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Review Article

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Review on Microwave Assisted Extraction Technique

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ABSTRACT

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. 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. This study deals with the review of research work reported on the scenario of microwaves and microwave assisted techniques. Reviews on application of microwave technology in extraction of essential oil has been elaborately presented.

Key words: *Distillation, Dielectric heating, Cytoprotective effect, Ionic Polarization, Dipolar rotation*

INTRODUCTION

Microwaves

Datta and Anantheswaran[7] 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). Based on the laws of optics and also on the type of material, microwaves can be reflected, absorbed or transmitted.

Guan *et al*[14]., described the other advantages of microwave food processing such as uniformity in heating the product, low operation cost due to a drastic reduction in processing time, ease of operation, low maintenance, flavor and nutritional changes in food were very less and surface browning and crusting of the product can be prevented due to heating from inside.

Chandrasekaran *et al*[4]., and Jermann *et al*[17]., reported that microwaves found wide applications in the area of food processing such as drying, pasteurization, cooking and preservation of food materials.

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Apart from conventional thermal processing techniques heat is generated volumetrically throughout the product at faster rates in microwave heating. Solid and pumpable foods can be processed by means of microwaves effectively. This includes fluids containing large particles.

Microwave Heating

During microwave and thermal processing of honey changes pertaining to antioxidant activity and formation of 5-hydroxymethylfurfural (HMF) was observed by Kowalski[19]. Four types of honey (honeydew, lime, acacia and buckwheat) were analyzed in that study. The samples were subjected to the action of a microwave field (MW) with constant power of 1.26 $W.g^{-1}$ up to 6 min and also to conventional heating in a water bath (WB) at 90°C up to 60 min. Percentage of free radical scavenging ability was taken as a measure to determine the changes in the antioxidant capacity of honeys. Determination of changes in the total polyphenols content (TPC) (equivalents of gallic acid mg/100 g of honey) were also done. The main observation is that compared with conventional process, honey treated with microwave field has faster formation of HMF. Though the effect of a microwave field accelerated greatly the formation of HMF, but it is suitable for honey processing because of the drastic reduction in operation time.

Maria *et al*[29]., 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*[28]., 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°C – 30 s) pasteurization and storage (4, 10, 22°C for up to 63 days) on individual as well as total carotenoids and chlorophylls in kiwifruit puree was evaluated. Studies pertaining to bio accessibility of carotenoids, before and after pasteurization, during storage was also studied. Remarkable changes in carotenoid (62–91 per cent losses) as well as chlorophyll (42–100 per cent losses) contents were observed during conventional and microwave heating. The decrease of total carotenoids and chlorophylls over time, were explained properly by first and second order kinetics respectively. During processing and storage bio accessibility of carotenoids remained ($p < 0.05$) unaltered. These results showed that the pigment composition of microwaved kiwifruit was most likely to that of the fresh fruit and no changes were observed during storage.

Saritha *et al*[33]., conducted a study on influence of microwave energy on pectic principles of mango peel. The main conclusion was that when used microwave energy for heating purpose, sufficient heat energy was generated within shorter periods of time. Also, where there is a high increase in temperature is desired there microwave energy can be substituted. Compared with conventional mode of extraction, maximum pectin yield was obtained in a short period of time when microwave energy was used. Better gelling characteristics, high viscosity and methoxyl content of pectin were observed in the samples treated with 660 and 1000 W for 20 min. Maximum yield of pectin was found in the samples exposed to microwave energy of 1000 W for 20 min. If samples exposed to microwave energy for duration of 25 min, then there is a decrease in methoxyl content, viscosity and galacturonic acid were noticed.

In order to achieve or possibly enhance the shelf life of tomato juice, relating to quality and nutritional aspects, microwave heating is considered as one of the appreciable alternative among the novel thermal technologies. Stratakos *et al*[34]., compared microwave heating and conventional pasteurization by processing tomato juice. The results were displayed in terms of antioxidant activity, microbial load and physicochemical characteristics. No significant changes were observed in physicochemical and colour characteristics of juices were observed during storage. Microorganisms were inactivated and found to be at low levels throughout the storage in both conventional and microwave pasteurization. Increase in cytoprotective effect against H_2O_2 were observed in the juice processed with the microwave energy. Similarity in the two tomato juices was proved by the organoleptic analysis. Hence, the continuous microwave volumetric heating system seems to be a feasible alternative to conventional pasteurization.

Principle of Microwave Heating

Datta *et al*[7]., stated that, the phenomenon of materials to absorb microwave energy and transform into heat is the main reason for microwave heating of materials. The two important mechanisms by which microwave heating of food materials mainly occurs are ionic polarization and dipolar rotation. Water has dipolar nature and dielectric heating is due to the presence of water or moisture in a given material. 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. As the microwave frequency is very high about 2450 MHz, the water molecules vibrate 2450 million times per second which causes internal friction in between the molecules. This friction between the molecules leads to volumetric heating of the material.

Fan *et al*[9]., concluded that there is no significant difference in the ordered structure of starch granules when heated using microwaves and rapid heating in an oil bath. Slight variations in the proportions of amorphous starch, double helices and V-type single helices, were determined by conventional heating while heating using microwave energy did not have a significant impact on the ordered structures of starch granules.

Bakibaev *et al*[2]., concluded that the process of getting polylactic acid (PLA) by microwave energy is hundreds of times faster compared with conventional heating. This observation was made when performed an experiment on polymerization of lactic acid using microwave and conventional heating. There is no change in the optical properties of PLA samples obtained in both the processes.

Liu and Lanier[24] stated that microwave heating results in an increase in palatability of the product especially comminuted meat batters, and the cooking properties were acceptable especially water/fat holding and texture (fracture and small strain mechanical properties). The experiments were performed with meat batters containing high fat content.

Microwave Assisted Oil Extraction

Handa[15] 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. Steam distillation is not practiced for extracting the essential oils which are highly soluble in water and to those which are likely to decompose when subjected to heat. In order to adopt steam distillation the oil should be steam volatile. All most all the essential oils in commerce are steam volatile, do not decompose when subjected to high heat and practically not soluble in water. Such essential oils are suitable to be processed 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 hydro distillation is that there must be always enough water present in the tank 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 along with the steam which is formed. Hydro distilled oil is slightly darker in color and has much stronger still notes than oils produced by other methods[35].

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[6,25].

Hong *et al*[16]., observed the changes in the extraction of phenolic compounds from grape seeds using microwave energy as heating source. In this optimization study microwave power varied from 150 to 300 W and extraction time varied from 20 to 200 s. By using Folin-Ciocalteau reagent the polyphenol content of the final extracts was measured in terms of mg of tannic acid equivalent per gram of crude extract (mg TAE/g of crude extract). The main observation is that, the yield of extract increased to 15.2 per cent and the polyphenol content increased to 429 mg TAE/g of crude extract when the solvent polarity was changed by the addition of 10 per cent water.

Luque-Garcia *et al*[27]., proposed a new method for extraction of fat from prefried and fried meat and fish. A drastic reduction in the process time (55 min versus 8 h) was achieved with same reproducibility that may be obtained by the conventional method. Besides, the proposed method has the advantage of recycling around 75-80 per cent of the extractant and it is cleaner when compared with conventional Soxhlet.

Chemat *et al*[5]., studied microwave accelerated steam distillation of essential oil (MASD) from lavender flowers. It was revealed that in steam distillation (SD) and MASD the extraction temperatures were same as the boiling point of water i.e. 100°C at atmospheric pressure. When SD and MASD were compared, the time taken to attain the extraction temperatures and also to get the first essential oil droplet, MASD requires only 5 min when compared with SD which requires almost 30 min. As a result the yield of oil obtained at an extraction time of 10 min in MASD was same as that obtained after 90 min by means of SD, which is one of the advanced methods in the stream of essential oil extraction. The final 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*[5]., 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*[26]., studied solvent-free microwave extraction (SFME) of cardamom essential oil. The results showed that compared to conventional hydro-distillation, rupture of glands and plant were more rapid when heated with microwave energy. In case of microwave heating, when the glands were subjected to more severe thermal stresses and localized high pressures, pressure build-up takes place within the glands which results in increased capacity for expansion, and leading to cell rupture more rapidly than in conventional extraction. Statistical treatment of the results revealed that the selected parameters i.e. extraction time, irradiation power and moisture content of the seeds have significant effect on the output parameters.

Golmakani *et al*[12]., 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 has taken only 75 min for extracting essential oil when compared to 4 h in HD. Also, MAHD was superior in terms of saving energy. Scanning electron microscopy (SEM) of thyme leaves undergone HD and MAHD provided evidences that sudden rupture of essential oil glands takes place in 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 energy did not adversely affect the composition of the essential oils.

Leslie and Maria[22] 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 when compared with conventional 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[8] 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*[18]., 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 effect 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) on dependent variables i.e. total phenols, antioxidant activity (1,1-diphenyl-2-picrylhydrazyl (DPPH) and ferric ion reducing antioxidant power (FRAP)) were determined. Central Composite Design was used to optimize the Microwave-assisted extraction (MAE). Extraction time and solvent concentration were the two independent variables significantly influenced the total phenols that were expressed as gallic acid equivalents (GAE), catechin equivalents (CAT), and tannic acid equivalents (TAE). The optimized conditions were maximized for 6 min of MAE of grape seed with 32.6 per cent ethanol at 121 W with a desirability function of 0.947. Per gram of grape seed the predicted extraction yields were 13±0.89, 21.6±1.59 and 15.9±1.32 mg GAE, CAT, and TAE, respectively.

Baron and Villa[3] studied microwave assisted extraction of essential oil and pectin from orange peel in different stages of maturity. The results mainly highlighted the yield of essential oil and the limonene content of the samples obtained using microwave energy. It revealed that the essential oil 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 noticed that at a low power of 200 W, no essential oil was extracted and at a high power level more than 600 W results in an oil colour of dark yellow or even black due to the presence of suspended material. During microwave irradiation intercellular expansion of plant tissues occurred and it was found using Scanning electron microscopy (SEM) analyses after essential oil extraction. Without the addition of solvent, leads to orange peel carbonization 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[3,20].

Gopika and Ghuman[13] developed a microwave assisted extraction unit for extraction of essential oil from celery seeds. The extraction unit was developed by modifying a domestic microwave oven and attaching a Clevenger apparatus to extract the essential oil. Effect of various independent variables such as soaking time, temperature and power density on celery seed during MAE was noticed. Box-Behnken design which is a multivariate study was used to evaluate the influence of the process parameters on the performance of MAE on celery seed. A comparative study was made with the oil yield, extraction time and energy consumption ($MJ.kg^{-1}$ oil) obtained by MAE with those of traditional hydro-distillation (HD). Results revealed 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). It implies 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*[1]., studied the effect of different process parameters during microwave assisted extraction (MAE) on the yield of essential oil obtained from orange peel. There was a significant effect ($p < 0.05$) of the particle size, moisture content and its interaction on the essential oil yield obtained and had an influence on the extraction phenomenon. 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 reduces the processing times. Decrease in particle size, increases the superficial area which provides a better contact of the sample with the solvent and penetration of microwaves and improves the extraction process.

High moisture content enhances the extraction recovery in most cases, due to rapid heating and temperature increase. This is because of microwaves interacting selectively with the free water molecules present in the gland and vascular system, leading to rupture of walls and release of the essential oil into the solvent[1,23]. But Ferhat *et al*[11]., stated that during lavender flowers essential oil extraction by microwave, there was no much difference in the yield of essential oil obtained by steam diffusion but the process time has got reduced drastically.

Sagarika *et al*[31]., observed the influence of process parameters on quality characteristics of microwave assisted extracted nutmeg mace essential oil and revealed that the physical quality characteristics of oil were found to be similar in microwave assisted process and conventional hydro distillation process whereas the main chemical constituent myristicin was found to be slightly higher in microwave assisted process than the hydro distillation method.

Combination Technologies

Though there are many advantages with the microwave technology like reduction of process times, energy consumption etc. but the major problem was localized heat zones related with the variation in physical, dielectric and thermal properties of food components. In order to decrease the localized heat zones in foods, microwave heating assisted with conventional heating methods such as vacuum and microwave absorbents was advantageous.

You *et al*[36]., 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 other hand, the low recoveries at short irradiation time might be due to 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*[30]., 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*[21]., developed a dual cylindrical microwave and ohmic combination heater for minimization of thermal lags in the processing of particulate foods. Results showed that two parameters in ohmic heating mainly salt concentration and particle size affected temperature variations between solution and particulates. Irrespective of particle size and mass fraction, the solution temperature was less than the particle temperature in microwave heating up to 12.5 g/l salt concentrations. However, if salt concentration in food mixtures increased to 20 g/l an opposite tendency was observed.

Samani *et al*[32]., analyzed the combinative effect of ultrasound and microwave power on *Saccharomyces cerevisiae* in orange juice processing. Due to overheating of juice at the heat-exchange surface in conventional heat pasteurization of orange juice off flavour in juice was detected. Taste of the juice is not changed when heated using combination technology. Also, complete inactivation of bacteria and pectin methyl esterase was obtained in the combined process. There was no significant effect on juice flavour. Also the appearance of orange juice in the combinative method was better than those of conventional method (57 per cent vs. 43 per cent).

Chen *et al*[6]., 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. Usage of extreme extraction condition, especially high temperature leads to instability of essential oil i.e. bringing negative effects such as thermal degradation of essential oil. Also when microwave power is low prolonging extraction time would be helpful to complete extraction of target compounds. 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 suggested as an 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*[10]., 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.

Summary and Conclusion

The essential oils have gained their importance in therapeutic, cosmetic, aromatic, fragrant and spiritual uses. They are generally extracted by distillation. The commonly used distillation methods carry the disadvantages mainly deals 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 modification of the essential oil extraction is the microwave assisted process. 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.

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