



Review Paper

Novel oil extraction methods in food industry: A review

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Abstract

Many fruits and cereals contain essential oils which are attaining importance in food, drug, and cosmetic industries owing to their desirability, and nutraceutical properties. Soxhlet extraction is the method used for isolation of oil from plant material. Usually, plant materials are subjected to mechanical shear using expellers to release the volatiles in virgin state. A new method of extraction has been solvent extraction involving use of polar solvents. The disadvantage with the process is the use of hazardous, and flammable liquid organic solvents, potentially toxic emission during extraction, non-selective extraction, and time consuming. In order to obtain a good yield from extraction as well as to prevent the uses of volatiles, correct alternative of solvent is very important. Novel extraction methods like ultra sound extraction, microwave assisted extraction, and supercritical fluid extraction has been successfully employed for extraction of oils with higher yield, and better physiochemical, and functional properties. This article reviews the recent work on extraction using novel methods like ultra sound extraction, microwave assisted extraction, and supercritical fluid extraction.

Keywords: *Extraction, microwave assisted extraction, novel methods, supercritical fluid extraction*

Introduction

Oil is extracted from a number of fruits, nuts and seeds for use in cooking and soap making (Potts and Michell, 1993) or as an ingredient in other foods such as baked or fried goods. Oil is a valuable product with universal demand, and the possible income from oil extraction is therefore often enough to justify the relatively high cost of setting up and running a small scale oil milling business. World production of oils and fats currently about 117 million tonnes per annum comes from vegetable and animal sources. Oil world production recognise 17 commodity oils, of which four are of animal origin, the remainder are from vegetable source. Of the total production of oils and fats, about 80% is used for food purposes, 6% is used in animal feed and the remaining 14% provides the basis of the oleochemical industry (Gunstone and Hamilton, 2001).

Traditionally plant materials were subjected to mechanical shear to release the volatiles in virgin state. Oil extraction was done mechanically with an oil press, expeller, or even with a wooden mortar and pestle—a traditional method that originated in India. Several types of small-scale extractors are commercially available, both imported from other countries and manufactured in the U.S. Oil extraction presses have a number of different designs, which can be grouped into screw or hydraulic operation. Both types can be manual or motor driven. Screw types are more reliable than hydraulic types but are slower and produce less pressure. Motorised presses are faster than manual or animal types but are more expensive. Hydro-distillation, steam distillation, steam and water distillation, soxhlet extraction are also some of the conventional methods used for extraction of essential oil. Hydro-distillation was the method used in primitive countries. Although the method was simple but suffered from several

disadvantages. The risk involved with this process was that the still can run dry or be overheated, burning the aromatics and resulting in an essential oil with a burnt smell. In order to overcome the drawback microwave assisted hydro-distillation was used to enhance the quality of the extract and also to reduce the operation cost (Cox, 1988). Soxhlet extraction is the method used for isolation of oil from plant material. In order to obtain a good yield from extraction as well as to prevent the uses of volatiles, correct choice of solvent is very important. The disadvantage related to this technique is use of hazardous and flammable liquid organic solvents, potential toxic emission during extraction, non-selective extraction, time consuming process (Naude *et al.*, 1998). Various types of essential oil are also extracted using steam distillation as it is cheaper, does not require any solvent and is also safer than other methods (Govender, 2010; Agarwal *et al.*, 2012; Prasad *et al.*, 2012). However the initial cost of equipment in steam distillation is higher and also more care has to be taken during the process. The development of new extraction techniques for the food industries has received a lot of attention due to the environmental restrictions, the need for minimizing the energy costs, and human health regulations (Coelho *et al.*, 1996). The new extraction techniques have shortened the extraction time, reduced the solvent consumption, and results in special care for thermolabile constituents. Novel extraction methods used nowadays include microwave assisted extraction, supercritical fluid extraction, ultrasound assisted extraction and pressurized solvent extraction (Sukhdev *et al.*, 2008; Vivekananda *et al.*, 2007).

Ultrasound assisted extraction

Ultrasound is an emerging technology that has been utilized in food science for processing, preservation and extraction. Ultrasound imparts positive effects in food processing in terms of productivity, yield and selectivity with better processing time, food preservation, assistance of thermal treatments and is environmentally friendly (Knorr *et al.*, 2011; Chemat *et al.*, 2004). Ultrasound offers advantages over traditional analytical techniques because measurements are rapid, precise, fully automated, can facilitate the extraction process of a variety of

food components from plant and animal source (Zbigniew *et al.*, 2007; Viikhu *et al.*, 2008).

Ultrasound is a form of energy generated by sound waves of frequency range that encompasses from 20 KHz (that exceeds the hearing limit of human) to GHz with division between power Ultrasound (20-100 KHZ) within which cavitation is a predominant force, and diagnostic ultrasound (5 MHz-GHz) (Luque *et al.*, 2007). Ultrasound assisted extraction is an emerging potential technology that has been successively used in extraction field. Ultrasound waves alter the physical and chemical properties of the subjected plant material and due to cavitation results in the release of extractable compounds. The collapse of cavitation bubble creates a transitory hot spot generating extreme temperature (5000 K) and pressure (1000 atm), which can accelerate dramatically the chemical reactivity into the medium (Flint and Suslick, 1991; McNamara *et al.*, 1999). Fig. 1 shows the chemical effect generated by cavitation. The extend of cavitation can be determined by energy and intensity along with the medium viscosity, surface tension, vapour pressure, presence of solid particle and temperature and pressure of treatment (Patist and Bates, 2008).

Two types of ultrasound equipment are generally used in laboratory. First one is the ultrasound cleaning bath used for solid dispersion into solvent, where the sizes of the solid particles are reduced which enhances its solubility. The ultrasound baths are less used for chemical reaction. The second one is the ultrasonic probe or horn system. This is more powerful because the ultrasonic intensity is delivered on small surface compared to the ultrasonic bath. A liquid medium and a source of high energy vibration are the two main requirements. The vibrational energy source is called a transducer which transfers the vibration to probe which is in direct contact with the processing medium (Patist and Bates, 2007).

High intensity ultrasound is used as an inexpensive alternate method for extraction of a variety of food components (e.g oil, protein) and bioactive ingredients (e.g antioxidants) from plant and animal source. Ultrasound assisted extraction shows no significant changes in the functional and structural properties of most bioactives and is thus more favourable for

thermally unstable compound (Wu *et al.*, 2001; Soria and Villamiel, 2010). Ultrasound assisted extraction has been recognised to improve efficiency and reduce extraction time of edible oil (Babaei *et al.*, 2006). In some cases, the efficiency of extraction increased at low temperature, thus producing a power product in a shorter time (Mason *et al.*, 1996). Ultrasound assisted extraction resulted in 2 fold increase in the extraction of carvone and liminene from caraway seeds (Chemat *et al.*, 2004). Ultrasound in combination with supercritical carbon dioxide significantly improved the extraction rate of amaranth oil from seeds (Brumi *et al.*, 2002), Almond oil (Riera *et al.*, 2004), Gingerols from ginger (Balachandran *et al.*, 2006).

The effect of different solvents and ultrasound extraction was investigated in carnosic acid from rosemary. It was seen that ultrasound improved the relative performance of ethanol as compared to other solvents like butanone and ethyl acetate alone. Thus it is possible to enhance the aqueous extraction where organic solvents can be replaced by generally recognised as safe (GRAS) solvents (Vilku *et al.*, 2008; Albu *et al.*, 2004). Flaxseed has been proved to give great recovery results under ultrasound assisted extraction as compared to conventional extraction (Zhang *et al.*, 2008). The application of ultrasound increased the yields of flavonols as compared to traditional extraction process at same conditions. Higher yield with reduced extraction time

Table 1: Performance of ultrasound assisted extraction on various food components

Product	Ultrasound Process	Solvent	Performance	Author
Carnosic acid from rosemary	Batch, 40 kHz	Butanone and ethyl acetate	Reduction in extraction time	Albu <i>et al.</i> , 2004
Polyphenols, amino acid and caffeine from green tea	Batch, 40 kHz	Water	Increased yield at 65C, compared with 85C	Xia <i>et al.</i> , 2006
Oil from seeds of semi oriental tobacco	Batch, 40 kHz	Hexane	Relatively high yield at 25 C in 20 min	Ivana <i>et al.</i> , 2007
Ginger	Batch, 20 kHz	Supercritical carbon dioxide	30% increased yield or extraction time reduction	Balachandran <i>et al.</i> , 2006
Almond oil	Batch, 40 kHz	Hexane	Increased oil recovery and reduction in extraction time	Zhang <i>et al.</i> , 2009
Pomegranate oil	Batch, 20 kHz	Hexane	Extraction yield obtained was 60%, solvent amount reduction and extraction time reduction	Goula <i>et al.</i> , 2013
Tomato seed oil	Batch, 28-34 kHz	Hexane	Extraction time reduction, 60 min at 60C	Kamazani <i>et al.</i> , 2014
Papaya oil	Batch, 40 kHz	Hexane	Shorter extraction time (30 min) and maximum yield and stability	Samavam <i>et al.</i> , 2014

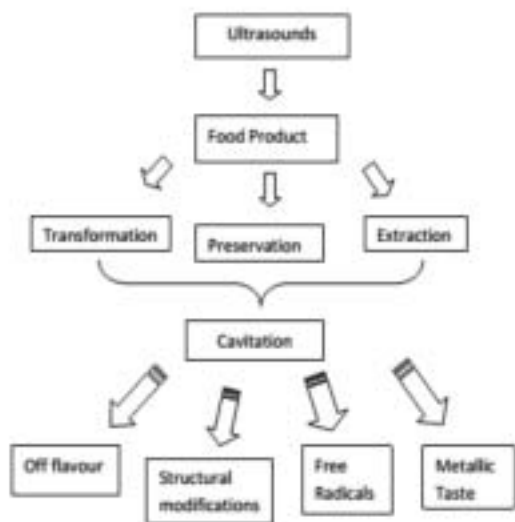


Figure 1: Chemical effects generated by cavitation phenomena.

was observed in almond and apricot seeds which were pre-treated with ultrasonic before aqueous oil extraction and aqueous enzymatic oil extraction (Shah *et al.*, 2006). Table 1 shows the effect and performance of ultrasound assisted extraction on various food components.

Apart from the benefit modification in food quality parameters, ultrasound also results in quality impairment of some products by the appearance of off flavours, modification in physical parameters and degradation of major and minor compounds. Acoustic cavitation is capable of producing radicals in the liquid medium and molecules such as OH and H radicals which accumulate on the surface of the cavitation bubble, which can be responsible for initiating formation of degradation products that can trigger radical chain reaction and provoke substantial quality defects in those products (Czechowska *et al.*, 1983). The effect of ultrasound treatment was studied on processing of sunflower, olive and soybean oils and significant negative changes were found in their composition due to oxidation produced during treatment (Cheong *et al.*, 2004). OH radicals formed in extraction process can be quenched by the addition of ascorbic acid and ethanol (Ashokkumar *et al.*, 2008).

Supercritical fluid extraction

The unique properties of supercritical fluid have prompted their use for a variety of application in analytical disciplines (Jerry, 1989). It can be used as one of the method for extraction of oil from oilseeds as it has attracted considerable attention in recent years as a promising alternate to conventional extraction methods (Taylor *et al.*, 1993; Norulaini *et al.*, 2009). Supercritical fluid extraction is used on a commercial scale for the extraction of essential oil, pharmaceutical products and in textile industries (Abbas *et al.*, 2008; Knez *et al.*, 2014). The supercritical fluid extraction is a separation process that makes use of supercritical fluid as the solvent. A supercritical fluid is the substance at a pressure and temperature above its critical point. It can diffuse through solids like a gas and dissolve material like a liquid (Sapkale *et al.*, 2010). Carbon di oxide is the most promising solvent used in the supercritical fluid extraction due to its critical temperature and pressure being 31 °C and 73.8 bar respectively, as well as compatibility with solutes, lack of toxicity, non-flammable, odourless and are available at a reasonable rate (Sihvonen *et al.*, 1999; King and France, 1992). Carbon di oxide can be used for the separation of many compounds having different polarity and molecular compound. In supercritical fluid, the density, diffusivity, viscosity and dielectric constant of a given fluid can be controlled by changing the pressure or temperature without crossing the phase boundary (Bravi *et al.*, 2007). However carbon dioxide is not considered as a good solvent for high molecular weight and polar compounds. Hence, the salvation characteristics of supercritical carbon dioxide can be modified by the addition of an entertainer such as ethanol, methanol. These entertainers interact with the solute and significantly increase the solubility (Brunner, 2005).

Based on specific requirements, the design of a supercritical fluid extraction system can be simple or complex. Supercritical fluid extraction can be performed in both pilot and industrial scale. Samples are charged in the extraction cell and the chamber is equipped with temperature controllers and pressure vessels at both ends to keep desired extraction conditions. The fluid is pressurized in the

Table 2: Application of supercritical fluid extraction in food products

Product	Analyte	Pressure (MPa)	Temperature (C)	Reference
Dried orange peel	Essential oil	10.0- 28.0	40-50	Blasco <i>et al.</i> , 1999
Aromatic Plants	Essential oil	20.0	40	Blasco <i>et al.</i> , 1999
Eucalyptus leaves	Oil with high antioxidant activity	20.0	50	Fadel <i>et al.</i> , 1999
Hops	Humulone, lupulone and essential oil	20.0	40	Langezaal <i>et al.</i> , 1990
Cassia tora L seeds	Volatile oil	250 bar	45	Zhang, 2007
Chamomile	Essential oil	250 bar	40	Kotnik <i>et al.</i> , 2007
Black cumin	Essential oil	400 bar	40	Alhaj <i>et al.</i> , 2008
Ginger	Essential oil	200 bar	80	Zhannan <i>et al.</i> , 2009
Coriander	Volatile oil	90 bar	40	Grosso <i>et al.</i> , 2008
Silkworm pupae	Oil	200-300 bar	34- 45	Wei, 2009
Hyssop	Essential oil	90 bar	40	Langa, 2009
Fish by products	Fish oil	250 bar	40	Nuria <i>et al.</i> , 2012
Palm kernel oil	Residual oil	413.6 bar	70	Nik <i>et al.</i> , 2012

extraction tank with the help of pumps, which are also needed for the circulation of the fluid in the system. If carbon di oxide is used as a solvent, then entertainers are used to increase the solubility. From the cell, the fluid and the solubilized components are transferred to the separator where the solvation power of the fluid is decreased by increasing the temperature or by decreasing the pressure of the system. The product is then collected via a valve which is located in the lower part of the separator (Perrigo and Jyont, 1992; Brunner, 2005; Bravi *et al.*, 2007).

Supercritical fluids are used for the extraction of valuable products as many natural compounds such as vitamins, aromas, natural pigments or essential oils are good soluble in supercritical fluids (Gupta and Shin, 2006; Skerget and Knez, 1997). Compared to conventional methods where the valuable products has to be thoroughly cleaned to remove the residual solvent, supercritical fluid extraction process results in complete removal of solvent from products by depressurization with better stability of compounds due to lower process temperature (Marr and Gamse, 2000; Capuzzo *et al.*, 2013). Faster extraction and higher yields have been obtained from the extraction of the essential oil of *Verbena officinalis* using Supercritical fluid extraction as an extraction process

(Safaralie *et al.*, 2008). Supercritical fluid extraction has been compared to conventional technique and has shown to be more effective for the extraction of antimicrobial compounds (Liu *et al.*, 2009; Michelin *et al.*, 2009). Residual oil was successfully separated from the palm kernel cake matrix using supercritical carbon di oxide (Rahman *et al.*, 2012). Supercritical carbon di oxide has been used for the extraction of grape seed oil (Lao and Ito, 2003), pumpkin seed oil (Yu *et al.*, 2005), corn oil (Lopes and Gil, 2005), Canola seed oil (Razori and Temelli, 2001), wheat germ oil (Shaoa *et al.*, 2008). Table 2 shows the application of supercritical fluid assisted extraction in different food products.

Supercritical fluid has been proved to be effective in the separation of essential oil producing high quality oil with more satisfactory composition (Ehlers *et al.*, 2001; Ozer *et al.*, 1996). Supercritical fluid extraction is performed at low temperature, hence is ideal technique for thermally labile compounds (Dron *et al.*, 1997). In supercritical fluids, the solvents strength can be varied by change in the pressure and to a lesser extent in the temperature. Supercritical fluids are inert; non-toxic can be readily disposed off after extraction (Sapkale *et al.*, 2010)

Microwave assisted extraction

Microwaves are electromagnetic fields in the frequency range of 300 MHz to 300 GHz. Two perpendicular oscillating fields are used to make these are electrical fields and magnetic fields. In late 1970's microwave energy was used in analytical laboratories as a heating source (Samra *et al.*, 1975). The main reason for increased interest in microwave assisted extraction lies in its fast heating which results in much shorter operation time; the application in food is performed at frequencies of 915 MHz at industrial scale and 2450 MHz in domestic ovens (Routray *et al.*, 2012).

The microwave assisted extraction process is a high speed method used to selectively extract target compounds from various raw materials. Microwave assisted extraction has been considered as an important alternative in extraction technique because it reduces the extraction time and solvents, selectivity, volumetric heating and controllable heating process, minimizes environmental impact by emitting less CO₂ in atmosphere (Farhet *et al.*, 2009). The isolation of herbal essential oil using microwave assisted extraction is an interesting alternative as it provides more effectiveness than other conventional extraction processes (Bousbia *et al.*, 2009).

Table 3: Dielectric constants and dipole moment values of commonly used solvents

Solvent	Dielectric Constant (20 °C)	Dipole Moment (25 °C) (Debye)
Hexane	1.89	<0.1
Toluene	2.4	0.36
Dichloromethane	8.9	1.14
Acetone	20.7	2.69
Ethanol	24.3	1.69
Methanol	32.6	2.87
Water	78.5	1.87

In microwave assisted extraction of analyte from the matrix is governed by the factors like solubility of the analyte in the solvent, the mass transfer kinetics of the analyte from the matrix to the solution phase, the strength of analyte interaction. The rate of diffusion increases due to higher temperature and swelling of the matrix and promote faster extraction kinetics (Poole *et al.*, 1996). The solvent used in microwave assisted extraction depends on the solubility of the target analyte, interaction between the solvent and plant matrix and by the microwave

absorbing properties of the solvent. The extraction time of 15-20 minutes is considered to be sufficient but excellent recovery has been demonstrated at even 40 seconds. Solvents like water, ethanol and methanol may heat up tremendously on longer exposure thus risking the future of thermolabile constituents (Marie *et al.*, 2004).

Two types of microwave assisted extraction system are commercially available: closed vessel system and open cells. Closed vessel systems are generally used

Table 4: Extraction conditions of different botanical species using microwave assisted extraction

Botanical species	Sample size	Extraction conditions	Reference
<i>Xylopiia aromatic</i> (Lamarck)	100g	2000 ml water, 800 W, 30 min	Stashenko <i>et al.</i> , 2004
<i>Coriandrum sativum</i> L	100g	1000 ml water, 500 W, 60 min	Kosar <i>et al.</i> , 2005
<i>Anethum graveolens</i> L	100 g	1000 ml water, 500 W, 60 min	Kosar <i>et al.</i> , 2005
<i>Lavandula angustifolia</i> Mill.	80g	1500 ml water, 500 W, 20 min	Iriti <i>et al.</i> , 2006
<i>Zataria multiflora</i> Boiss	60 g	1200 ml water, 990 W, 120 min	Golmakani <i>et al.</i> , 2008
<i>Satureja hortensis</i>	30 g	600 ml water, 660 W, 180 min	Rezvanpanah <i>et al.</i> , 2008
<i>Satureja montana</i>	60g	1200 ml water, 660 W, 90 min	Rezvanpanah <i>et al.</i> , 2008
<i>Eryngium foetidum</i> L	300g	100 ml water, 900 W, 27 min	Thi <i>et al.</i> , 2008

Table 5: Extraction of essential oil using microwave extraction technique

Essential oil	Type of Equipment	Benefits	Reference
<i>Cuminum cyminum</i> / <i>Zanthoxylum</i> <i>bungeanum</i>	Microwave oven with stirrer, with a microwave absorption medium	Rapid and energy efficient method	Want and Weller, 2006
<i>Eletaria</i> <i>cardamomum</i>	Focused microwave oven	No solvent/reduces extraction time	Lucchesi <i>et al.</i> , 2007
<i>Lavandula</i> <i>angustifolia</i> Mill, Lamiaceae	Microwave accelerated steam distillation	Superior to steaistillation in terms of energy saving, rapidly (product yield, cleanliness and product quality)	Chemat <i>et al.</i> , 2006
<i>Laurus nobilis</i>	Microwave oven with stirrer, with water as solvent	No solvent, safe energy efficient	Flamini <i>et al.</i> , 2007
<i>L. angustifolia</i> Mill. Lamiaceae	Microwave steam distillation	Significantly faster than steam distillation, reduced costs	Sahraoui <i>et al.</i> , 2008

for extraction under drastic conditions, since the solvents may be heated to about 100 C above their atmospheric boiling point (Barnabas *et al.*, 1995; Jassie *et al.*, 1997). The maximum power delivered in closed vessel system is about 600-1000 W (Pare 1990, 1991; Young, 1995). Filtration is necessary to remove the solid residue. In open cells, maximum temperature is determined by the boiling point of the solvent used (Renoe, 1994; Letellier *et al.*, 1999). In open cells, larger samples can be extracted and it offers more safety in sample handling as compared to closed systems. In order to avoid filtration step, the sample to be extracted is placed into soxhlet type cellulose cartridge. Collin *et al.* (1991) compared Microwave assisted extraction with classical hydrodistillation for extraction of essential oils from 10 different species. The yields were generally similar but the chromatographic profiles varied dramatically. Volatile compounds of *Vitex pseudo-negundo* samples were extracted by steam hydrodistillation and microwave assisted hydrodistillation. Extraction yield were achieved at significantly shorter extraction time when using microwave instead of conventional hydrodistillation (Farjam *et al.*, 2014). Microwave assisted extraction was performed to obtain essential oil from two different herbs (basil and epazote). Significant reduction in time, solvent and no change in the yield of essential oil was shown with respect to steam

distillation (Gabriel *et al.*, 2013). Saoud *et al.* (2005) carried out investigation of microwave extraction of eucalyptus essential oil using ethanol as solvent. Microwave exposure time of 3 minutes was enough for microwave extraction process. Extraction of essential oil using microwave assisted extraction has been summarised in Table 5.

The importance of microwave assisted extraction lies in the performance of heating source. The high temperature reached by microwave heating reduces dramatically the extraction time as well as the volume of solvent required (Kaufmann and Christen, 2002). Extraction time is shorter (15-30 minutes). Temperature and pressure sensors provide exact reaction control. Microwave assisted extraction is a novel extraction method for the extraction of nutraceuticals. This is the simple and rapid method and thus suitable for thermolabile constituents (Wang and Weller, 2006). However an additional step of filtration or centrifugation is necessary to remove the solid residue. The efficiency of microwave reduces when the target compound or the solvent used are non-polar or if they are volatile.

Conclusions

Novel extraction methods in the food industry were reviewed in this article. Ultrasound assisted extraction, microwave assisted extraction and

supercritical fluid extraction is proving beneficial to food industries than the primitively used solvent extraction methods. The importance of microwave assisted extraction lies in the performance of heating source. The high temperature reached by microwave heating reduces dramatically the extraction time as well as the volume of solvent required. Supercritical fluid is an effective process for separation of essential oils, producing high quality oil with more satisfactory composition and is performed at low temperature, proving ideal technique for thermally labile compounds.

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