULTS AND DISCUSSIONS

CHAPTER 4

RESULTS AND DISCUSSIONS

This chapter outlines the results on evaluation of developed system towards extraction of nutmeg seed essential oil. The outcomes of the procedures laid out for the evaluation leading to the standardization of the main process parameters are discussed in detail. Also, the effect of various process variables on the physical and chemical quality characteristics of the extracted essential oil through microwave assisted process are analyzed, discussed and compared with conventional extraction process.

4.1 DETERMINATION OF MOISTURE CONTENT

The moisture content of the fresh nutmeg seed which was collected from local farmer was determined by using Dean and Stark distillation method as explained in section 3.5.1. The average moisture content of nutmeg seed was found to be 14.5 per cent (wb).

4.2 STANDARDIZATION OF THE PROCESS PARAMETERS OF THE MICROWAVE ASSISTED EXTRACTION SYSTEM

In order to evaluate the developed system towards extraction of nutmeg seed essential oil and for optimization of the process parameters, a series of experiments with solid: water ratios of 2:1 ,4:3 and 1:1, power densities 7.2, 8.4 and 9.6 $W.g^{-1}$ and soaking times 2 h, 3 h and 4 h as input variables were performed. The experiments were performed as per the experimental procedure laid out in section 3.5. The results of the experiments conducted towards the microwave assisted extraction process with mean values of extraction time, oil yield and energy consumption are tabulated in Table 4.1.

Table 4.1. Effect of process variables towards extraction of nutmeg seed essential oil

Sl. No.	Sample	Extraction time (h)	Oil yield (ml)	Energy consumption (kWh)
1.	$S_1 P_1 T_2$	3.2	3.6	1.39
2.	$S_3 P_1 T_2$	3.9	3.1	1.1
3.	$S_1 P_3 T_2$	3.2	3	1.39
4.	$S_3 P_3 T_2$	3.5	3.5	1.49

5.	$S_1 P_2 T_1$	3.1	3.6	1.18
6.	$S_3 P_2 T_1$	3.26	3.3	1.19
7.	$S_1 P_2 T_3$	3	3.5	1.1
8.	$S_3 P_2 T_3$	3.3	4.1	1.4
9.	$S_2 P_1 T_1$	3.33	3.5	1.42
10.	$S_2 P_3 T_1$	3.2	4	1.39
11.	$S_2 P_1 T_3$	3	3.2	1.1
12.	$S_2 P_3 T_3$	4	3.6	1.47
13.	$S_2 P_2 T_2$	3.1	3.4	1.19
14.	HD	7	3.5	2.5

For optimizing the parameters, the results obtained in Table 4.1 were used as responses and listed as per the order mentioned in design, as explained in section 3.4. Only fourteen experimental data were used in the design to optimize the parameters as per response surface methodology

4.4 OUTPUT CHARACTERISTICS OF MAE SYSTEM

4.4.1 Extraction Time

The time taken for extracting nutmeg seed essential oil during various combinations of process parameters are presented in Table 4.1. The extraction time varied between 3 to 4 h. The minimum time for extracting maximum amount of nutmeg seed essential oil was obtained when the solid: water ratio is 2: 1, soaking time of 4 h and power density of 8.4 $W.g^{-1}$.

Response surface methodology was used to enquire the relationship between the independent and dependent variables.

4.4.2 Oil Yield

The yields of nutmeg seed essential oil obtained in various combinations of experiments are shown in Table 4.1. The total yield of oil varied from 3 to 4.1 ml. The maximum oil yield was obtained for a solid: water ratio of 2:1, power density of 8.4 $W.g^{-1}$ and soaking time of 4 h.

4.4.3 Energy Consumption

Energy consumption for extracting nutmeg seed essential oil obtained in various combinations of experiments are shown in Table 4.1. The energy consumption varied between 1.1 and 1.49 kWh. The least energy consumption was obtained for solid: water ratio of 2:1, power density of 8.4 $W.g^{-1}$ and a soaking time of 4 h.

4.5 DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS

The physical quality characteristics of the nutmeg seed essential oil extracted using conventional hydro distillation (HD) method and microwave assisted extraction (MAE) method are listed in Table 4.2.

Table 4.2. Physical quality characteristics of nutmeg seed essential oil

Sl. No.	Sample	Refractive index	Specific gravity	Solubility (v/v)
1.	$S_1 P_1 T_2$	1.470	0.7	0.6
2.	$S_3 P_1 T_2$	1.484	0.7	0.6
3.	$S_1 P_3 T_2$	1.477	0.9	0.8
4.	$S_3 P_3 T_2$	1.482	0.6	0.7
5.	$S_1 P_2 T_1$	1.482	0.6	0.8
6.	$S_3 P_2 T_1$	1.479	0.8	0.9
7.	$S_1 P_2 T_3$	1.465	0.7	0.5
8.	$S_3 P_2 T_3$	1.481	0.8	0.6
9.	$S_2 P_1 T_1$	1.466	0.8	0.8
10.	$S_2 P_3 T_1$	1.476	0.7	0.6
11.	$S_2 P_1 T_3$	1.475	0.7	1.0
12.	$S_2 P_3 T_3$	1.482	0.6	0.6
13.	$S_2 P_2 T_2$	1.464	0.6	0.6
14.	HD	1.479	0.7	1.5

4.5.1 Refractive Index

The values of refractive index of nutmeg seed essential oil obtained in various experiments are shown in Table 4.2. The values of refractive index varied from 1.464 to 1.484.

4.5.2 Specific Gravity

The specific gravity of nutmeg seed essential oil obtained in various experiments were tabulated in Table 4.2. The values of specific gravity varied between 0.6 and 0.9.

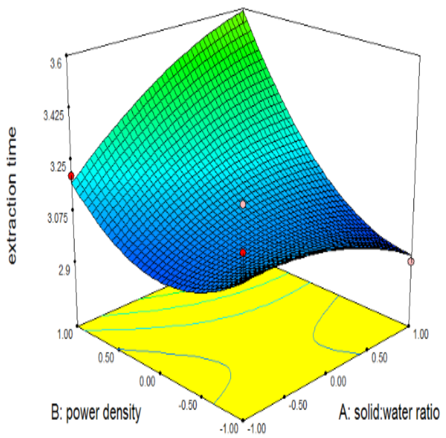
4.5.3 Solubility

The solubility of nutmeg mace essential oil obtained in various experiments in 85 per cent ethanol were tabulated in Table 4.2. The values of solubility varied from 0.5 to 1.5 v/v. The solubility values obtained in MAE nutmeg seed essential oil were in close relation with HD nutmeg seed essential oil.

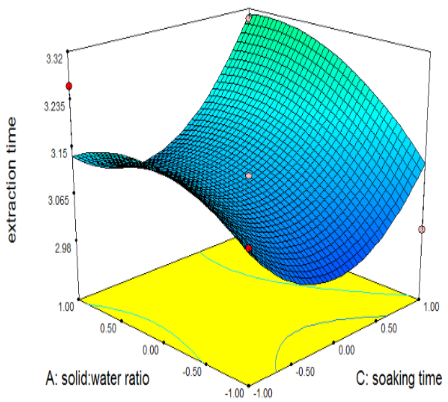
4.6 OPTIMIZATION OF PROCESS PARAMETERS

4.6.1 Effect of Process Parameters on Extraction Time of Oil

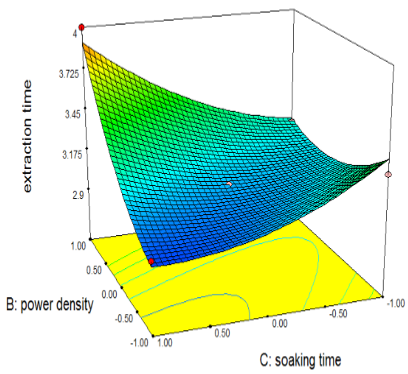
The relationship between independent (Solid: water ratio, power density and soaking time) and dependent variables are illustrated by plotting 3D graphs representing the response (extraction time) surface generated by the model. The 3D responses were shown in Fig. 4.1, comprising of three graphs a, b and c.



a



b



c

Figure 4.1. Effect of process parameters on extraction time

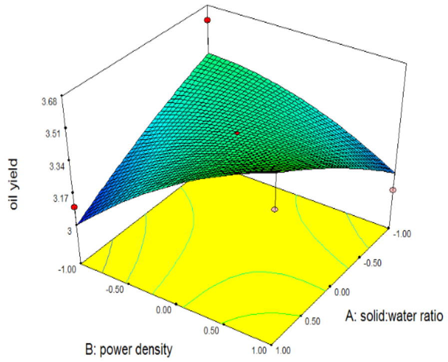
It is perceived from the Fig. 4.1 (a) and (b) that as solid: water ratio increases the total extraction time decreases. The total time of extraction varied from 3 h to 4 h. The least time consumption i.e. 3h was obtained when the experiments were performed with a solid: water ratio of 2:1. It may be observed from the Fig. 4.1 (b) and (c) that as soaking time increases from 2 h to 4 h, there is only a slight decrement in the total extraction time. This indicates that soaking time has an insignificant effect on the total time of extraction. This was also supported by the surface plot in Fig. 4.1 (b) and (c) showing soaking time effect by a straight line. Similar findings were also reported by Nukasani *et al*.,(2016) for nutmeg mace essential oil.

Also, from Fig. 4.1 (a) and (c) it is concluded that at low power densities the extraction time was found to be high. As power density increases the total extraction time increases to a certain point and then gets decreased. The decrement in extraction time is due to the fact that, with increase in power level, the vapour pressure of water present inside the nutmeg seed increased leading to leaching out and evaporation of volatile oil along with vapour (Chemat *et al.*, 2006).

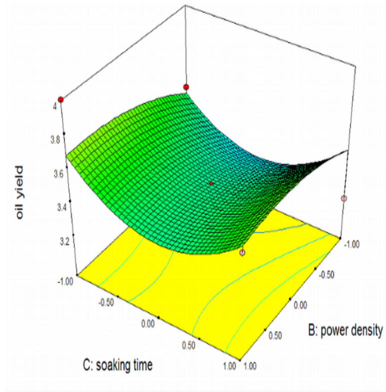
When compared with conventional hydro distillation process, the total time taken for extracting nutmeg seed essential oil from 50 g of sample was found to be 3 h. Whereas for the same oil yield hydro distillation process took 7 h. Therefore, it could be inferred that microwave assisted extraction was superior in terms of saving extraction time.

4.6.2 Effect of Process Parameters on Oil Yield

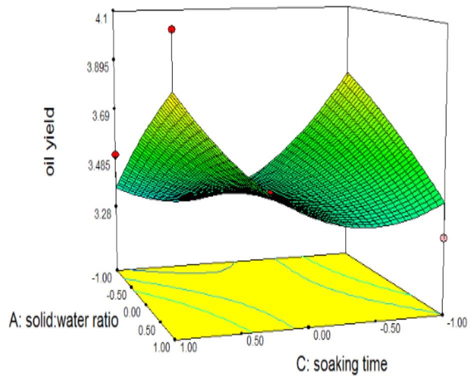
The relationship between Solid: water ratio, power density and soaking time on total yield of oil is illustrated by plotting 3D graphs representing the response surface generated by the model. The 3D responses were shown in Fig. 4.2, (a), (b) and (c).



a



b



c

Figure 4.2. Effect of process parameters on oil yield

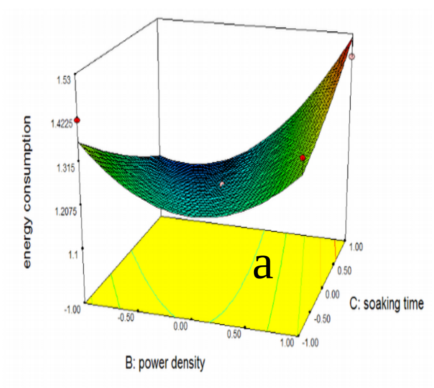
From Fig. 4.2 (a) and (c) it is concluded that power density has a significant effect on total yield of oil. At a low power density of 7.2 W.g^{-1} the essential oil yield was found to be less. This trend might be due to the temperature being not enough to burst open the oil glands. With increase in power density, total yield of oil increased to a maximum of 4.1 ml at a power density of 8.4 W.g^{-1}. Further increase in power density leads to a decrease in the yield of essential oil. This decrease in yield of oil at higher power density is due to higher evaporation rate of essential oil than the condensation rate (Desai and Parikh, 2012).

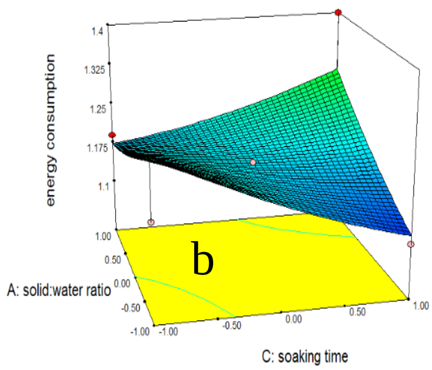
Fig. 4.2 (b) and (c) shows that soaking time has a significant effect on the yield of essential oil. Increase in soaking time leads to an increase in the yield of essential oil up to a period of 3 h. Beyond 3 h there was not much effect in the yield of oil. Increase in soaking time leads to an increase in pressure inside the seed resulting in bursting of outer layers. This bursting leads to release of oil, which increased the oil yield when compared to conventional hydro distillation process (Lucchesi *et al.*, 2007; Gopika and Ghuman, 2014).

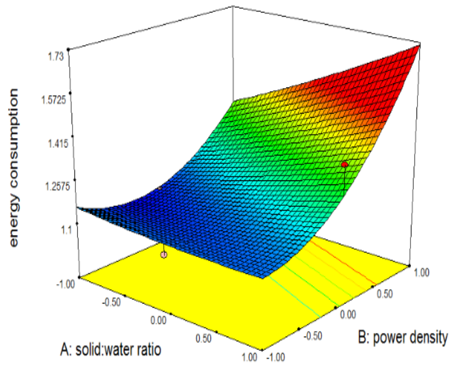
From Fig. 4.2 (a) and (b), it may be inferred that solid: water ratio had an insignificant effect on total yield of essential oil and showing solid: water ratio effect by a straight line. When compared with conventional hydro distillation, the total yield of oil obtained in both the process were almost similar (Desai and Parikh, 2012; Chen *et al.*, 2016). About 2-4 per cent essential oil found in nutmeg seed through both process.

4.6.3 Effect of Process Parameters on Energy Consumption

The relationships of Solid: water ratio, power density and soaking time with that of the response 'energy consumption' are illustrated by plotting 3D surface graphs generated by the model. The 3D responses were shown in Fig. 4.3, (a), (b) and (c).







c

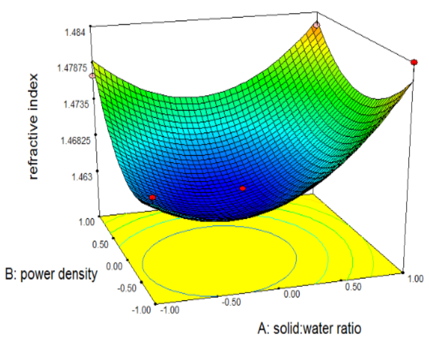
Figure 4.3. Effect of process parameters on energy consumption

Fig. 4.3 (a) and (c) shows that increase in power density increases total energy consumption up to a certain extent and then gets decreased. This might be due to increase in power density resulting in a decrease in total extraction time and thus to a decrease in total energy consumption (Desai and Parikh, 2012). When compared with hydro distillation the total energy consumed for microwave assisted extraction is 1.09 kWh whereas for the same oil yield HD process resulted in an energy consumption of 2.43 kWh.

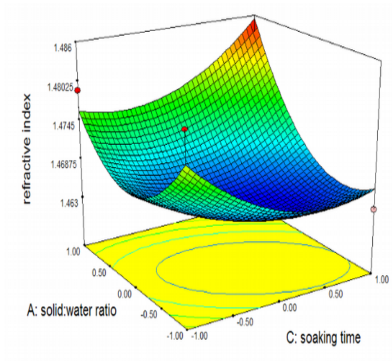
It can be concluded from Fig.4.3 (b) and (c) that soaking time had insignificant effect on total energy consumption. Solid: water ratio had a significant effect on the total energy consumption. Increase in solid: water ratio showed a decrease in total energy consumption, due to decrease in total extraction time.

4.6.4 Effect of Process Parameters on Refractive Index of Oil

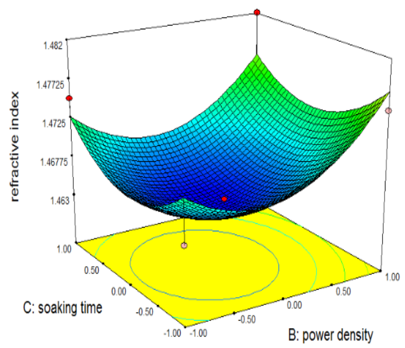
The relationship between Solid: water ratio, power density and soaking time on refractive index are illustrated by plotting 3D graphs representing the response surface generated by the model . The 3D responses were shown in Fig. 4.4, (a), (b) and (c).



a



b



c

Figure 4.4. Effect of process parameters on refractive index of oil

The solid: water ratio was found to have no effect on the refractive index of the essential oil. The power density and soaking time have an insignificant effect on the refractive index of the oil. As the power density 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 medium resulting in lower refractive index values, as refractive index is the ratio of speed of light in vacuum to the speed of light in medium (Anon., 2014). But when compared with the refractive index of hydro distilled oil, the refractive index of MAE essential oil also falls within the range of 1.474 to 1.4880. Therefore, the refractive index was found to be similar for essential oil obtained in both the processes (Guan *et al.*, 2011).

4.6.5 Effect of Process Parameters on Specific Gravity of Oil

The relationship between Solid: water ratio, power density and soaking time on specific gravity are illustrated by plotting 3D graphs representing the response surface generated by the model. The 3D responses were shown in Fig. 4.5, (a), (b) and (c).

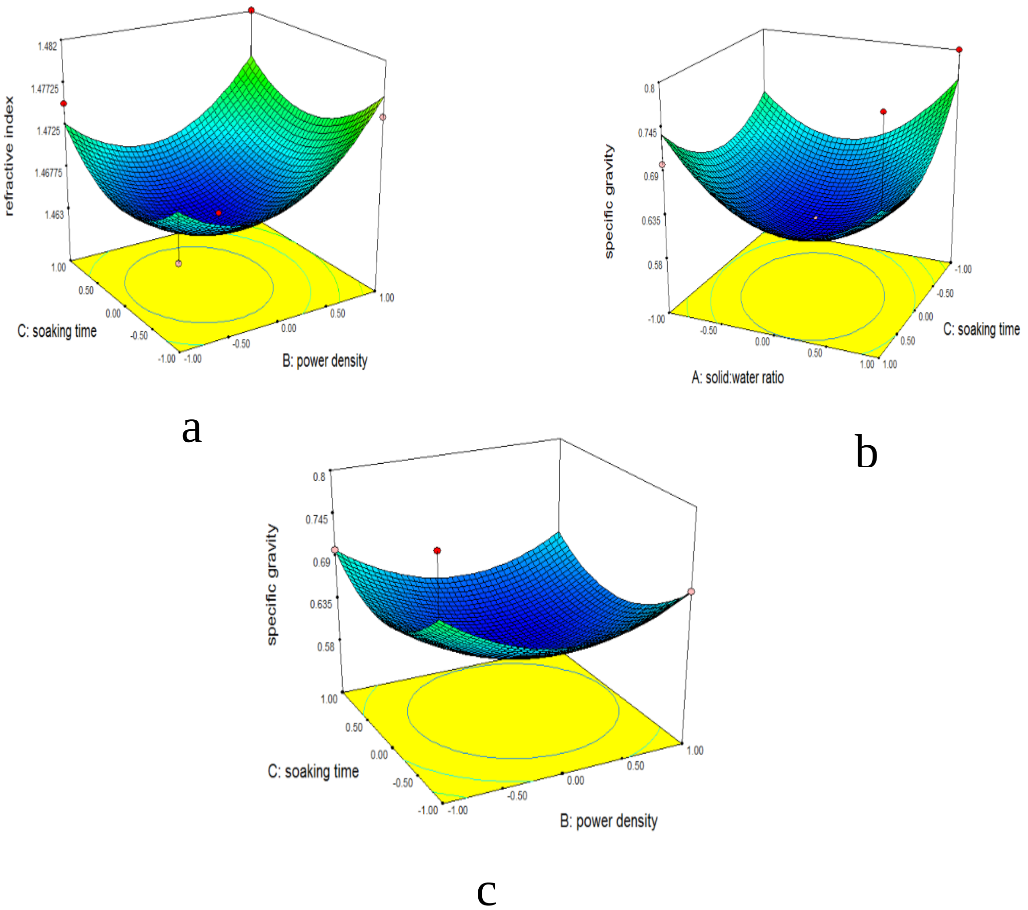


Figure 4.5. Effect of process parameters on specific gravity of oil

From Fig. 4.5 (a) and (c) it may be revealed that specific gravity increases with increase in power density and illustrates that solid: water ratio and power density had a significant effect on specific gravity of oil, whereas soaking time has an insignificant effect. Burdock *et al.*, (1995) had reported specific gravity values of hydro distilled nutmeg seed essential oil. No significant difference in specific gravity values were observed between the MAE nutmeg seed oil and conventional hydro distilled oil as found experimentally and the reported values.

4.6.6 Effect of Process Parameters on Solubility of Oil

The variation of Solid: water ratio, power density and soaking time with that of solubility is shown by plotting 3D graphs representing the response surface generated by the model. The 3D responses were shown in Fig. 4.6, (a), (b) and (c).

From Fig. 4.6, it may be perceived that solid: water ratio has no effect on solubility of essential oil, whereas power density has negative effect and soaking time has positive effect on the solubility of nutmeg seed essential oil. However, there is no much difference between the solubility values of oil obtained by HD and MAE processes. Similar results were also reported by Golmakani *et al.*, (2008) for thyme essential oil.

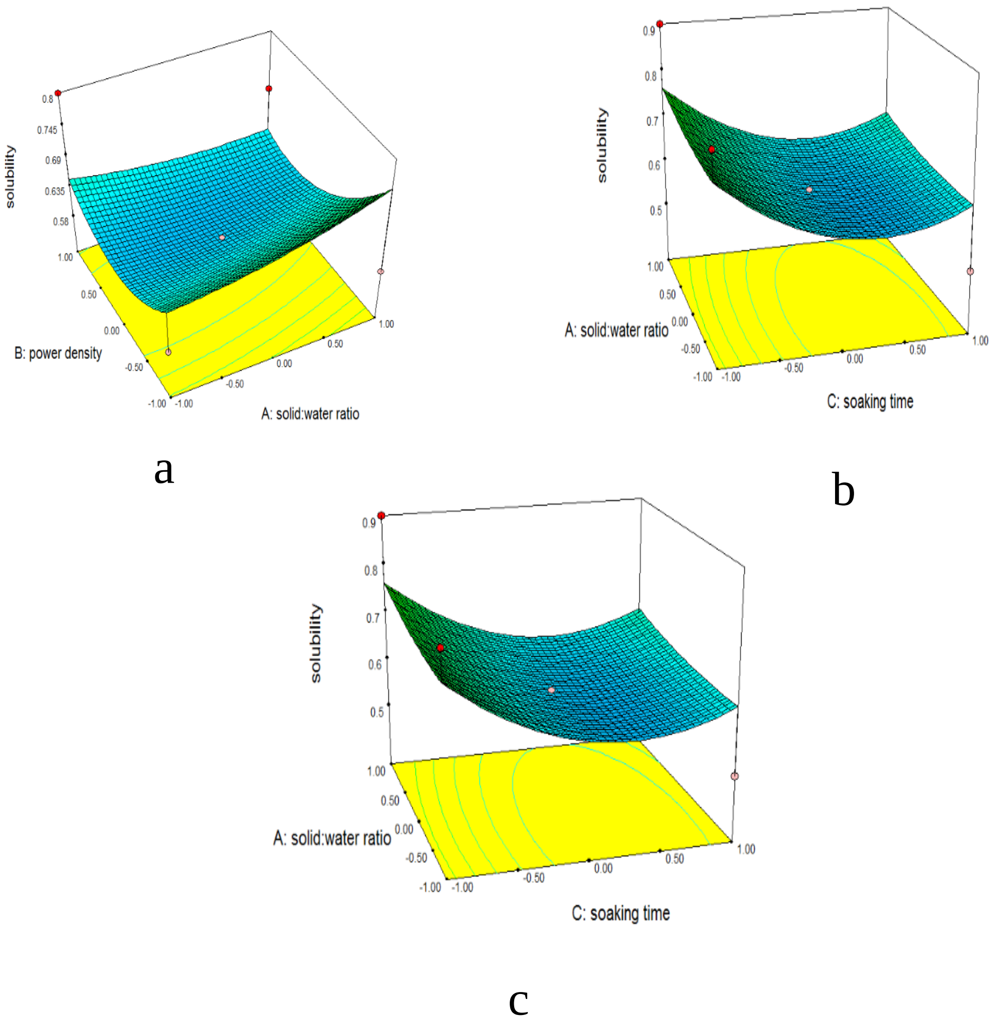


Figure 4.6. Effect of process parameters on solubility of oil

4.6.8 Desirability

Desirability analysis was performed by employing the design expert software. Desirability ranges from zero to one for any given response. The program combines individual desirability into a single number and then searches for the greatest overall desirability. A value of one represents the ideal case. A zero indicates that one or more responses fall outside desirable limits (Myers *et al.*, 2009).

From the desirability analysis, the optimal level of various parameters were found and listed in Table 4.4. From the analysis a Solid: water ratio of 2:1; Power density of 8.4 $W.g^{-1}$; and Soaking time of 4 h were found to be the optimum values. The extraction time, yield of nutmeg seed oil and energy consumption at this optimum process parameter levels for microwave assisted process were found to be 4 h, 3.5 ml/100 g sample, and 1.1 kWh, respectively whereas the same were found to be 7 h, 3.5 ml/ 100 g sample and 2.5 kWh, respectively for conventional hydro distillation process. These results clearly indicate that for same oil yield, the microwave assisted 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 42.85 and 56 per cent, respectively.

Table 4.3. Optimal level obtained from the desirability analysis.

Sl. No.	Response	Desirability	Optimal level	Low level	High level
1.	Extraction time	Minimize	3	3	4
2.	Oil yield	Maximize	4.1	3	4.1
3.	Energy consumption	Minimize	1.3	1.1	1.49
4.	Refractive index	Is in range	1.475	1.464	1.484
5.	Specific gravity	Is in range	0.76	0.6	0.9
6.	Solubility	Is in range	1.05	0.5	1.5

4.7 CHEMICAL ANALYSIS

The main active component present in nutmeg seed essential oil is myristicin which is usually taken as a standard for comparison. The presence of myristicin in MAE oil under optimized process values was determined by gas chromatography method. The presence of myristicin was analyzed by comparing with the chromatograph of myristicin standard (Fig. 4.10). The chromatograph of the optimally produced MAE nutmeg seed essential oil is shown in Fig. 4.11. The myristicin content was then compared with chromatograph of hydro distilled nutmeg seed oil. The chromatograph of HD nutmeg seed oil is shown in Fig. 4.12.

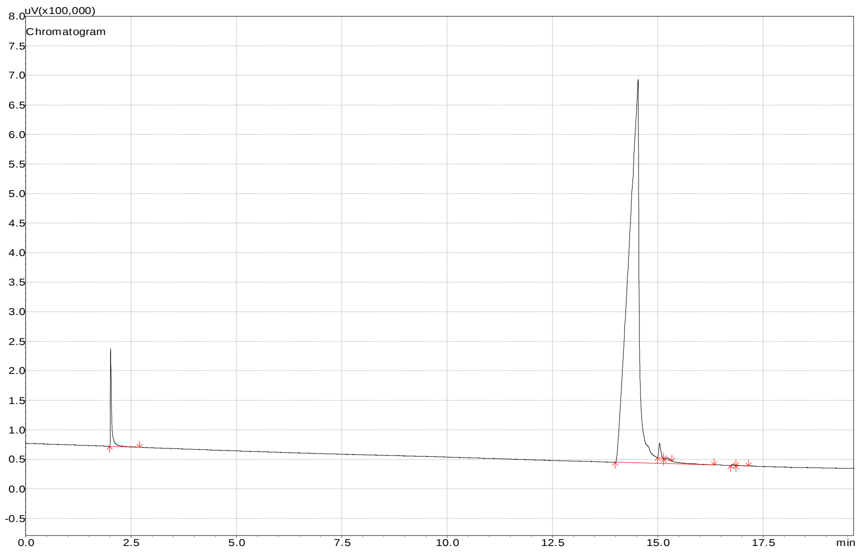


Figure 4.7. Gas chromatograph of myristicin standard

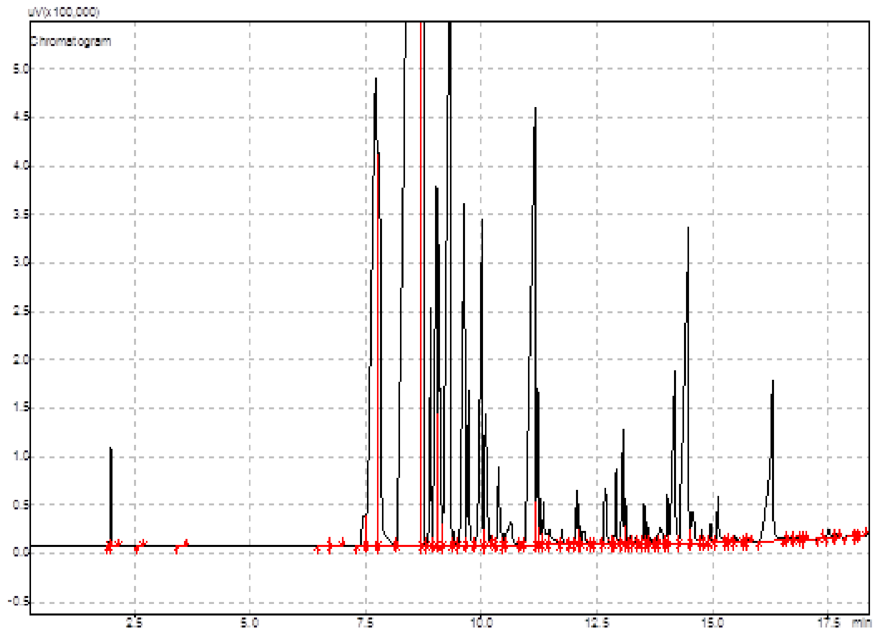


Figure 4.8. Gas chromatograph of MAE nutmeg seed oil

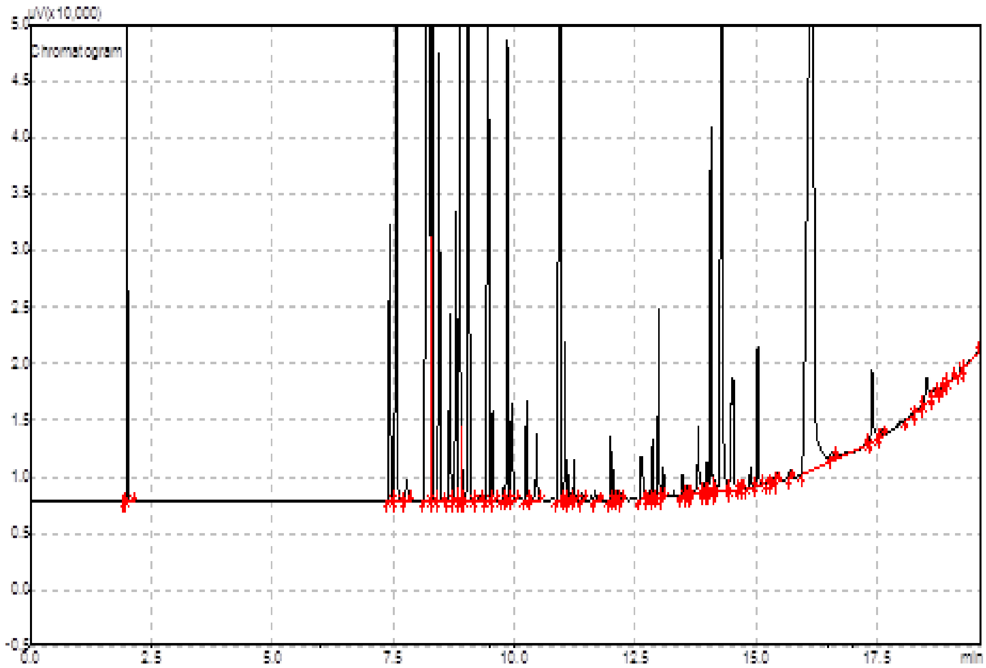


Figure 4.9. Gas chromatograph of HD nutmeg seed oil

From the Fig. 4.8 and 4.9, it may be concluded that the peak of myristicin in the chromatograph of MAE nutmeg oil at optimized conditions is slightly higher compared to the peak of myristicin in the chromatograph of HD nutmeg seed oil. This might be due to the degradation of myristicin at high temperatures of around

100ºC in HD process. Also, the myristicin content of MAE 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 myristicin.

SUMMARY

AND

CONCLUSION

CHAPTER 5

SUMMARY AND CONCLUSION

Spices are seed, fruit, root, bark, or other plant substance which are primarily used for flavoring, coloring or preserving food. Essential oil generally refers to concentrated volatile oils that are hydrophobic, lipophilic and carry distinct scent through various parts of a plant or herbs. Before now, essential oil has been used in wide range of applications such as flavoring in foodstuffs, cosmetics, cleaning products, pesticides and fragrances. And also essential oil has gotten extended application into aromatherapy. They are generally extracted by distillation.

The commonly used distillation methods are hydro distillation, steam distillation, solvent extraction, cold pressing etc. But these methods carry the disadvantages mainly concerned with the quality of final product such as loss of some volatile notes, low extraction efficiency and degradation of unsaturated ester compounds through thermal or hydrolytic effects. These processes also requires high extraction times and energy consumption.

A recent development of the essential oil extraction is the microwave assisted process. In microwave heating of food materials, the internal heating of the already present water within the plant material by the microwaves leads to the rupture of the glands and odoriferous receptacles freeing the essential oil which is then evaporated by the in-situ water of the plant material. The water then evaporated could then be passed through a condenser outside the microwave cavity where it is condensed. 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 increased yield with quality of the extracted oil superior to that of other conventional methods due to the mild conditions.

In this study the MAE system developed at KCAET ,Tavanur was used for extracting nutmeg seed essential oil. The developed extraction system consists of a microwave cavity, extraction unit, supporting stand and energy meter. A microwave oven with maximum microwave power delivery of 1200 W was chosen as microwave source. The oven consists of a control panel where cooking time, power, action indicators and clock time are displayed and controlled. The oil is extracted by micro mode since the temperatures are low at this mode. The Extraction unit consists of a Clevenger hydro distillation system in which recycling of distilled water also takes place. A hole of ½ inch diameter was drilled on the top of the microwave cavity, and a glass tube of same diameter with both ends open is inserted into the hole of microwave cavity which acts as carrier of vapors from round bottomed flask which is inside the cavity to the condenser outside the microwave cavity. A supporting stand is fabricated and is placed outside the oven for supporting the glass extraction unit. The stand comprises of a circular ring (outer diameter 48 mm, inner diameter 43mm and height 69 mm) and three stainless steel pipes (each of diameter 12.7 mm and length 80 mm) which acts as supporting legs for the ring. For keeping the distillation unit straight without tilting a silicon rubber cork was inserted into the circular ring through which the glass stem passes. A digital energy meter was connected to the microwave assisted extraction system to measure the energy consumed during the distillation process.

In order to evaluate the developed system towards extraction of nutmeg seed essential oil, the process parameters like solid: water ratio, power density and soaking time which would influence the essential oil yield, extraction time and energy consumption were chosen as independent variables. The physical quality characteristics like refractive index, specific gravity, solubility and colour of essential oil were selected as dependent variables. Based on the preliminary studies the levels of process parameters were fixed as solid: water ratios of 2:1, 4:3 and 1: 1, power densities of 7.2, 8.4 and 9.6 $W.g^{-1}$ and soaking times of 2, 3 and 4 h.

The experiments were performed by taking 100 g of soaked nutmeg seed for a stipulated period of time as mentioned above into the round bottomed flask of the extraction unit. The microwave power level along with time is set in the control panel for various treatment conditions. Microwaves heat the water and vapors of water as well as essential oil in nutmeg seed gets vaporized and passes out of the microwave cavity through the distillation stem into the condenser. These vapors then passed through the condenser where they gets condensed and falls back into the bottom of the extraction unit where the oil and water gets separated and oil which is lighter than water settles on the top and water which is denser settles on the bottom. The oil is collected by means of a stopper provided on the extraction unit. The water is drained off and the essential oil thus collected is dehydrated with anhydrous sodium sulphate and stored at 2°C in amber coloured glass bottles for further analysis.

Hydro distillation was performed as control for comparing the microwave assisted process. The physical quality characteristics of essential oil obtained by both the processes were measured and compared. For determining the myristicin content which is considered as a main chemical constituent in nutmeg seed oil, Gas chromatography was used.

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 optimized for the response variables using the above software.

The results showed that with increase in soaking time above 3 h, the total time of extraction was found to decrease. Also, soaking time beyond 3 h did not have much effect in the yield of oil. Increase in power density results in a decrease in total extraction time and thus to a decrease in total energy consumption. The process parameters have an insignificant effect on the physical quality characteristics of the oil. Microwave assisted process resulted in an oil yield of 4-11 per cent, with an extraction time of 3 h and energy consumption of 1.1 kWh. The time taken for extracting the seed essential oil in hydro distillation process was around 7 h for the same oil yield, with an energy consumption of 2.5 kWh. This indicates that for the same oil yield, the microwave assisted process resulted in a rapid extraction process with about 55per cent saving in energy when compared to hydro distillation process. The physical quality characteristics of oil in both the process was found to be similar whereas the chemical constituent i.e. myristicin content was slightly higher in microwave assisted process compared with hydro distillation method.

The optimized conditions of solid: water ratio, power density and soaking time for extracting essential oil in microwave assisted process was found to be 2:1, 8.4 $W.g^{-1}$and 4 h, respectively. Therefore, microwave assisted extraction of nutmeg seed oil could be considered as an extraction technique that results in the production of high quality oil in higher quantity in less time with minimum energy consumption.

REFERENCES

REFERENCES

Abdullah, M.H.R.O., Chng, P.E., and Lim, T.H. 2010. Determination of some physical properties of nutmeg (*Myristica fragrans*) Seeds. *Res. J. Appl. Sci. Eng. Technol.* 2(7): 669-672.

Agbogidi, O.M. and Azagbaekwe, O.P. 2013. Health and nutritional benefits of nutmeg (*Myristica fragrans* Houtt.). *Sci. Agric*. 1(2): 40-44.

[Anonymous]. 2010. *Major spice/ state wise area and production of spices.* State Agri/Horticulture departments, DASD Calicut.

[Anonymous]. 2014. [on line]. Available: https://www.quora.com/Does-the-refractive-index-relate-to-temperature-inversely-or-directly. [17 May. 2014].

AOAC (Association of Official Agricultural Chemists) 2000. *Determination of moisture (Dean and Stark toluene distillation method)* (17th Ed.). AOAC International, pp. 3-5.

Avelina, F., Nelly, R., Palou, E., and Aurelio, L. 2016. Estimation of mass transfer coefficients of the extraction process of essential oil from orange peel using microwave assisted extraction. *J. Food. Eng*. 170: 136-143.

Bakibaev, A.A., Gazaliev, A.M., Kabieva, S.K., Fedorchenko, V.I., Guba, G.Y., Smetanina, E.I., Dolgov, I.R., and Gulyaev, R.O. 2015. Polymerization of lactic acid using microwave and conventional heating. *Proc. Chem*. 15: 97-102.

Balick, M.J. and Paul, A.C. 2000. *Plants that heal people: culture of science of ethno botany*. Scientific American Library, New York.

Barceloux, D.G. 2009. Nutmeg *Myristica fragrans* Houtt. *Res. J. Spices* 55(6): 373-379.

Baron, R.D. and Villa, A.L. 2014. Microwave assisted extraction of essential oil and pectin from orange peel in different stages of maturity. *Rev. Fac. Agron.* 31: 145-158.

Bavappa, K. V. A. and Reuttimann, R. A. 1981. Nutmeg cultivation and processing. *Tech. Bull*. 2.

Box, G.E. and Behnken, D.W. 1960. Some new three level designs for the study of quantitative variables. *Technol*. 4: 455-475.

Burdock, G.A. 1995. *Fenaroli's Handbook of Flavor Ingredients* (3rd Ed.). CRC Press, Boca Raton, FL, 1613p.

Chandrasekaran, S., Ramanathan, S., and Tanmay, B. 2013. Microwave food processing – a review. *Food Res. Int*. 52: 243-261.

Chemat, F., Lucchesi, M.E., Smadja, J., Favretto, L., Colnaghi, G., and Visinoni, F. 2006. Microwave accelerated steam distillation of essential oil from lavender: A rapid, clean and environmentally friendly approach. *Anal. Chimica. Acta*. 555: 157-160.

Chen, Q., Zhuoyan, H., Yao, F.Y., and Liang, H. 2016. Study of two-stage microwave extraction of essential oil and pectin from pomelo peels. *LWT – Food. Sci. Technol*. 66: 538-545.

Cho, J.Y., Choi, G.J., Son, S.W., Jang, K.S., Lim, H.K., Lee, S.O., Sung, N.D., Cho, K.Y., and Kim, J.C. 2007. Isolation and antifungal activity of lignans from *Myristica fragrans* against various plant pathogenic fungi. *Pest. Manag. Sci*. 63(9): 935-940.

Datta, A.K. and Anantheswaran, R.C. 2000. *Handbook of microwave technology for food applications*. Marcel Dekker Inc, New York, 558p.

Desai, M.A. and Parikh, J. 2012. Microwave assisted extraction of essential oil from cymbopogon flexuosus (steud.) wats.: A parametric and comparative study. *Sep. Sci. Technol*. 47(13): 1963-1970.

Dorman, H.J., Figueiredo, A.C., Barroso, J.G., and Deans, S.G. 2000. In-vitro evaluation of antioxidant activity of essential oils and their components. *Flavor Fragrans* 15:12-16.

Duke, J.A. 1994. Biologically active compounds in important species. In: *Charalambous, Spices, herbs and edible fungi*. Elsevier Science Oxford, pp. 225-250.

Essam, F.A. and Maythm, H.A. 2012. Extraction and purification of terpenes from Nutmeg (*myristica fragrans*). *J. Al-Nahrain Univ*. 15(3): 151-160.

Ester, R.C., Silvia, N.Z., Walter, G.M., Alfredo, F.S., and Gustavo, A.V. 2013. *Study of the Chemical Composition of Essential Oils by Gas Chromatography*. UTN QUIMOBI Group, Argentina, pp. 307-324.

Fan, D., Wenrui, M., Wang, L., Huang, J., Zhang, F., Jianxin, Z., Zhang, H., and Chen, W. 2013. Determining the effects of microwave heating on the ordered structures of rice starch by NMR. *Carbohyd. Polym*. 92: 1395-1401.

Fangyuan, G., Tingting, Z., Yongsheng, H., Liai, L., Heyden, Y.V., Jacques, C., Guocai, L., and Guorong, F. 2016. Cyclodextrin-based ultrasonic-assisted microwave extraction and HPLC-PDA-ESI-ITMS separation and identification of hydrophilic and hydrophobic components of polygonum cuspidatum: A green, rapid and effective process. *Ind. Crops. Prod*. 80: 59-69.

Ferhat, A., Ginies, C., Romdhane, M., and Chemat, F. 2009. Eco-friendly and cleaner process for isolation of essential oil using microwave energy: experimental and theoretical study. *J. Chromat*. 1216: 5077-5085.

FCC (Food Chemical Codex) 2009. (4th Ed.). Washington DC: National Academic Press, pp. 413-414.

Ferhat, M.A., Meklati, B.Y., Smadja, J., and Chemat, F. 2006. An improved microwave clevenger apparatus for distillation of essential oils from orange peel. *J. Chromat*. 1112: 121-126.

Forrest, J.E. and Heacock, R.A. 1972. Nutmeg and mace, the psychotropic spices from myristica fragrans. Lloydia, 35(4): 440-449.

Gill, L.S. 1992. *Ethno medicinal use of plants in Nigeria*. University of Benin Press, Benin-City.

Golmakani, M.T. and Rezaei, K. 2008. Comparison of microwave-assisted hydrodistillation with the traditional hydrodistillation method in the extraction of essential oils from *Thymus vulgaris* L. *Food Chem*. 109: 925-930.

Guan, J.J., Zhang, T.B., Hui, M., Yin, H.C., Qiu, A.Y., and Liu, X.Y. 2011. Mechanism of microwave – accelerated soy protein isolate-saccharide graft reactions. *Food Res. Int*. 44: 2647-2654.

Gupta, A.D., Vipin Kumar, B., Vikash babu., and Nishi, M. 2013. Chemistry, antioxidant and antimicrobial potential of nutmeg (*Myristica fragrans* Houtt). *J. Genet. Eng. Biotechnol.* 11: 25–31.

Haber, A. and Runyon, R. 1977. *General Statistics* (3rd Ed.). Addison-Wesley, Reading, MA.

Hallstrom, H. and Thuvander, A. 1997. *Toxicological evaluation of Myristica fragrans* (2nd Ed.). CRC Press, Boca Raton, 5: 186-192.

Handa, S.S. 2008. An overview of extraction techniques for medicinal and aromatic plants. In: Khanuja, S.S., Longo, G., and Rakesh, D.D. (eds), *Extraction technologies for medicinal and aromatic plants.* International Centre for Science and High Technology, Trieste, pp. 21-52.

Hong, N., Varoujan, A.Y., Raghavan, G.S., Pare, J.R., and Jacqueline, M.R. 2001. Microwave-assisted extraction of phenolic compounds from grape seed. *Nat. Prod. Res*. 15(3): 197-204.

IISR [Indian Institute of Spices Research]. 2013. IISR home page [on line]. Available: http://www.spices.res.in/. [24 Jun. 2013].

Ikediala, J.N., Tang, J., Drake, S.R., and Neven, L.G. 2000. Dielectric properties of apple cultivars and codling moth larvae. *Trans. ASAE*. 43: 1175-1184.

Iwu, M.M. 1993. *Handbook of African medicinal plants*. R.C. Press Incooperated, Florida.

Jermann, C., Tatiana, K., Edyta, M., Craig, L., and Valquiria, R. 2015. Mapping trends in novel and emerging food processing technologies around the world. *Innov. Food Sci. Emrg. Technol.* 31: 14-27.

Jiao, S., Johnson, J.A., Tang, J., and Wang, S. 2012. Industrial-scale radio frequency treatments for insect control in lentils. *J. Stored Prod. Res.* 48: 143-148.

Joseph, M. and Rachael, D. 2003. The health benefits of cinnamon, nutmeg and other favourite holiday spices [on-line]. Available: http://articles.mercola.com/sites/ articles/archieve/2003/12/13/holiday-spices.aspx [13 Dec.2003].

Kasahara, H., Miyazawa, M., and Kameoka, H. 2005. Absolute configuration of 8-0-4 Neolignanas from *Myristica fragrans*. *Photochem*. 40: 515-517.

Kiruba, K., Valerie, O., Yvan, G., and Thangavel, K. 2013. Optimization of microwave-assisted extraction of phenolic antioxidants from grape seeds (*Vitis vinifera*). *Food Bioprocess. Technol*. 6: 441-455.

Kowalski, S. 2013. Changes of antioxidant activity and formation of 5-hydroxymethylfurfural in honey during thermal and microwave processing. *Food Chem*. 141: 1378-1382.

Kratchanova, M., Pavlova, E., and Panchev, I. 2004. The effect of microwave heating of fresh orange peels on the fruit tissue and quality of extracted pectin. *Carbohyd. Polym*. 56(2): 181-185.

Krishnamoorthy, B. 2000. Sex conversion in nutmeg. Spice India 13(5): 11-12.

Latha, P.G., Sindhu, P.G., Suja, S.R., Geetha, B.S., Pushpangadan, P., and Rajasekharan, S. 2005. Pharmacology and chemistry of *Myristica fragrans* Houtt. - a review. *J. Spices. Aromat. Crops*. 14 (2): 94-101.

Lee, B.K., Kim, J.H., Jung, J.W., Choi, J.W., Han, E.S., Lee, S.H., Ko, K.H. and Ryu, J.H. 2005. Myristicin-induced neurotoxicity in human neuroblastoma SK-N-SH cells. *Toxicol. Lett*. 157: 49-56.

Lee, W.C., Yusof, S., Hamid, N.S., and Baharin, B.S. 2006. Optimizing conditions for enzymatic clarification of banana juice using response surface methodology (RSM). *J. Food Eng*. 73(1): 55–63.

Lee, S.H., Choi, W., Kim, C.H., and Jun, S. 2015. Development of a dual cylindrical microwave and ohmic combination heater for minimization of thermal lags in the processing of particulate foods. *LWT - Food Sci. Technol*. 63: 1220-1228.

Leslie, NG. and Maria, P.C. 2011. Focused microwaves-assisted extraction of theobromine and caffeine from cacao. *Food. Chem*. 129(4): 1819-1824.

Letellier, M. and Budzinski, H. 1999. Microwave assisted extraction of organic compounds. *Anal*. 27: 259-271.

Liu, W. and Lanier, T.C. 2016. Rapid (microwave) heating rate effects on texture, fat/water holding, and microstructure of cooked comminuted meat batters. *Food Res. Int*. 81: 108-113.

Llave, Y., Mori, K., Kambayashi, D., Fukuoka, M., and Sakai, N. 2016. Dielectric properties and model food application of tylose water pastes during microwave thawing and heating. *J. Food Eng*. 10: 3-28.

Lucchesi, M.E., Chemat, F., and Smadja, J. 2004. Solvent free microwave extraction of essential oil from aromatic herbs: comparison with conventional hydro-distillation. *J. Chromat*. 1043: 323-327.

Lucchesi, M.E., Smadja, J., Steven, B., Louw, W., and Chemat, F. 2007. Solvent free microwave extraction of *Elletaria cardamomum* L.: A multivariate study of a new technique for the extraction of essential oil. *J. Food Eng*. 79: 1079-1086.

Luque-Garcia, J.L., Velasco, J., Dobarganes, M.C., and Luque de castro, M.D. 2002. Fast quality monitoring of oil from prefried and fried foods by focused microwave-assisted soxhlet extraction. *Food. Chem*. 76: 241-248.

Maikhubu, I. 2006. Traditional medicine. *Swaziland Afr. J. Tradit. Compliment. Altern.. Med*. 2: 23-36.

Maria, B., Nuria, M., and Dolores, R. 2014. Impact of temperature on lethality of kiwi fruit puree pasteurization by thermal and microwave processing. *Food Control*. 35: 22-25.

Maria, B., Anouk, K., Joana, C., Dolores, R., Nuria, M., and Torsten, B. 2015. Chlorophylls and carotenoids of kiwifruit puree are affected similarly or less by microwave than by conventional heat processing and storage. *Food Chem*. 187: 254-262.

Montgomery, D.C. 2001. *Design and Analysis of Experiments*. New York Wiley, pp.416-419.

Morita, T., Jinno, K., Kawagishi, H., Arimoto, Y., Suganuma, H., Inakuma, T., and Sugiyama, K. 2003. Hepatoprotective effect of myristicin from nutmeg (Myristica *fragrans*) on lipopolysaccharide/d-galactosamine-induced liver injury. *J. Agric. Food Chem*. 51: 1560-1565.

Myers, R.H. 2009. Response surface methodology. *Ann Arbor, Edwards Brothers Inc*.

Nelson, N.L. 2013. Mace essential oil [on-line]. Available: http://maceoil.com/tag/ what-is-the-difference-between-nutmeg-and-mace [19 Apr. 2016].

Nguyen, L.T., Choi, W., Lee, S.H., and Jun, S. 2013. Exploring the heating patterns of multiphase foods in a continuous flow, simultaneous microwave and ohmic combination heater. *J. Food. Eng*. 116: 65-71.

Nukasani,S., Prince ,M.V., and Sreeja, R. 2016.Influence of process parameters on quality charecteristics of microwave assisted extracted nutmeg mace essential oil *adv.life science*.5:5464-5470.

Nukasani,S. 2016. Development and optimization of microwave assisted process for extraction of nutmeg mace essential oil. M.Tech (Agrl Engg) thesis, Kerala Agricultural university,Thrissur.120 pages.

Okoegwale, E.E. and Omofezi, J.U. 2001. Some herbal preparations among the people of Isoko clan of Delta State, Nigeria. *J. Appl. Sci*. 4(4): 2359-2371.

Osemene, K.P., Ilori, M.O., and Elujoba, A.A. 2013. Examining the nature of herbal research and development in Nigeria. *Int. J. Biol. Pharma. Allied Sci*. 1(2): 133-142.

ABSTRCT

ABSTRACT

Essential oils which are the volatile components distilled from the aromatic plant materials, have gained importance in cosmetic, therapeutic, aromatic, fragrant and spiritual uses. But the conventional methods of distillation carry the disadvantages mainly concerned with the quality of final product such as loss of some volatile notes, low extraction efficiency and degradation of unsaturated ester compounds through thermal or hydrolytic effects. These processes also requires high extraction times and energy consumption. However, in order to reduce these difficulties microwave energy could be effectively used to mediate extraction of essential oil in place of steam or water heating in order to introduce its inherent advantages. As in the case of microwave heating of food materials, the internal heating of the in-situ water within the plant material by the microwaves leads to the rupture of the glands and oleferous receptacles freeing the essential oil which is then evaporated by the in-situ water of the plant material. The water then evaporated could then be passed through a condenser outside the microwave cavity where it is condensed. This study envisages evaluation of developed microwave assisted extraction system towards extraction of nutmeg seed essential oil. The developed extraction system consists of a microwave cavity, extraction unit, supporting stand and energy meter. In order to evaluate the developed system towards extraction of nutmeg seed essential oil, the process parameters like solid: water ratios of 2:1, 4:3 and 1:1, power densities of 7.2, 8.4 and 9.6 W/g and soaking times of 2, 3 and 4 h which would influence the essential oil yield, extraction time and energy consumption were chosen as independent variables. The physical quality characteristics like refractive index, specific gravity, solubility and colour of essential oil were selected as dependent variables. The optimized conditions of solid: water ratio, power density and soaking time for extracting nutmeg seed essential oil in microwave assisted process was found to be 2:1, 8.4 W/g and 4 h respectively. The extraction time, yield of nutmeg seed oil and energy consumption at this optimum process parameter levels were found to be 4h, 3.5 ml/100g and 1.1 KWh respectively whereas the same were found to be 7h, 3.5 ml/100g and 2.5 KWH respectively for conventional hydrodistillation process. It was also revealed that active component myristicin was found to be higher in MAE nutmeg seed oil extracted under optimized condition compared to hydrodistilled nutmeg seed oil. Therefore, microwave assisted extraction

could be considered as an extraction technique that results in the production of high quality oil in higher quantity in less time with minimum energy consumption.