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A review on developments in oil extraction from oilseeds

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ABSTRACT

Oilseeds have formed the backbone of agricultural economies from time immemorial. They are mainly used for edible oil production. Oils are generally plant based lipophilic compounds. Lipids are triglycerides chemically being fatty acid esters of glycerol. Natural antioxidants need to be protected to enhance shelf life of lipids and beneficial effects on human health to enhance the nutritional quality of oil. Extraction of oil is the first step in the refining process. Oils and fats are extracted from their original source using a variety of different methods. Before the development of solvent extraction techniques, the oil extraction industry was based on mechanical methods such as hydraulic pressing and continuous screw pressing. Oil extraction methods ever reported have been discussed in detail here.

***Keywords*:** Dry extrusion, enzymatic treatment, microwave system, pulsed electric field treatment, solvent extraction, supercritical fluid extraction and ultrasound or sonication

Being a high value agricultural commodity, oilseeds from times immemorial have formed the backbone of several agricultural economies. They are mainly used for edible oil production, the oil being an important source of fatty acids. The most commonly known oilseeds are groundnut, soybean, palm kernel, cotton seed, olive, sunflower seed, rapeseed, sesame seed, linseed, safflower seed, *etc*. Oils are generally plant based lipophilic compounds. Lipids are triglycerides chemically being fatty acid esters of glycerol. The fatty acids make up the major component of fats and oils consumed as food while the minor components include mono and diglycerides, free fatty acids, phosphatides, sterols, phytosterols, fatty alcohols, fat soluble vitamins, tocopherols, carotenoids, chlorophyll and other substances (Anthea *et al*., 1993). The major oil portion in its free form is found within the vacuoles while the remaining significant portion lies dispersed within the cytoplasm in the form of minute droplets bound to colloids.

Extraction of oil is the first step in the refining process. Oils and fats are extracted from their original source using a variety of different methods. Before the development of solvent extraction techniques, the oil extraction industry was based on mechanical methods such as hydraulic pressing and continuous screw pressing. In conventional screw pressing operations, the beans are subjected to dry heating for sufficient length of time and under temperature of 116-132° C (Nelson *et al*., 1987) causing cell disruption. This causes a reduction of moisture content to 2-5%, which is preferred in commercial expelling operations. The hot oil is initially released within the matrix but later extracted under pressure. Prior to the development of solvent extraction techniques, the oil extraction industry was based on mechanical methods such as hydraulic pressing and continuous screw pressing, defattening and deoiling the oilseeds. Pressing is usually viable when the fat content is relatively high (>25% by weight) and residual fat content in the meal is around 5per cent by weight, whereas solvent extraction are suitable for oilseeds with low fat content and residual oil content in the meal is reduced to Â1% by weight (Erickson *et al*., 1984; Hamm and Hamilton, 2000). Conventional expelling methods emphasizes on oil recovery and in doing so, often results in excessive heating of the cake in the process darkening and deteriorating the oil. In all the works which have been performed, hexane appears to be the most commonly used solvent (Pérez-Serradilla *et al*., 2007). (Li, 1999) studied the extraction, recovery and purifying lipophilic ingredients contained in plant tissue. To enhance the mass transportation of the target compounds mechanical agitation to disperse the cell matrix and solvent extraction are conventional extraction procedures. The use of the extraction by solvent allows its vaporization by recovery of its residual oil (Meziane and Kadi, 2008) but the disadvantages that limit their commercialization potential include oil degradation due to high thermal and pressure stresses, reduced mass transfer coefficient requiring longer extraction periods, and requirements of large amounts of extracting agents (Eskilsson and Bjorklund, 2000; Kaufmann and Christen, 2002).

Natural antioxidants need to be protected to enhance shelf life of lipids and beneficial effects on human health and enhances the nutritional quality of oil (Dorman *et al*., 2003; Senorans *et al*., 2000; Thorsen and Hildebrandt, 2003). Oil extraction methods have been

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reported extensively by several authors (Salgin *et al.*, 2006; Li *et al.*, 2004; Luque-Garcia and de Castro, 2004; Orsat *et al.*, 1996; Yoshida *et al.*, 1991) would be discussed in the review.

Solvent extraction

The high solubility of oil in organic solvents forms the basis of this extraction method. Preferential solubilization of the compound of interest and low boiling point for facilitating the removal by distillation are the preferred solvent characteristics, in addition they should be insoluble in water, readily available, inexpensive and reusable. The polarity of the solvent should also match that of oil to effectively penetrate the cells. Bligh and Dyer is a popular method using a combination of solvents like chloroform, methanol and water. The polarity of water is reduced under high pressure condition thus enhancing the solubility of water which thereby allows extraction of otherwise insoluble compounds. Accelerated solvent extraction (ASE) refers to the usage of organic solvents at high temperature and pressure to enhance the extraction quality. Elevated temperatures assists in enhancing solubility, decreasing viscosity thereby allowing better sample penetration into the matrix and increasing analyte mass diffusion rate (Erickson *et al.*, 1984). Simultaneous extraction and trans-esterification can be implemented to recover fatty acids. Trans esterification enhances oil yield when solvents are added in order of increasing polarity. Similarly concurrent solvent extraction and saponification also enhances oil extraction.

Highly polar solvents are commonly employed for extraction of bioactive compounds (Spigno and de Faveri, 2009) such as antioxidants, isoflavones and pigments from a variety of crops (Pan *et al.*, 2003; Rostagno *et al.*, 2007; Terigar *et al.*, 2010a). Terigar *et al.*, (2010)b reported that 20 per cent more rice bran oil was extracted with ethanol (20 ± 0.21% of dry mass) as compared to n-hexane (16.05 ± 0.35% of dry mass). The presence of certain ethanol soluble proteins in the rice bran and an ethanol mediated extraction at elevated temperature may have contributed to the difference. In case of food samples, the major limitation of the process is solvent residue. Often moisture plays an important role acting as barrier restricting solvent access to cells thus decreasing process efficiency.

Supercritical fluid extraction

Usage of supercritical ûuids has gained acceptance due to its ability to extract high-value products. The extraction and separation are quick and best suited for thermally sensitive products (Sahena *et al.*, 2009). It also leads to production of high-quality and solvent free extracts (Reverchon, 1997). The unsaturated acids are protected by inert atmosphere of CO_2 and the relatively low extraction temperature ensures superior product quality. The most favoured fluid for extraction is CO_2 as it behaves both as solid and liquid when raised above critical temperature and pressure thereby increasing its solvent property Sovova, H. 1994. Several models of supercritical carbon dioxide extraction from ground seeds have been published which describe extraction rate using either mass transfer coefficient in the solvent phase (Lack, 1985) or mass transfer coefficient in the solid phase (Pekhov and Goncharenko, 1968). The extraction efficiency is dependent on pressure, temperature, CO_2 ûow rate and extraction time (Harun *et al.*, 2010, Sahena *et al.*, 2009). This method has been investigated to obtain oil fractions with high concentrations of vitamins, especially b-carotene and tocopherols (Birtigh *et al.*, 1995; de Franca *et al.*, 1999). Often co solvents are added to optimize extraction which alters viscosity. Ethanol, when added, helps in increasing the solvating power by increasing the polarity thus facilitating efficient extraction even at low temperature and pressure. The advantages of supercritical fluid extraction include retention of high quality materials usually present in low amount.

The investigations of the applicability of supercritical fluid extraction to the deacidification of olive and husk oils were performed by Brunetti *et al.*(1989) and Goncalves *et al.*(1991). Ronyai *et al.* (1998) studied the nutritive value and the phospholipid content of defatted proteins of the oil extracted from corn germ obtained with supercritical CO_2. Friedrich *et al.* (1982) found that the soybean oil extracted with CO_2 was lighter in color and contained less iron and about one-tenth the phosphorus of hexane-extracted crude oil. Extraction of the rapeseed oil with supercritical carbon dioxide was investigated by several groups of workers (Lee *et al.*, 1986; Brunner, 1984). High moisture hinders the process of extraction by acting as barrier between the solvent and sample. Mass transfer co efficient both in case of CO_2 diffusing into the matrix and lipid in solution diffusing out of the tissue matrix is hampered. List *et al.*, 1993; List *et al.*, 1984 reported that supercritical CO_2 extracted oils are devoid of natural antioxidants (phosphatides), in is another impediment to the popularization of the technique. The technology also suffers from the high cost involved as reported by Oszagyan *et al.* (1996), Temelli (1992) and Temelli *et al.* (1988).

Ultrasound or sonication

A potential new technology that improves extraction of lipophilic compounds from plants is usage of high intensity ultrasound. Ultrasonication have been reported to dramatically improve oil extraction from plant materials (Cravotto *et al*., 2008; Zhang *et al*., 2008) and also has the advantage of being incorporated with other methods of extraction (Shah *et al*., 2005). The mechanical effect of ultrasonication promotes the release of soluble compounds from the plant body by disrupting cell walls thus enhancing mass transfer and facilitating solvent access to the cell content (Vinatoru, 2001). This effect is much pronounced at low frequencies 18-40 kHz and is practically negligible at 400-800 kHz. The extraction using ultrasonic sound waves causes cavitation, the process whereby the existing pressure of liquid becomes lower than its vapour pressure leading to the formation of bubbles which expands and contracts depending on the variation of the applied pressure and often collapses thereby damaging the cell structure and releasing its contents. Acoustic cavitation also causes the agitation of the solvent permitting greater penetration into the cell matrix.

The main advantage of ultrasonication includes reduction of extraction time and solvent consumption (Wu *et al*., 2001). In addition the extraction process can be carried out at a lower temperature thus avoiding thermal damage to the extracts and minimizing the loss of bioactive compounds (Visentainer *et al*., 2005). Fairbanks, 2001 and Mason *et al*., 1996 both reported improvement of extraction process meditated by ultrasonication. Zang *et al*. (2008) reported that the yield of flaxseed oil increases almost linearly with increasing ultrasonic power. As the power increased from 20 to 50 W, the yield of flaxseed oil increased from 66.7 to 84.9 per cent (18.2% increase). Li (2004) reported increased oil yield with increase in the intensity of ultrasonic waves. Oil yield after 3 h increased by 2.2, 10.1 and 11.2 per cent for ultrasound intensity of 16.4, 20.9, and 47.6 W/cm^2 respectively compared to the non sonicated control.

Microwave system

Microwave-assisted extraction (MAE) has advantages over conventional and other extraction methods, as intact organic compounds can be extracted more selectively and more rapidly (Pare *et al*., 1994), with lower energy consumption, reduced byproduct formation, and less solvent (Letellier and Budzinski, 1999).

Unlike conductive heating, MW heats the whole sample volume simultaneously. It disrupts weak hydrogen bonds by promoting the rotation of molecular dipoles, an effect that is opposed by the viscosity of the medium. Furthermore, the movements of dissolved ions increase solvent penetration into the matrix and thus facilitate analyte solvation. The effect is strongly dependent on the nature of both solvent and matrix. Sometimes MW affects mainly the latter, while the surrounding liquid, having a low dielectric constant, remains relatively cold.

In recent times the use of microwave assisted solvent extraction as an alternative to the conventional solvent extraction of oil from vegetal materials, has gained popularity mainly due to the reduction in extraction time and solvent consumption (Chen *et al*., 2007; Molins *et al*., 1996; Spigno and De Faveri, 2009; Kusuma and Mahfud, 2010). This method performed at atmospheric pressure and the effect of microwave radiation depends essentially on the nature of both the solvent and the solid matrix.

In case of microwave assisted extraction, rapid heat development due to the polar property of the available moisture causes a sudden buildup of pressure within the plant cells enabling disruption at microscopic level (Terigar *et al*., 2010a). This enhanced extraction in MASE can be explained as follows: the internal heating of the in situ water within the sample (residual water) accelerates cell rupture by sudden temperature rise. This allows a rapid dissolution of the oil released from broken cells by the solvent (Lucchesi *et al*., 2007; Sun *et al*., 2007; Zhang *et al*. (2008). The temperature of water and solvent molecules inside cells on being subjected to microwave irradiation, reaches the boiling point readily leading to the formation of high pressure gradients, accelerated rupture of cell walls, and increased mass transfer rates (Bhattacharya and Basak, 2006). Oil yield increased with increase in temperature, reaching its maximum value close to the solvent boiling point, according to Fick's law (Geankoplis, 2003). Microwaves cause direct generation of heat within the volume, with important impacts on heating kinetics, and pressure effects on the cell wall membrane structure. As a result, solutes within the raw material move or partition into solvent phase and diffuse out of the solid matrix faster. Similar results have been reported by Zhu *et al.* (2006). The short exposure time to microwave radiations preserves even the most thermo labile compounds from degradation reactions and hence the oil obtained is of much better quality as compared to that obtained through conventional extraction (Anison *et al.*, 2003). The microwave heating rate is influenced by many factors *viz*. microwave power level, initial temperature, frequency, dielectric properties of the material, and design of microwave applicator.

Designing a cavity in a resonant monomode microwave such that the microwave energy remains concentrated at the cavity center, where the sample is located can be attributed to a recent development in technology (Bowman *et al*., 2008; Terigar, 2009). Microwaves at 915 MHz have much higher penetration depths into the material as compared to the frequency of 2450 MHz. Microwave penetration depths for a ethanol and soy flour mixture in the ratio of 2:1 at 915 and 2450 MHz at 50 °C was reported to be 0.0323 and 0.0081 m, respectively (Terigar *et al*., 2010b). Amarni *et al*. (2010) reported that when the power variation ranges from 180 to 720 W, the yield increase is 15.0 per cent at a contact time of 0.5 min and 17.7 per cent at 2 min. On the other hand, when the contact time varies from 0.5 to 2 min, this increase is only 8.1per cent at 180 W and 10.7% at 720 W.

Enzymatic treatment

Aqueous enzymatic oil extraction has emerged as a promising technique for extraction of oil from plant materials. (Rosenthal *et al*., 2001; Sharma *et al*., 2002). The advantages of enzymatic oil extraction includes higher oil yields and also higher quality of the meal. Development of pilot and industrial processes with olive (Montedoro & Petruccioli, 1973; Santos, 1978; Alba *et al*., 1987), pilot processes with rapeseed (Olsen, 1987) and semipilot processes with coconut (Cintra *et al*., 1986) have been reported. Maximum enzyme activity is exhibited in media with water content of 35 per cent or more (Montedoro and Petruccioli, 1973), temperature and pH being the other important parameters for optimum enzymatic activity. Successive enzymatic as well as mechanical and thermal treatment damages the cell wall thereby favouring oil permeability. Enzymes like amylase, glucanase, protease, pectinase, as well as cellulolytic and hemicellulolytic enzymes, prepared from vegetable cell degrading microorganisms have been used to enhance the extractability of oil (Fullbrook, 1984). As, in the oilseeds, oil is present in intracellular vacuoles linked to other macromolecules, its extraction is enhanced by the hydrolytic action of carbohydrases, thus justifying the use of exogenous enzymes to increase the oil recovered. The mild conditions so employed guarantees a higher yield and preservation of valuable extracted components (Olsen, 1988). Assay of several thermophilus moulds by Bhatnagar and Johari (1987) revealed that the enzymes secreted by them helped to improve oil recovery to a greater degree than purified cellulose or hemicellulose.

Pulsed electric field treatment

In case of Pulsed Electric Field, the application of direct current (d.c.) electric field to enhance the solid/ liquid expression was intensively studied in the literature (Orsat *et al*., 1996 and Yoshida *et al*., 1991) This method is based on the electro-kinetic phenomena on the liquid/ solid interface and permits to remove some quantity of liquid due to combined effect of pressure and electro osmosis. The increasing extraction yield by electrical treatment was explained as a result of electrical breakage of cells named electroplasmolysis (Lazarenko *et al*., 1977) and reported that very short electrical pulses of high enough intensity might be effective method of non-thermal electrical breakage of cells.

Application of direct current through a material placed between two electrodes constitutes pulsed electric field treatment whereby high voltage pulses are applied for very short period of time ranging between microseconds to milliseconds. This technology has proved to be an effective method for irreversible cell membrane permeabilization in case of plant and animal tissues without any significant increment of temperature of the sample and involving low cost operation (Toepfl *et al.* 2006). PEF application induces permeabilization of cell membranes and facilitates its subsequent rupture by mechanical compression. Application of pulsed electric field (PEF) for enhancing the extraction yield of juices from fruits and vegetables, reducing the drying times or improving the extraction of intracellular valuable compounds such as colorants, sucrose, or polyphenols have all been investigated in studies conducted in laboratories and in pilot scale basis (Donsì *et al.* 2010; Knorr *et al.*, 2011; Vorobiev and Lebovka 2008). Several studies have been conducted to understand the quantum of improvement in the extraction of different vegetable oils such as maize, soybeans, or rapeseeds (Guderjan *et al.*, 2005; Guderjan *et al.*, 2007) through the application of a PEF pretreatment. Sánchez-Gimeno *et al.* (2010) reported that application for a longer time period or more intense electric field strengths did not increase the oil extraction yield. Hence, to increase efficiency of extraction studies were done employing higher temperatures and longer period of malaxation (Torres and Maestri 2006; Cruz *et al.*, 2007; Espínola *et al.*, 2009). Abenoza *et al*., 2013 reported application of a PEF treatment to the olive paste resulted in an improvement in the extraction yield. The extraction yield improved by 23 and 54 per cent respectively when the olive paste was treated with PEF at 1kV/cm and 2 kV/cm. At 15 °C, the permeabilization of the olive cells by a PEF treatment of 2 kV/cm improved the extraction yield by 14.1 per cent, which corresponded with an enhancement of 1.7 kg of oil per 100 kg of olive fruits.

Dry extrusion

Extrusion is a convenient method where both disruption of tissue and heating is occurring. The rise in temperature is rapid and the residence time is less which is the primary reason of retention nutritional value of oil. Extrusion as pre treatment before solvent extraction increases yield reported by Nelson *et al*., 1987. The extrudate is granular till there is a considerable rise in temperature and with higher temperature the extrudate turns into a viscous liquid. Due to compression shear force develops which releases the oil within the matrix and due to increase in temperature the viscosity of the oil is reduced. The process parameters that affect fluidization are feed moisture, feed rate, pressure on the die, and the configuration of the extruder. The oil yield also increased when the extrudate was pressed immediately after emergence.

However, once the extrudate emerges, the fluid nature is rapidly lost and the mass becomes granular. Re-absorption of the oil into the matrix, drop in oil viscosity (due to drop in temperature), and flash evaporation of residual moisture may be factors contributing to the observed reversal of the physical state. It is important to control extrusion parameters so that the extrudate emerges from the die in a semi-fluid state. It was possible to obtain fluidization of the extrudate over a range of temperatures by manipulation of the above process parameters. In general, fluidization occurred at extrusion temperatures above 121 °C. However, the material tends to scorch when the temperature rises above 148 °C.

Conclusion

It is the continuous endeavor of researchers to improve quality and produce nutritionally superior edible oil. To this effect, it is not only necessary to develop new varieties with improved oil yields but also to enhance the extraction efficiency. Recent developments in the field of technology, aids in enhanced production and as well as improvement of the nutritional profile of several conventional oils. Solvent extraction is the most accepted technique for extraction of oil. Often solvent extraction is coupled with mechanical disruption to obtain better yield. There are various novel technologies being used at present like the use of pulse electric field, enzymes, microwave assisted extraction, ultrasonic energy on extraction; but the effect of these methods on the chemical stability of valuable compounds which are prone to oxidation (*i.e*. omega-3 fatty acids) remains to be investigated. These recent developments have resulted in significant oil extraction yields in some cases but needs to be implemented on a large scale for acceptance.

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