DEVELOPMENT AND OPTIMIZAION OF MICROWAVE ASSISTED PROCESS FOR EXTRACTION OF ESSENTIAL OIL FROM ALLSPICE LEAVES

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CERTIFICATE

Certified that this thesis entitled **"DEVELOPMENT AND OPTIMIZAION OF MICROWAVE ASSISTED PROCESS FOR EXTRACTION OF ESSENTIAL OIL FROM ALLSPICE LEAVES"** is a record of research work done independently by **ABINSHA SM (2017-06-002) ,AKSHAYA THOMAS (2017-06-004) ,AMRUTHA MADHU (2017-06-007) ,ARATHI P (2017-06-009) ,ARSHA SUGATHAN (2017-06-010)** under my guidance and supervision and that it has not previously formed the basis for the award of any degree, diploma, fellowship or associateship to them.

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DECLARATION

We hereby declare that this thesis entitled **"Development and Optimization of microwave assisted process for extraction of oil from allspice leaves"** is a bonafide record of research work done by us during the course of academic programme in the Kerala Agricultural University and the thesis has not previously formed for the award of any degree, diploma, associateship, fellowship or other similar title, of any other University or Society.

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CHAPTER 1

INTRODUCTION

A spice may be defined as an aromatic vegetable substance used as a condiment and for seasoning food. It adds piquancy, aroma and sometimes color. Spices owe their fragrance and pungency to some volatile oils that may occasionally be associated with an alkaloid. The volatile oils that give the spices the taste and flavor render their quality ephemeral. Spices are obtained from various parts of plants such as flowers, fruits, seeds, leaves, bark and rhizomes.

Demand and price of essential oil and herbal products are increasing constantly in national and international market. Essential oil contribution in world of fragrance and flavor industry is about 17%. Estimated production of perfumery raw material is around 5000t/annum valued at Rs 400 crores in India (Skria et al., 2007).With an estimated production volume of over 3 million metric tonnes, the state of Madhya Pradesh was the largest producer of spices across India in 2020. Rajasthan and Gujarat are the major producers in that year. The country's total spice production was estimated to be around 9.4 million metric tonnes. Kerala is known as spice capital of India. Major spices grown are Pepper, clove, Cardamom, star anise, nutmeg, Jamaica pepper.

Allspice also known as Jamaica pepper, Myrtle pepper , Pimenta is the dried un-ripened berry of pimento dioica, a mid-canopy tree native to the greater Antilles, southern Mexico, central America and now cultivated in many warm parts of the world. The minerals, vitamins, and anti-oxidants found in allspices may have several health benefits. Many of the compounds are being studied as potential treatments for inflammation, nausea, and even cancer. Allspice is also an excellent source of manganese, calcium, iron, vitamin B5, quercetin, ericifolin. Allspice can be added to sweet or savory food or even brewed as tea.

The oil of the plant known as "Essential oil" consists of fragrances which are oily in nature and represent the active constituents of plants. Essential oils are generally extracted by distillation which includes hydro distillation, steam distillation, solvent extraction, expression and cold pressing. The main disadvantages when conventional methods of distillation are followed as low quality of final product, low extraction efficiency and loss of unsaturated ester compounds through thermal or hydrolytic effects (Ferhat et al., 2006). However in order to reduce the extraction time , improve the extraction yield , enhance the quality of extract and to reduce operational cost latest technology such as microwave assisted extraction, pressurized solvent extraction , super critical fluid extraction and ultrasound assisted extraction have been sought. 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 including high extraction efficiency, less extraction time, and increased yield with superior quality of oil compared to conventional methods. Besides, microwave extraction classified as a simple and green technology and energy efficient. Since microwave heating is a volumetric process in which heating occur through kinetic effects and biomaterials respond differently with the microwave energy. Therefore, it is an urgent need to optimize the microwave process parameters for extraction of essential oil from each plant material.

Keeping the above cited facts the present study was conducted with the following objectives.

1. To conduct Microwave assisted extraction of essential oil from allspice leaves.
2. To optimize process parameters for microwave assisted extraction of allspice essential oil.
3. To conduct comparative studies of normal steam distillation and microwave assisted extraction of allspice leave in terms of extraction yield and physical parameters of oil.

REVIEW OF LITERATURE

CHAPTER 2

REVIEW OF LITERATURE

This chapter deals with the review of research work reported on the scenario of allspice and benefits of allspice 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.

ALLSPICES

Pimenta is a genus of the flowering plants in the Myrtaceae family, mostly found in the Caribbean region of America, and has about 15 species. All spice is having Caribbean origin. It is a slow growing, beautiful little tree with aromatic leaves. Oil pressed from the fruits is used in perfumes and cosmetics. The liqueurs, Benedictine and Chartreuse, contain allspice flavoring. Allspice can also be found in essential oil form eugenol. In many countries P. dioica are used in edible food plants, which may be used as iron rich leafy condiment in our country (Kamble Vaishali S et al., 2012).

Allspice, Pimenta dioica (L.) Is commonly used for culinary and medicinal purposes, because of the high content of phenolic compounds and various biological activities. Allspice, well known for its fruits called pimento, has been used as an important spice in the food industry, as an ingredient in cosmetic products, as well as a therapeutic agent for diverse diseases. It is also known as allspice due to its intricate aroma which is a medley of aroma from spices such as clove, nutmeg and cinnamon. In India, the leaves of pimenta are used to flavor rice giving it a typical aroma (Aleksandra Milenković et al. 2020).

Nutritional and Health Benefits

Traditionally the dried mature but not ripe berries are consumed as spice and commonly used for flavoring food, but the essential oil of this fruit and the aqueous extracts of leaves of the pimento tree have been used as carminative, hypoglycemic, stimulant, antimicrobial, acaricide, and antifungal pharmaceutical preparations. Moreover, since mankind has chosen to consume natural products the allspice essential oil is used massively in food, pharmaceutical, and perfume industries. Due to its high content of eugenol, methyl eugenol, myrcene, and caryophyllene. The high levels of eugenol contained in clove essential oil give it strong biological activity and antimicrobial activity. This phenolic compound can denature proteins and reacts with cell membrane phospholipids changing their permeability. This essential oil has been studied for different applications. The antioxidant potential of allspice oil was evaluated by many researchers and all of them have found that this essential oil has good antioxidant activity and can be utilized as a natural antioxidant. In more specific applications the allspice essential oil was used as an anti-inflammatory. Also extraordinary applications of the leaves and berries extracts of allspice have been reported in the fields of medicine and materials science (Yasvet Y. Andrade-Avila et al. 2017)

Principle	Amount	Percentage DV
Energy	263 Kcal	N/D
Carbohydrates	72.12 g	55.48
Protein	6.09 g	12.18
Total Fat	32.38 g	162
Cholesterol	8.69 g	24.83
Dietary Fiber	21.6 g	56.84
Vitamins		
Folates	36 µg	9
Niacin	2.86 mg	17.88
Pyridoxine	0.160 mg	12
Riboflavin	0.063 mg	4.85
Thiamin	0.101 mg	8.42

Vitamin-A	27 µ	3.86
Vitamin C	39.2 mg	43.56
Electrolytes		
Sodium	77 mg	5.13
Potassium	1044 mg	22.21
Minerals		
Calcium	661 mg	66.10
Copper	0.553 mg	61.44
Iron	7.06 mg	88.25
Magnesium	135 mg	32.14
Manganese	2.943 mg	127.96
Phosphorus	113 mg	16.14
Zinc	1.01 mg	9.18

Table 2.1 Nutritional value of Allspice (Gill, 1992). Percent daily values (% DVs) are based on 2000 calories diet intake.

Health benefits

Allspice is considered to have got therapeutic qualities. Allspice is about 4% oil. A substance within the oil, known as Eugenol, has germ killing as well as pain reducing features. Research claims that Allspice might also compact particular bacteria , viruses, as well as fungi and also enhance digestion of food .Some of the benefits of using Allspice include strong bones , anti-inflammatory qualities ,provides energy , enhances digestion , cancer prevention ,improves immune system, slows ageing, reduced constipation, enhances circulation(Gill, 1992).

Allspice has long history in folk medicine. Allspice are used in both sweet and savory dishes, it is the main ingredient in sauces, stews and desserts, and the key flavoring in liquors and Benedictine.in addition to being sold in whole berry and ground forms , allspice is also available as essential oil. It relieves pain, and kills bacteria and fungus. Allspice essential oil can be diffused into the air which may ease head ache or sinus pains, was observed by Cathy Wong (2021).

Medicinal use

Leiszhang et al. (2012) reported the medicinal use of allspice, the kind of medicine developed over centuries by empirical and in often times and anecdotal evidences. There is a long history of using allspice berries for folk healing. Allspice with hot tea has preferred for cold, menstrual cramps and dyspepsia crushed allspice berries are applied on bruises, sore joints and for myalgia. The most common ingredients tested are polyphenols, lignins and terpenoides. Extracts of allspice leaves have hypotensive effect, menopause treatments, cancer.

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^{o}C up to 60 min or to the action of a microwave field (MW) with constant power of 1.26W.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 (97^{o}C – 30 s) pasteurization and storage (4, 10, 22^{o}C 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 22°C) promoting chlorophyll stability to a greater extent than conventional heating (k = 0.0015–0.034 100 $g.mg^{-1}.day^{-1}$ at 4 to 22°C). 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:

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

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 (100^{o}C). 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 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 90°C might be due to the temperature being not enough to burst open the oil glands. Oil yield was also lower at 110°C 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 8.0^{o}C, 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 ± 2.9°C, respectively, while the combination heating exhibited little significant temperature gaps (maximum difference <3.08°C) 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 flavor 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.

From the review of literature it is understood that microwave energy could be effectively used for extraction of essential oil in place of steam or water heating. The main advantage of using microwave energy is it significantly increases the speed of the processes and reduces the thermal gradients. The essential oil from many plant materials were successfully extracted using microwave energy, but the studies pertaining to allspice essential oil extraction using microwave energy has not been found reported. With this background, the present study envisages development of a system for microwave extraction of allspice oil and optimization of the process parameters leading to high extraction efficiency and quality of the extracted oil. Such a study could produce allspice oil of high quality and quantity and reduce operational cost.

MATERIALS AND METHODS

CHAPTER 3

MATERIALS AND METHODS

This chapter describes the developed system of a microwave assisted process for extraction of allspice 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 allspice 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

Table 3.1 Specifications of microwave sources

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.

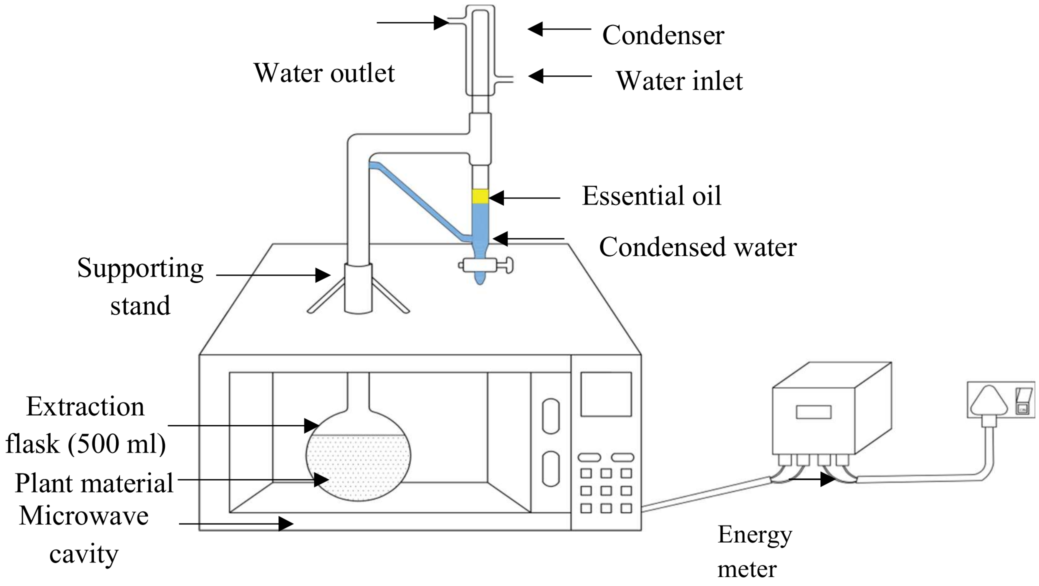

Figure 3.1. Schematic of the microwave assisted extraction unit

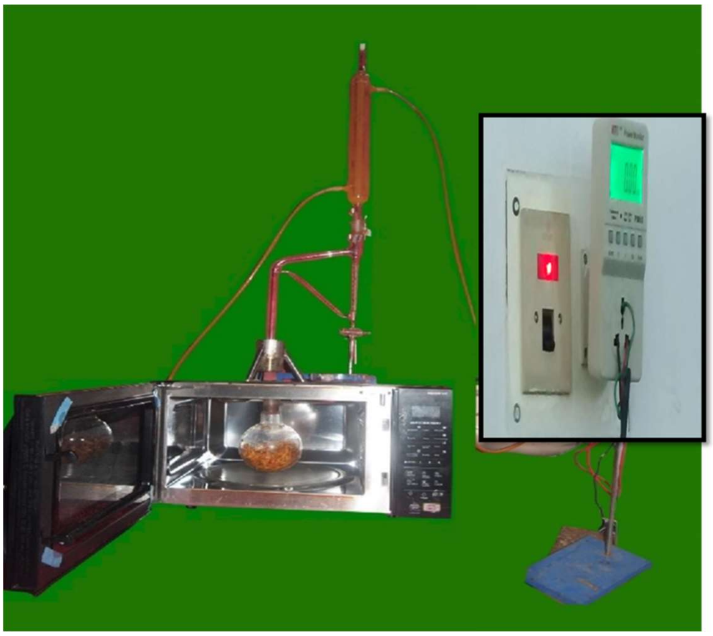

Plate 3.1. Developed microwave assisted extraction unit (Nukasani, 2016).

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).

Plate 3.2. Components of the extraction system

Plate 3.3. Supporting stand

3.2 PHYSICAL QUALITY CHARACTERISTICS OF OIL

3.2.1 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).

3.2.2 Solubility Test

The solubility of allspice essential oil was determined based on the procedure suggested by Food Chemical Codex (FCC, 1996). One ml sample of allspice leaves essential oil was transferred into a calibrated 10 ml glass stoppered cylinder graduated in 0.1 ml divisions. The oil was then diluted with 0.1 ml of 85 per cent (v/v) ethanol repeatedly each time. The temperature was maintained at 25°C, and the contents mixed thoroughly after each addition of alcohol. The dilution procedure was continued 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”.

3.2.3 Colour

The Hunter's lab colorimeter (Make: Colour Flex EZ) was used to determine the colour of allspice essential oil as shown in figure 3.2. The principle of working is by focusing the light and measuring energy reflected from the sample across the entire visible spectrum. The colour of allspice essential oil was measured by filling the sample in the transparent cup without any void space at the bottom. Thus, the colour value of the samples was obtained as L, a and b values.

3.3 EXPERIMENTAL DESIGN

Based on a thorough review of literature and the preliminary studies conducted, the process parameters which would influence the allspice leaves 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) Microwave power (P):

1) P_1 : 30W

2) P_2 : 40W

3) P_3 : 50W

b) Extraction Time (t):

1) t_1 : 1 h

2) t_2 : 2 h

3) t_3 : 3 h

3.3.2 Dependent Variables

Microwave assisted extraction system output parameters:

a) Essential oil yield

Physical quality characteristics of allspice essential oil:

a) Specific gravity
b) Solubility
c) colour

Table 3.2 Experimental design used for extraction of MAE of allspice leaves essential oil.

SI.No	Solid: Water	Power(W)	Extraction Time(hr)	Treatment
1	2:1	30	1	T1
2	2:1	30	2	T2
3	2:1	30	3	T3
4	2:1	40	1	T4
5	2:1	40	2	T5
6	2:1	40	3	T6
7	2:1	50	1	T7
8	2:1	50	2	T8
9	2:1	50	3	T9
10	2:1	-	5	HD

3.4 EXPERIMENTAL PROCEDURE

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

3.4.1 Determination of Moisture Content

Moisture content of fresh allspice leaves are determined by infrared moisture analyzer.

3.4.2 Extraction of Essential Oil

The desirable amount of allspice i.e. 200 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 leaves 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 2^oC 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, 100 g of sample and 300 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 100^oC. 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

3.5 DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS OF ALLSPICE ESSENTIAL OIL

3.5.1 Specific Gravity

The specific gravity of the essential oil was determined using the procedure explained in section 3.2.1.

3.5.2 Solubility

The solubility of the essential oil in 85 per cent (v/v) ethanol was determined using the procedure explained in section 3.2.2.

3.5.3 Colour

The colour of essential oil was determined using the procedure explained in section 3.2.3.

RESULT AND DISCUSSION

CHAPTER 4

RESULTS AND DISCUSSIONS

The outcomes of the various experiments conducted to optimize the process parameter for extraction of essential oil from allspice are narrated and discussed in this chapter. Also, the effect of various process variables on the physical 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 allspice leaves which was collected from local farmer was determined by using infrared moisture analyzer as explained in section 3.5.1. The average moisture content of fresh allspice leaves was found to be 44.36 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 allspice essential oil and for optimization of the process parameters, a series of experiments with solid: water ratios of 2:1, power 30, 40, 50 and extraction time 1hr, 2hr, 3hr as input variables were performed. The experiments were performed as per the experimental procedure laid out in section 3.4. The results of the experiments conducted towards the microwave assisted extraction process with mean values of extraction time, oil yield are tabulated in Table 4.1.

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

SI.No	Solid: Water	Power (W)	Extraction Time (hr.)	Treatment	Oil yield(ml)
1	2:1	30	1	T1	0.8
2	2:1	30	2	T2	0.9
3	2:1	30	3	T3	0.86
4	2:1	40	1	T4	1
5	2:1	40	2	T5	1.2
6	2:1	40	3	T6	1.5
7	2:1	50	1	T7	2

8	2:1	50	2	T8	3.6
9	2:1	50	3	T9	3
10	2:1	-	5	HD	0.5

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 3.5. Only ten experimental data were used in the design to optimize the parameters as per response surface methodology.

Experiments were also done with power of 60 W, but couldn't found oil yield.

4.3 OUTPUT CHARACTERISTICS OF MAE SYSTEM

4.3.1 Extraction Time

The time taken for extracting allspice essential oil during various combinations of process parameters are presented in Table 4.1. The extraction time varied between 1 to 3 hr. The minimum time for extracting maximum amount of allspice essential oil was obtained when the solid: water ratio is 2: 1, extraction time of 1hr and power 30 W.

4.3.2 Oil Yield

The yields of allspice essential oil obtained in various combinations of experiments are shown in Table 4.1. The maximum yield of oil obtained was 3.6 ml. The maximum oil yield was obtained for a solid: water ratio of 2:1, power 50 W and extraction time of 2 hr.

4.3.3 Power

Powers for extracting allspice essential oil obtained in various combinations of experiments are shown in Table 4.1. The power varied between 30 to 50 W. The least oil yield was obtained for solid: water ratio of 2:1, power 30 W and an extracting time of 1 hr.

4.4 DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS

The physical quality characteristics of the allspice essential oil extracted using microwave assisted extraction (MAE) method are listed in Table 4.2

Table 4.2. Physical quality characteristics of the extracted allspice essential oil

Sl. No.	Sample	Specific gravity	Solubility (v/v)	Color		
				L*	a*	b*
1.	T_1	0.7	1.0	1.29	-0.12	0.13
2.	T_2	0.7	1.2	3.63	-0.12	2.51
3.	T_3	0.71	1.0	0.75	-0.004	-0.14
4.	T_4	0.69	0.95	3.74	-0.089	2.69
5.	T_5	0.65	0.98	3.62	-0.002	3.2
6.	T_6	0.74	0.9	4.51	-0.28	3.82
7.	T_7	0.7	0.8	1.97	-0.08	-0.9
8.	T_8	0.75	0.75	3.61	-0.11	2.49
9.	T_9	0.8	0.7	3.47	-0.02	0.31
10.	HD	0.78	0.9	3.31	-0.04	-0.18

4.4.1 Specific Gravity

The specific gravity of optimized treatment (T8) of allspice essential oil obtained is 0.75.

4.4.2 Solubility

The solubility of optimized treatment (T8) allspice essential oil obtained in 85 per cent ethanol is 0.75 ml.

4.4.3 Colour

The colour of optimized treatment (T8) allspice essential oil is clear, greenish yellow liquid.

4.6 OPTIMIZATION OF PROCESS PARAMETERS

4.5.1 Effect of Process Parameters on Extraction Time of Oil

At the ratio of raw material to water 2:1 the yield of essential oil extracted from allspice leaves varies. It can be seen that the increased time from 1hr to 2hr, the yield increases. But no longer has significant increase after 2hours based on the results obtained 2hr been selected as the optimum time for extraction for a yield of 3.6 ml at a power of 50 W.

4.5.2 Effect of Process Parameters on Oil Yield

At the ratio of raw material to water 2:1 the yield of essential oil extracted from allspice leaves varies. It can be seen from T1,T2,..,T9 treatment maximum yield was obtained for T8(Sample to water ratio 2:1, Power 50W, Extraction time 2hr) and minimum yield was obtained for T1 (Sample to water ratio 2:1, Power 30W, Extraction time 1hr).

4.5.3 Effect of Process Parameters on Power

For the extraction of allspice essential oil the microwave power varied from 30 to 50 W. When the microwave power increased from 30 to 50 the yield increases significantly. However too high power can lead to volatile oil loss and degradation of components in essential oil. When power exceeds more than 50W there is no significant yield. Hence the optimum microwave power as per the experiments ids 50 W.

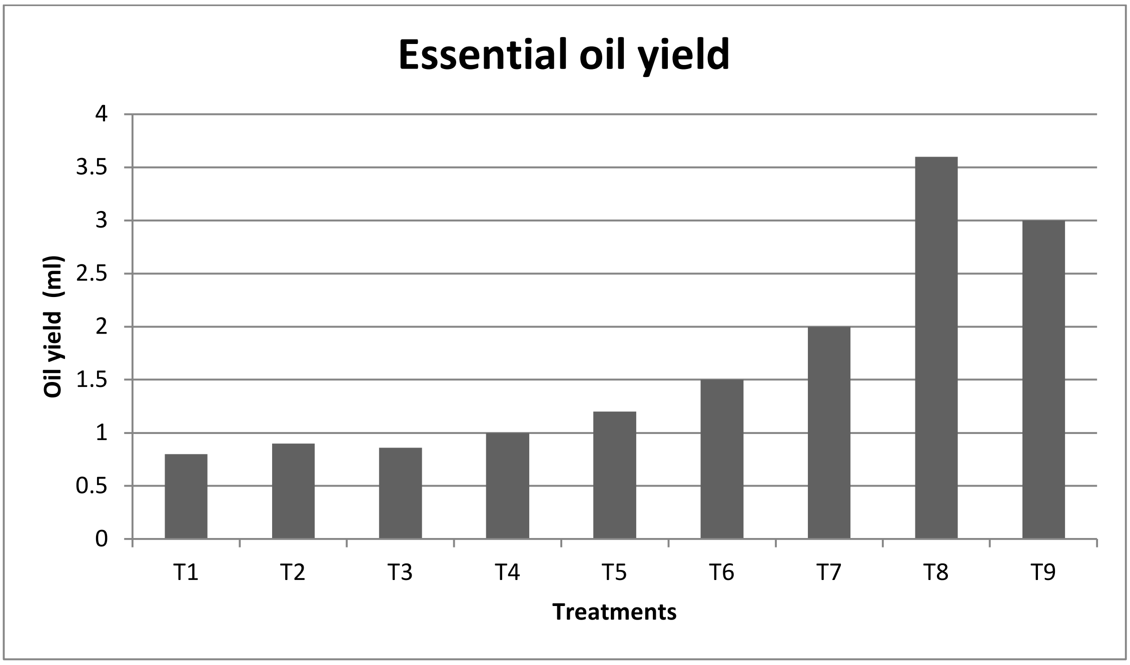

Figure 4.5 (a) Effect of Process Parameters on Oil Yield

4.5.4 Effect of Process Parameters on Specific Gravity of Oil

The relationship between power and extraction time on specific gravity are illustrated by the following graph.

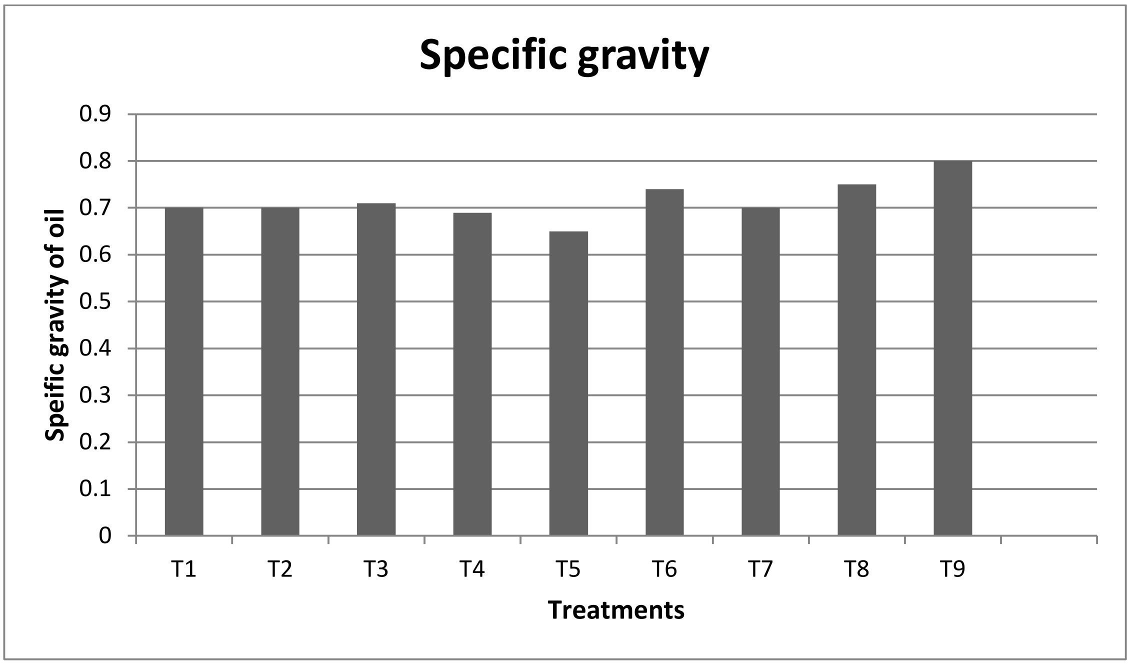

Figure 4.5 (a) Effect of Process Parameters on Specific Gravity of Oil

From Fig. 4.5 (a) it may be revealed that specific gravity increases with increase in microwave power. No significant difference in specific gravity values were observed between the MAE allspice essential oil and conventional hydro distilled oil as found experimentally and the reported values.

4.5.5 Effect of Process Parameters on Solubility of Oil

The variation of power and extraction time with that of solubility is shown by the following graph.

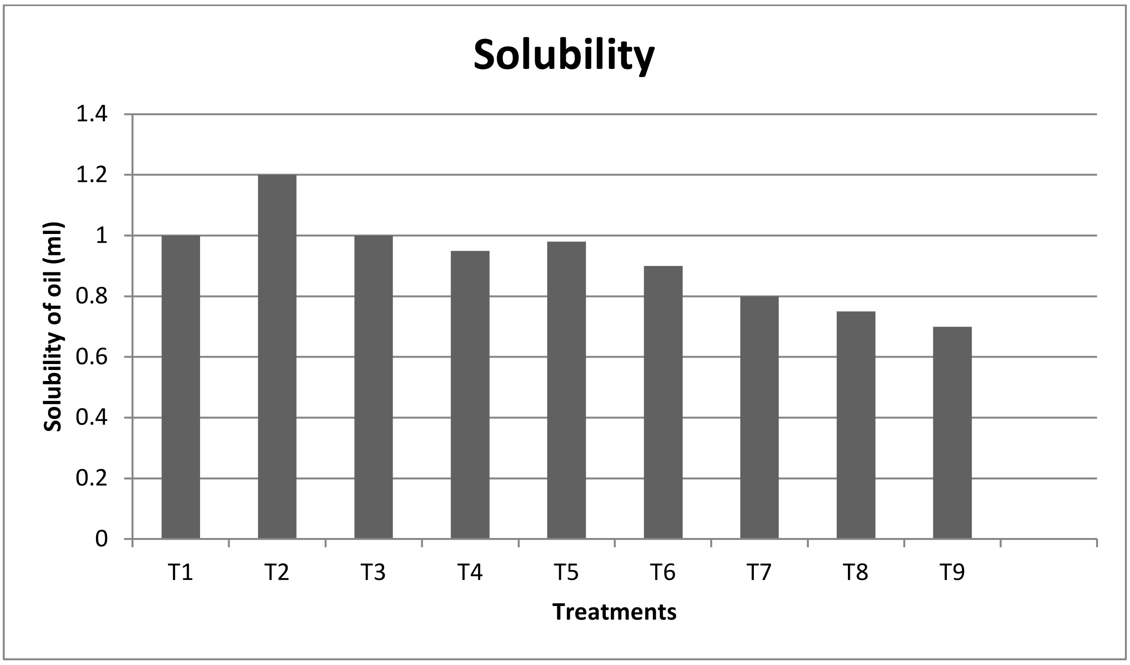

Figure 4.5 (b) Effect of process parameters on solubility of oil

From Fig. 4.5 (b) it may be perceived that power density has negative effect a on the solubility of allspice 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.

4.5.6 Effect of Process Parameters on Colour of Oil

The relationship between power and extraction time on colour of allspice essential oil are illustrated by the following graph.

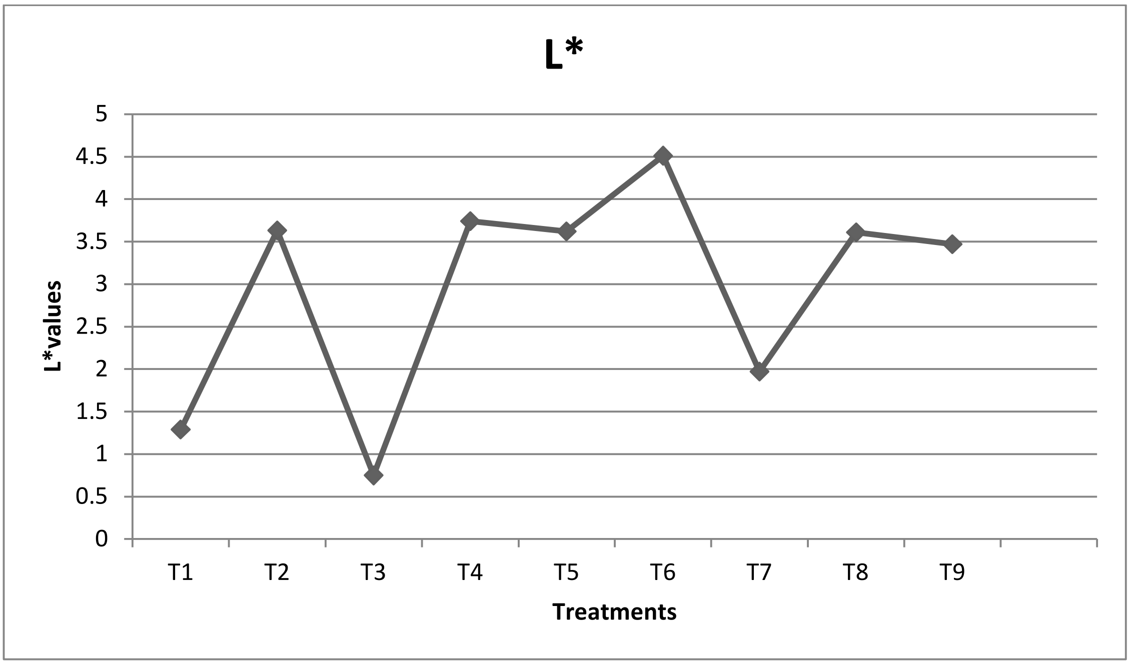

Figure 4.5 (c)

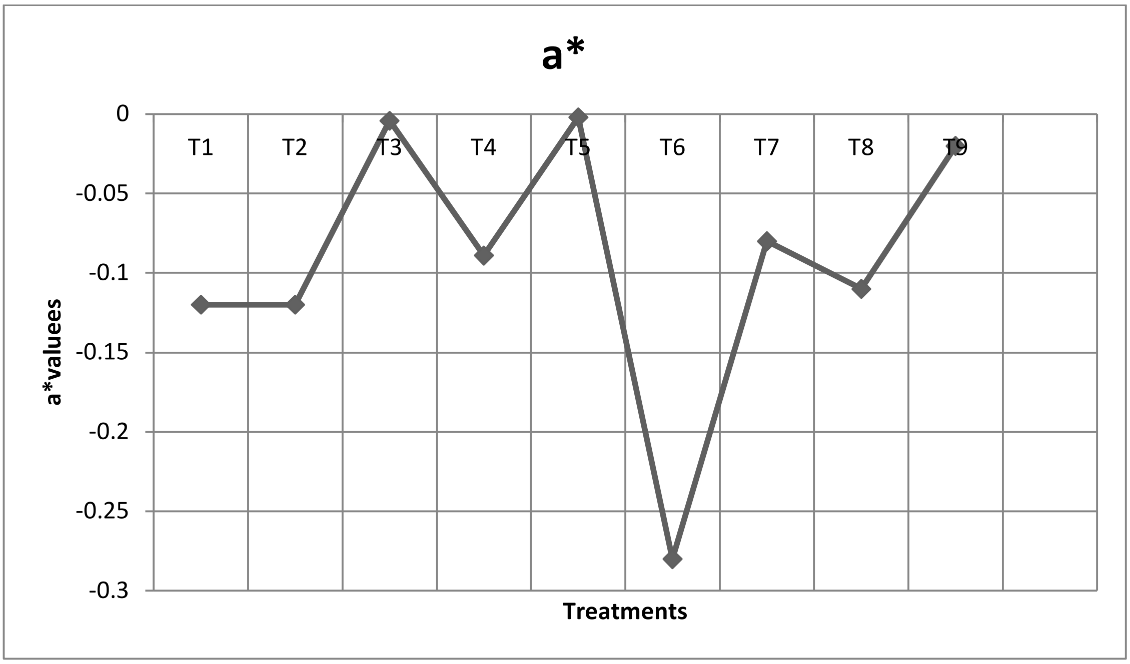

Figure 4.5 (d)

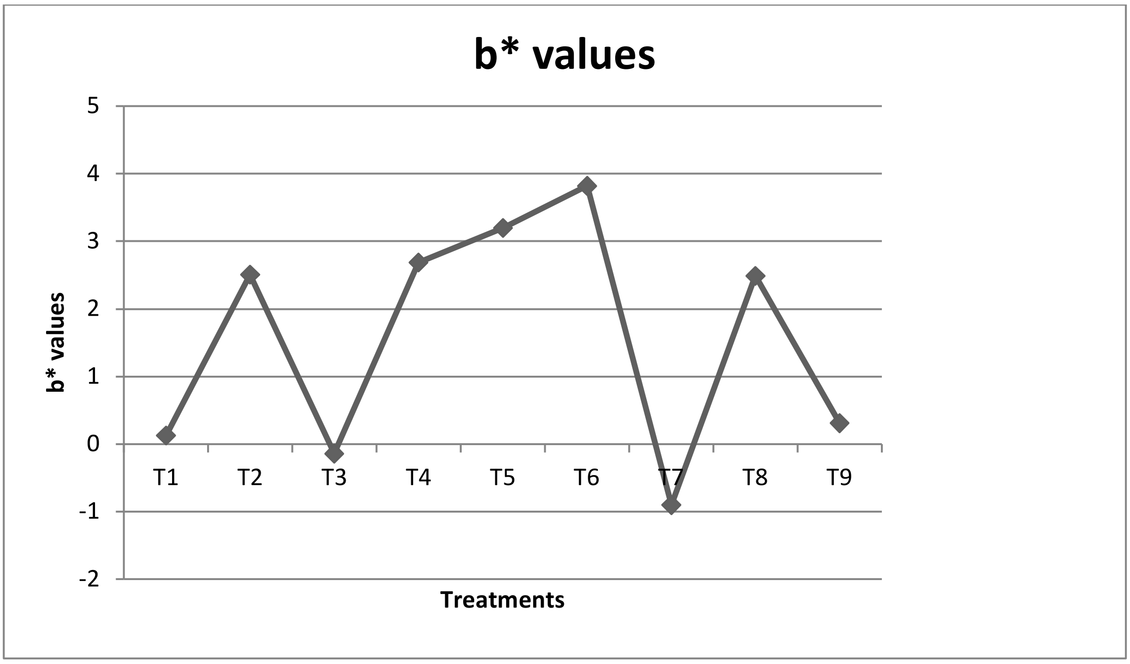

Figure 4.5 (e)

From Fig. 4.5 (c),(d) it may be perceived that the microwave power has no significant effect on the L and a values. However, increase in power leads to slight increase in b values fig.4.5 (e).

SUMMARY AND CONCLUSION

CHAPTER 5

SUMMARY AND CONCLUSION

Spices are aromatic or pungent dried vegetable substances used primarily to season food. Parts of plants from which they are derived include the bark, the flower, the roots, the seeds, and the fruits. They could be coloured, aromatic, phenolic, or pungent. Spices can be differentiated from herbs as dried part of plants other than leaves. Herbs could be fresh or dried leaves. They have strong flavours while herbs have subtle flavours. Spices have different origin and characteristic flavours. The best known use of spices is in food preparation and cooking. Essential oil generally refers to concentrated volatile oils that are hydrophobic, lipophilic and carry distinct scent through various parts of a plant or herbs.

Hydro distillation is the oldest and simplest oils extraction method. In essential plant oil extraction, steam distillation method is the broadest technique applied. The percentage of essential oils being extracted by this technique is 93% and the remaining 7% can be further extracted by other methods. Hydro diffusion extraction method is an extraction process in which steam is supplied to a container which holds plant materials. This technique is only applied on dried plant samples that can be damaged at boiling temperature. In solvent extraction technique ordinary solvents like acetone, petroleum ether, hexane, methanol, or ethanol have been implemented to extract fragile or delicate flower materials which cannot be extracted using heat or steam supplied. 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.

Microwave-assisted extraction (MAE) is a trending extraction technique considered to have high throughput and extraction efficiency when compared to other conventional methods. MAE relies on a microwave generator that delivers microwave energy to a polarizable material consisting of the solvent and the oil-bearing material. Microwave radiations interact with dipoles present in the sample matrix causing them to oscillate in response to the changing electromagnetic fields. The oscillation/rotation of the dipoles generates heat on the surface of the material and the heat is further transferred to the inside of the material by conduction. Besides the dipoles from the solvent used in the extraction process, microwave radiations interact with the water present within the cells of the oil-bearing material resulting into a quick and uniform penetration of the heat to the target tissues. This heat results in the formation of water vapor and electroporation effects, which disrupts the cell wall of the oilseed and enhances efficient extraction of intracellular metabolites.

In this study the MAE system developed at KCAET, Tavanur was used for extracting allspice 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 allspice essential oil, the process parameters like power and extraction time which would influence the essential oil yield, chosen as independent variables. The physical quality characteristics like specific gravity, solubility and colour of essential oil were studied. Based on the preliminary studies the levels of process parameters were fixed as solid: water ratios of 2:1, powers of 30, 40, 50 W and extraction times of 1, 2 and 3 hr.

The experiments were performed by taking 200 g of grounded allspice 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 allspice leaves 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^oC in amber coloured glass bottles for further analysis. Hydro distillation was performed as control for comparing the microwave assisted process. The essential oil yield obtained by both the processes were measured and compared.

The results showed that with increase in extraction time above 2 hr, the total essential oil yield was found to decrease. Increase in power results in an increase in total essential oil yield, however too high power can lead to volatile oil loss and degradation of components in essential oil. When power exceeds more than 50W there is no significant yield. Hence the optimum microwave power as per the experiments is 50 W.

Microwave assisted process resulted in a maximum oil yield of 3.6 ml, with an extraction time of 2 hr and power of 50 W. The time taken for extracting the allspice essential oil in hydro distillation process was around 5 hr for the oil yield of 0.5 ml. This indicates that the oil yield, in microwave assisted process resulted in a rapid extraction process when compared to hydro distillation process.

The optimized conditions of solid: water ratio, power and extraction time for extracting allspice essential oil in microwave assisted process was found to be 2:1, 50W and 2 hr, respectively. Therefore, microwave assisted extraction of allspice 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.

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ABSTRACT

DEVELOPMENT AND OPTIMIZAION OF MICROWAVE ASSISTED PROCESS FOR EXTRACTION OF ESSENTIAL OIL FROM ALLSPICE LEAVES

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ABSTRACT

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DEPARTMENT OF PROCESSING AND FOOD ENGINEERING

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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 allspice essential oil. The developed extraction system consists of a microwave cavity, extraction unit, supporting stand. In order to evaluate the developed system towards extraction of allspice essential oil, the process parameters like solid: water ratios of 2:1, power densities of 30, 40, and 50 W and extraction times of 1, 2 and3 h which would influence the essential oil yield were chosen as independent variables. The physical quality characteristics like specific gravity, solubility and colour of essential oil were selected as dependent variables. The optimized conditions of solid: water ratio, power and extraction time

for extracting allspice essential oil in microwave assisted process was found to be 2:1, 50W and 2h respectively.

The results showed that with increase in extraction time above 2 hr, the total essential oil yield was found to decrease. Increase in power results in an increase in total essential oil yield, however too high power can lead to volatile oil loss and degradation of components in essential oil. When power exceeds more than 50W there is no significant yield. Hence the optimum microwave power as per the experiments is 50 W.

Microwave assisted process resulted in a maximum oil yield of 3.6 ml, with an extraction time of 2 hr and power of 50 W. The time taken for extracting the allspice essential oil in hydro distillation process was around 5 hr for the oil yield of 0.5 ml. This indicates that the oil yield, in microwave assisted process resulted in a rapid extraction process when compared to hydro distillation process. Therefore, microwave assisted extraction of allspice 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.

Dedicated to all Farmers & food processing workers