

Encapsulation of Vegetable Oils for Enhancing Oxidative Stability of PUFA

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ABSTRACT: Vegetable oils are rich in essential fatty acids such as, mono-unsaturated and poly-unsaturated fatty acids which encourage the preservation of the quality. Poly-unsaturated fatty acids (PUFAs) have exceptional benefits in development and growth, brain functioning and disease control in humans. The reactions like oxidation, isomerization and polymerization change fatty acids profile, which further contribute to degradation in quality of vegetable oils and development of off-flavour in food products. Encapsulation of vegetable oils provides oxidative stability by preventing direct contact of oxygen, light, radicals and heat to the oil. It offers functional barrier to oxidation and masking of unfavorable flavors. Several techniques like, spray-drying, freeze drying, microwave drying, coacervation, particles from gas saturated solution (PGSS) and other improved and innovative techniques have been successfully applied to encapsulate vegetable oils. The process parameters with its effect on efficiency has been reviewed for different techniques. Further, the application of encapsulated vegetable oils in various food products has been explored. From the review, the encapsulation stabilize the vegetable oils from the auto-oxidation. Improvement in peroxide value was observed during storage of encapsulated vegetable oils. Dairy and bakery products were successfully enriched with PUFAs by incorporating vegetable oil encapsulates. The pilot scale production and in-depth study of the feasibility of encapsulating vegetable oils is required.

Keywords: Encapsulation, Oil powders, Oxidative stability, PUFA, Vegetable oils.

INTRODUCTION

Oils are fundamental components of food due to its large contribution to the taste, flavour and quality of product. Vegetable oils such as flaxseed oil, sunflower oil, sesame oil, rice bran oil, groundnut oil and other edible oils are abundant in mono-unsaturated fatty acids (MUFA) and poly-unsaturated fatty acids (PUFA). Oil containing omega-3, 6 and 9 (MUFAs and PUFAs) fatty acids is an interest for its application in food, agriculture and pharmaceuticals in present era due to multi-dimensional functional properties, healthy importance to human beings and natural and safe status (Chang and Nickerson 2018). Omega-3 and omega-6 (PUFAs) fatty acids gained interest due to exceptional benefit such as lowering of low-density lipoprotein (LDL). The structures of some of the omega-3 and omega-6 fatty acids are shown in Fig. 1. Omega-3 (ω -3) includes α -linolenic acid (ALA), eicosapentaenoic acid (EPA) and docoheptaenoic acid (DHA) that are essential for development and growth of human body. Linoleic acid (LA) and arachidonic acid (AA) are the major omega-6 (ω -6) polyunsaturated fatty acids. Along with

omega-3, omega-6 fatty acids also play a crucial role in normal growth, development and brain functioning. It helps in stimulation of skin and hair growth, maintenance of bone health, regulation of metabolism and maintaining the reproductive system.

However, PUFAs are chemically unstable structures; sensitive to oxidation, isomerization and polymerization when they are faced with environmental stresses (Rustan and Drevon 2005). The exposure of oxygen creates cleavage in the unsaturated bonds and elevate the chances of product to be rancid. The quality of food is being degraded by oxidation of fatty acids (Ferrari, 1998). It develops off-flavours that creates negative impact on sensory attribute and limits the application of oil in several food products (Peniche, 2004; Lee and Ying 2008).

In order to maintain the oxidative stability, vegetable oils are encapsulated thereby it could be prevented from direct contact with the environmental factors such as oxygen, light, radicals and heat (Juric *et al.*, 2020). A great interest has raised to generate oil capsules in the food industries that could be added and increased a consumption of an essential fatty acids such as omega-3

and omega-6 with meals. Several techniques such as, spray-drying, coaxial electrospray system, freeze-drying, microwave drying, coacervation, in situ polymerization, melt-extrusion, etc. are reported for encapsulation of essential compounds (Tonon *et al.*, 2011; Adelman *et al.*, 2012; Hee *et al.*, 2015; Bakry *et al.*, 2016; Timilsena *et al.*, 2016; Albert *et al.*, 2017; Nanda *et al.*, 2021; Pattnaik and Mishra 2021; Vinod *et al.*, 2021). The encapsulated oil have been successfully applied in dairy, bakery and meat products for functional benefits (Delshadi *et al.*, 2020). However,

the attempts made by the researchers are lack in detailed behaviour of the materials during the encapsulation process. Further, the application part need to be elaborate for the researchers across the globe. The current review paper make readers familiar with the various aspects of encapsulating the vegetable oils, stability against oxidative degradation and utilization of encapsulates to enrich the human diet with vegetable oils by encouraging and enhancing the knowledge about the encapsulation of vegetable oils.

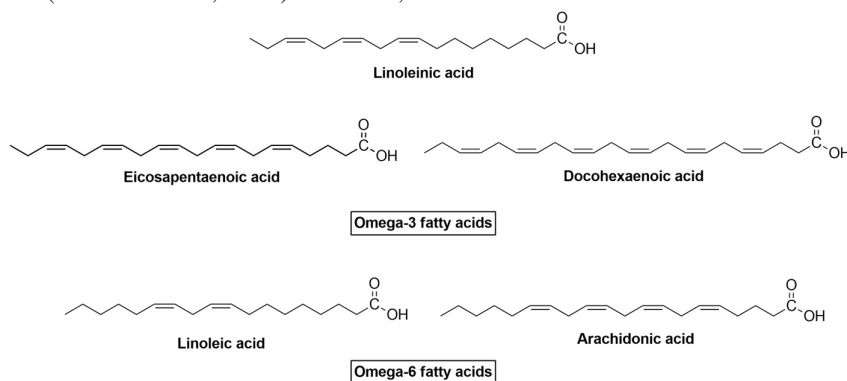


Fig. 1. Structures of essential poly-unsaturated fatty acids (PUFAs).

ENCAPSULATION OF VEGETABLE OILS

Encapsulation is the process of enveloping one substance termed as core material into another one wall material that improves stability and functional properties (Pattnaik and Mishra 2021). It is a packaging of bioactive compounds at mili, micro- or nano-scaled which isolates the compound from the environmental conditions (Panda *et al.*, 2022). The major goals of encapsulation are protection, stabilization and slow down the release of encapsulates. Various proteins and polysaccharides including gums such as maltodextrin, chitosan, lactose, gum Arabic, pectin, sodium alginate and glucose syrup and its complexes serves as a wall material (Chang and Nickerson 2018). The wall materials are food-grade and biodegradable which are able to form a barrier for separation and protection of the core (Carpena *et al.*, 2021). Further, this wall material matrix do not allow the oil to interact with other food component when it is incorporated in food products (Sanguansri and Augustin 2011). Encapsulation of oil is to be carried out in order to mask the off-flavour or colour, controlled release, increase in the stability during storage, incompatibilities with reactive substances, easy handling and more convenient way for storage as compared to sticky raw oil (Abang *et al.*, 2012). Fig. 2 shows the advantages of encapsulating essential fatty acids present in the vegetable oils in order to reduce the quality degradation.

Several methods have been attempted to encapsulate the vegetable oils. Due to inherent unsaturation of vegetable oils, some techniques failed with the reason of lower encapsulation efficiency that lead to more surface oil. Another reason is higher temperature which

degrade the compounds and deplete antioxidants and other compounds, followed by self-deterioration. Spray drying, freeze drying, microwave drying, coacervation, spray cooling/chilling and co-extrusion have been reported in different studies for the successful encapsulation of vegetable oils (Sun-Waterhouse *et al.*, 2011; Domian *et al.* 2014; Chew and Nyam 2016; Timilsena *et al.*, 2016; Domian *et al.*, 2018; Alexandre *et al.*, 2019; dos Santos Carvalho *et al.*, 2019; Mohseni and Goli 2019; Benito-Roman *et al.*, 2020; Holgado *et al.*, 2020; Pattnaik and Mishra, 2020).

Spray drying. In order to encapsulate the vegetable oils, most common technique applied in the food industry is spray drying (Desai and Park 2005). As vegetable oils are sensitive to heat, spray drying is succeeded due to short duration process. The emulsion is prepared containing wall and core materials, solvent and other ingredients such as emulsifier. The emulsion is pumped to the spray dryer and atomized into small droplets with the help of high pressure nozzle or a centrifugal wheel into hot medium, typically air. It results in transformation of a feed from the liquid solution to the solid powder (capsules) at higher temperature, generally 160-180°C (Zuidam and Shimoni 2010). Size of the particles depends on the nozzle size of the spray drying system, ranges from 10-50 micron to 2-3 mm (Kaushik *et al.*, 2015). It affects the quality and stability parameters of the encapsulated oil. Higher moisture content lead to caking, collapse, agglomeration, browning and oxidative reaction of the product (Encina *et al.*, 2016). Effect of wall materials and process parameters on encapsulation efficiency of vegetable oils using spray drying technique is reviewed critically and presented in Table 1.

Tonon *et al.* (2011) investigated on influence of inlet air temperature on microencapsulation of flaxseed oil by spray drying. The results revealed that bulk density was decreased with higher inlet air temperature, probably due to faster drying that may lead to larger volume. Particle size was increased with increase in the inlet air temperature. Encapsulation efficiency was increased for rice bran oil as the temperature was increased from 140 to 160°C. Higher temperature formulates the semi-permeable membrane rapidly and thereby stop leaching of volatiles to the surface and decrease the surface oil content (Murali *et al.*, 2016).

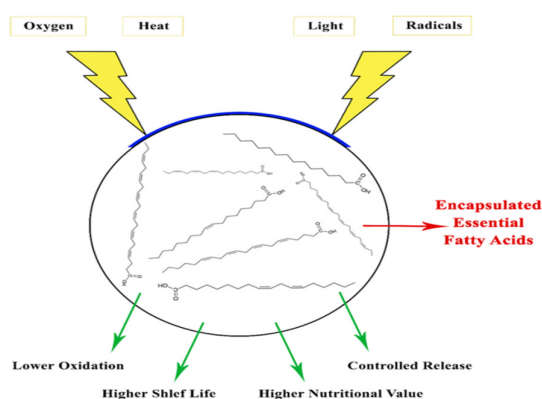


Fig. 2. Advantages of encapsulating the essential fatty acids to reduce quality degradation.

Fig oil, a nutritious vegetable based oil rich in polyunsaturated fatty acids, has been encapsulated by Icyer *et al.* (2016). Optimum condition was resulted with encapsulation ratio of 8/1/3 for maltodextrin (MD)/gum Arabic (GA)/oil and 150° temperature. Lower moisture, higher water solubility, homogeneous size of the particles and good morphology was observed with optimum condition with moderate encapsulation efficiency. Karaca *et al.* (2013a) encapsulated flaxseed oil in either chickpea protein isolate (CPI) or lentil protein isolate (LPI) and maltodextrin wall matrix with bench top spray dryer (Table 1). Encapsulation efficiency was ranged from 83.62 to 90.42%. The efficiency was decreased with increase in the oil concentration. Generally, the presence of oil on the surface had an adverse effect on characteristics of spray-dried powders such as flow and dispersion (Bae and Lee 2008). Virgin coconut oil (VCO) was encapsulated in maltodextrin (MD) and sodium caseinate (SC) as the basic wall materials mixed with gum Arabic, whey protein concentrate and gelatin (Hee *et al.*, 2015). Produced capsules were spherical in shape without fissures and cracks, low in moisture content and high in bulk density. Gallardo *et al.* (2013) encapsulated linseed oil by spray drying for the functional food application (Table 1). Linseed oil encapsulates prepared with 100% GA and mixtures of GA, MD and WPI resulted with more than 90% encapsulation efficiencies.

Table 1: Spray drying technique for encapsulation of vegetable oils.

Oil	Wall materials	Process parameters	Encapsulation efficiency (%)
Coconut oil	Gelatine and maltodextrin (MD)	Inlet temperature: 160°C Outlet temperature: 42°C Pressure: 0.35 MPa	57-82 Le and Le (2014)
Flaxseed oil	Zein	Inlet temperature: 135°C Outlet temperature: 55-60°C Feed flow rate: 9 mL/min	75.42-93.26 Quispe-Condori <i>et al.</i> (2011)
Flaxseed oil	Gum Arabic (GA)	Nozzle diameter: 1.5 mm Inlet temperature: 132-202°C Feed flow rate: 12 g/min Pressure: 0.06 MPa	51.5-92.0 Tonon <i>et al.</i> (2011)
Flaxseed oil	Maltodextrin (MD) and gum Arabic (GA)	Nozzle diameter: 1.5 mm Inlet temperature: 140°C Outlet temperature: 95°C Feed flow rate: 5.3 g/min	54.6-90.7 Rubilar <i>et al.</i> (2012)
Flaxseed oil	Whey protein concentrate (WPC) and gum Arabic (GA) or modified starch	Nozzle diameter: 1.5 mm Inlet temperature: 180°C Outlet temperature: 100°C Pressure: 0.06 MPa	37-97 Tonon <i>et al.</i> (2012)
Flaxseed oil	Maltodextrin (MD) and gum Arabic (GA), whey protein concentrate (WPC) or two types of modified starch	Nozzle diameter: 0.5 mm Inlet temperature: 180°C Outlet temperature: 110°C Feed flow rate: 12 g/min	62.3-95.7 Carneiro <i>et al.</i> (2013)
Flaxseed oil	Gum Arabic (GA), maltodextrin (MD), whey protein isolate (WPI) and methyl cellulose (MC)	Inlet temperature: 175°C Outlet temperature: 75°C Feed flow rate: 15 mL/min	25.5-91.4 Gallardo <i>et al.</i> (2013)
Flaxseed oil	Chickpea or lentil protein isolates (CPI or LPI) And maltodextrin (MD)	Nozzle diameter: 700 µm Inlet temperature: 180°C Outlet temperature: 90°C	83.62-90.42 Karaca <i>et al.</i> (2013a)
Flaxseed oil	Whey protein concentrate (WPC) or sodium caseinate (NaCas) and lactose	Inlet temperature: 170°C Outlet temperature: 75°C	84.51-86.77 Goyalet <i>et al.</i> (2015)

Flaxseed oil	Whey protein concentrate (WPC) and sodium alginate (SA)	Nozzle diameter: 0.406 mm Inlet temperature: 140°C Outlet temperature: 170°C Feed flow rate: 4 mL/min	30.69-59.88 Fioramonti <i>et al.</i> (2019)
Flaxseed oil	Polysaccharide gum (PSG)	Inlet temperature: 120-160°C Outlet temperature: 60-80°C Feed flow rate: 200-300 mL/h Atomization: 12000-20000 rpm	85.27-90.78 Shahid <i>et al.</i> (2020)
Olive oil	Gelatin, gum Arabic (GA), maltodextrin (MD), sodium caseinate (NaCas), modified starch and lactose	Nozzle diameter: 0.5 mm Inlet temperature: 165°C Outlet temperature: 80°C Feed flow rate: 360-540 mL/h Pressure: 5 bar	33.43-52.98 Calvo <i>et al.</i> (2010)
Olive oil	Soy protein isolate (SPI), pea protein isolate (PPI), defatted milk powder, octenylsuccinic anhydride-modified starch (OSA) and maltodextrin (MD)	Nozzle diameter: 1.5 mm Inlet temperature: 160°C Outlet temperature: 85°C Feed flow rate: 6 mL/min	77-88 Zhao and Tang (2016)
Rice bran oil	Topioca starch and soya protein isolate (SPI)	Inlet temperature: 140, 150 & 160°C Outlet temperature: 68, 76 & 84°C Feed flow rate: 2.5 mL/min Pressure: 2.5 kg/cm ²	53.81-78.12 Murali <i>et al.</i> (2016)
Rice bran oil	Jackfruit seed starch and whey protein isolate (WPI)	Inlet temperature: 140, 150 & 160°C Outlet temperature: 68, 76 & 84°C Feed flow rate: 2.5 mL/min Pressure: 2.5 kg/cm ²	52.15-87.50 Murali <i>et al.</i> (2017)
Rice bran oil	Maltodextrin (MD), gum Arabic (GA) and whey protein concentrate (WPC)	Nozzle diameter: 0.5 mm Inlet temperature: 180°C Outlet temperature: 90°C Feed flow rate: 1 L/h	38.5-78 Atta <i>et al.</i> (2020)
Rice bran oil	Pea protein isolate (PPI) and maltodextrin (MD)	Nozzle diameter: 1.5 mm Inlet temperature: 155°C Outlet temperature: 92-96°C Feed flow rate: 3 g/min	74 Benito-roman <i>et al.</i> (2020)
Sesame oil	Tamarind seed mucilage	Inlet temperature: 135°C Outlet temperature: 80°C Feed flow rate: 40 mL/min Pressure: 4 bar	91-81 Alpizar-Reyes <i>et al.</i> (2020)
Soyabean oil	Soy protein isolate (SPI)	Nozzle diameter: 0.5 mm Inlet temperature: 160-200°C Outlet temperature: 80°C	33.10-51.4 Tang and Li (2013)
Soyabean oil	Protein isolates	Nozzle diameter: 0.5 mm Inlet temperature: 180°C Outlet temperature: 80°C Feed flow rate: 6 mL/min	45.4-57.9 Liu <i>et al.</i> (2014)
Sunflower oil	Milk protein isolates (MPI) and dextrin	Inlet temperature: 160°C Outlet temperature: 95°C Feed flow rate: 1.6 L/h	70.2-96.6 Ahn <i>et al.</i> (2008a)
Sunflower oil	Milk protein isolates (MPI) and dextrin	Inlet temperature: 160°C Outlet temperature: 95°C Feed flow rate: 1.6 L/h	45.02-92.10 Ahn <i>et al.</i> (2008b)
Sunflower oil	Maltodextrin (MD)	Inlet temperature: 150-200°C Outlet temperature: 79-120°C Feed flow rate: 9 kg/h	52-87 Lewandowski <i>et al.</i> (2012)
Sunflower oil	Trehalose and whey Protein isolate (WPI) or sodium caseinate (NaCas)	Inlet temperature: 150°C Outlet temperature: 60°C	95.6-99 Domian <i>et al.</i> (2014)
Sunflower oil	Maltodextrin (MD) and hydroxypropyl methylcellulose	Nozzle diameter: 0.5 mm Pressure: 6 bar	73.13-87.00 Roccia <i>et al.</i> (2014)

Freeze drying. A less complicated and simple process for the encapsulation of heat-sensitive material is freeze drying. Encapsulation based on freeze drying involves the generation of an emulsion by the target compound and later, encapsulating material to convert them into capsules by applying the freeze drying technique (Carpena *et al.*, 2021). The emulsion was frozen at -90 to -40° and sublimed to the gaseous state from the solid by creating vacuum pressure (Pattnaik and Mishra 2021). Sublimation of water takes place at below the

triple point (<4.58 mm Hg pressure and < 0.0098° temperature). Freeze drying is the batch process results with higher retention of nutrition than spray drying. It is an expensive process that restricts application up to the high-value products which have specific biological and phytochemical properties. However, modifications in the freeze drying technique are applied by new technical solutions and transformed into time and cost effective process. The encapsulation efficiency obtained

by various researchers with variable process parameters of freeze drying technique is given in Table 2.

Cold- pressed pumpkin seed oil was encapsulated in whey protein isolate, maltodextrin and gum Arabic by Ozbek and Ergoniil (2020). The emulsion prepared was freeze dried at -80° and pellets were ground into powder. Flowability was evented good from the Hausner ratio (HR) and Carr's index (CI) values. Encapsulation efficiency were ranged from 62.41 to 71.04%. Morphological structure showed that the freeze dried capsules were irregular in shape, porous and cracked because of the grinding process. It is reported that due to porous structure, surface oil was observed higher. Sponge-like and porous structure was the disadvantage as it promotes the diffusion of air. Two compositions (12.28% WPC + 5% maltodextrin + 2.70% gum Arabic or 10% WPC + 5% maltodextrin + 5% gum Arabic) showed increase in encapsulation efficiency, solubility, total polyunsaturated fatty acids (PUFA) content and decrease in wettability time and total saturated fatty acids (SFA) content.

Perrechil *et al.* (2021) studied functional formulations composed of flaxseed oil, rice protein concentrate (RPC) and modified starch prepared with freeze drying (Table 2). Prepared emulsion was freeze dried at -30° at 0.5 mm of Hg for 72 h in aluminum recipients of bench-type freeze drier. Increased in modified starch content resulted with porous structure and higher encapsulation efficiency. Lower moisture content and hygroscopicity was observed with combination of RPC and modified starch. Product showed higher PUFAs and essential amino acids. Freeze drying for the encapsulation of sunflower oil was investigated by Holgado *et al.* (2019). The effect of changes in the oil droplet size during homogenization at pressure of 15 or 70 MPa in the emulsification step was studied by free and encapsulated oil fractions of freeze-dried samples. The samples were frozen at -32° for 24 h and freeze dried for 48 h and ground. Surface oil content was reduced, explained the increase in stability of the free oil.

Table 2: Freeze drying technique for encapsulation of vegetable oils.

Oil	Wall materials	Process parameters	Encapsulation efficiency (%)
Flaxseed oil	Chickpea or lentil protein isolates (CPI or LPI) And maltodextrin (MD)	Temperature: -40°C Pressure: 0.120 mbar	46.2-92.1 Karaca <i>et al.</i> (2013b)
Flaxseed oil	Whey protein isolate (WPI) and maltodextrin (MD)	Temperature: -20°C Pressure: 40 Pa	27.01-95.44 Fioramonti <i>et al.</i> (2017)
Flaxseed oil	Modified starch and rice protein concentrate	Temperature: -30°C Pressure: 0.5mm Hg	0.69-90.58 Perrechil <i>et al.</i> (2021)
Olive oil	Maltodextrin, carboxymethylcellulose and butylhydroxytoluene	Temperature: -80°C	36.90 and 69.09 Calvo <i>et al.</i> (2012)
Sunflower oil	D-lactose monohydrate and sodium caseinate (NaCas)	Temperature: -50°C	72.7 – 74.0 Velasco <i>et al.</i> (2006)

Microwave drying. Waves produced by magnetron in the presence of both electric and magnetic field are electromagnetic radiation, called microwaves. Microwave drying of the heat sensitive products in the food industry is well-known technique due to shorter time of drying, lower temperature of product, higher efficiency and drying rate (Haghi and Amanifard 2008). The process is environment friendly without emission of volatile organic compounds (VOCs) and exhibits low energy consumption (Lin *et al.*, 2011). Heat produced from microwaves is called volumetric direct heating as heat is produced by rapid movement of polar molecules. The molecules are oriented in the direction of electric field and thereby movement of molecules occur inside the product. However, high frequency makes that movement rapid in term of million times per second because of the alignment and re-alignment of the polar molecules (Chandrasekaran *et al.*, 2013). The rapid heating builds vapour pressure gradient and moisture comes out at a rapid rate and drying is accelerated. Dielectric properties of encapsulants revealed the application of microwave to dry an

emulsion of encapsulated vegetable oils. Dielectric constant is the parameter which defines the encapsulation properties and encapsulation efficiency in microwave drying. Wall and core materials must have different dielectric constant (Abbasi and Rahimi 2008). Comparatively, higher dielectric constant and dissipation factor was recommended for wall material when compared to core material (Spohner, 2017). There was no such a report found on measurement of dielectric properties for encapsulating vegetable oils. Microwave heating solidifies only wall materials, forms a shell around emulsified oil droplets by evaporating solvent, generally water (Pattnaik and Mishra 2020). Microwave assisted encapsulation of oil emulsion is a relatively novel idea, which results in reduction of time and consequently improves the quality (Feng *et al.*, 2012). Vegetable oils are sensitive to temperature and heat, enables the use of microwave for encapsulation which restricts higher temperature of product. Moving out the moisture due to vapour gradient prevents the powder structure collapse and preserves nutrition's and quality of encapsulates (Vadivambal and Jayas 2007).

Encapsulation of vegetable oils using microwave drying is explored in limit as less number of literature was found. Hassan and Muhamad (2017) used microwave drying for encapsulation of perah seed oil at a 110 W for 10 min after lyophilisation process. Encapsulation efficiency was achieved maximum up to 42%. Pattnaik and Mishra (2020) studied the effect of microwave treatment on preparation on vegetable oil encapsulates. The moisture content of vegetable oil powder was decreased with increment in microwave power. Colour value was adversely affected at higher power due to non-enzymatic browning. Higher power affected the structure and resulted with protein denaturation. Lower encapsulation efficiency of encapsulation was reported as the oil was released on surface due to produced porous structure. The quality of core material is affected by microwave power (Padzil *et al.*, 2018) and microwave time (Ahmad *et al.*, 2020).

Coacervation. One of the simplest, low cost, reproducible, scalable and most effective methods for encapsulating hydrophobic substances is coacervation. It is the process of phase separation in which two liquid phases are separated under controlled conditions (Gouin, 2004). Change in parameters such as, pH, temperature, addition of ions and alteration in solubility cause the condition that favors coacervation process. The resulting phase, called coacervate, is separated from other liquid phase. The process induced in a system of single polymer is known as simple coacervation, while in complex coacervation process, the one is taking place between two or more polymers (Timilsena *et al.*, 2017). In the complex method, two opposite charged molecules are separated based on electrostatic interaction. Generally for the food application, interaction between protein and polysaccharide is used (Schmitt and Turgeon 2011). The stages of coacervation are: a) emulsification of core material in aqueous solution; b) formation of immiscible phase; c) deposition of liquid polymers

around the core material and d) stabilization of capsules (El Asbahani *et al.*, 2015). The process formulates protein/carbohydrate complex and coacervates that depends on concentration and charge density of biopolymers, ionic strength, molecular weights, protein to carbohydrate ratio, solution pH and temperature, agitation and pressure (Schmitt and Turgeon 2011). Coacervation method is superior in encapsulating the oil than others. Efficiency was dependent on electrostatic interaction between wall materials.

Some of the studies reported for encapsulation of vegetable oils using coacervation is presented in Table 3. Higher efficiency for chia seed oil encapsulation was reported by using chia seed protein isolate-chia seed gum complex coacervates than individuals as a wall material (Timilsena *et al.*, 2016). Wang *et al.* (2014) encapsulated the omega-3 rich tuna oil by complex coacervation. The yield was affected by pH and starch-protein ratio. Stronger driving force generated by oppositely charged molecules was responsible for change in interaction of carbohydrate and protein. Encapsulation efficiency and release rate were dependent on oil load of olive oil, amount of crosslinker and polymer concentration (Devi *et al.*, 2012). Higher concentration of polymer increase the size of encapsulates. Release of peppermint oil encapsulated with complex coacervation was lower for lesser core to wall ratio in cold water. Release of core oil occurs in two stages; first quick release and release at lower rate (Dong *et al.*, 2011). Higher efficiency (~90%) was achieved for encapsulation of cold-pressed sesame oil using complex coacervation between gelatin and gum Arabic as a wall material (Dai *et al.*, 2020). Katona *et al.* (2010) encapsulated sunflower oil in ternary mixture of hydroxypropylmethyl cellulose (0.7%), sodium carboxymethyl cellulose and sodium dodecyl sulfate. Deposition and stability of the shell was based on concentration of dodecyl sulfate in emulsion.

Table 3: Process parameters and other techniques for encapsulation of vegetable oils.

Technique	Oil	Wall materials	Process parameters	Encapsulation efficiency (%)
Complex coacervation	Flaxseed oil	Gelatin and gum Arabic (GA)	Homogenization: 3000-15000 rpm	84 Liu <i>et al.</i> (2010)
Complex coacervation	Flaxseed oil	Flaxseed protein isolate (FPI) and flaxseed gum (FG)	Homogenization: 15000 rpm Time: 4 min Microfluidization: 12000 psi (3 passes)	87.8-95.4 Pham <i>et al.</i> (2020)
Complex coacervation	Olive oil	Gelatin and sodium alginate	Temperature: 60°C Time: 15 min	62.54-89.37 Devi <i>et al.</i> (2012)
Emulsification	Flaxseed oil	Gelatin, flaxseed mucilage (FM) and oxidized tannic acid (OTA)	Homogenization: 10000 rpm Time: 5 min	94.2-97.1 Mohseni and Goli (2019)
Emulsification	Wheat germ oil	Sodium alginate and starch	Homogenization: 1000 rpm Time: 10 min	88 Chan <i>et al.</i> (2000)
Emulsion gelation	Olive oil	Calcium alginate, tamarind gum (TG) and magnesium stearate	Homogenization: 5000 rpm Time: 15 min	78.70-50.70 Bera <i>et al.</i> (2015a)

		(MS)		
Emulsion gelation	Olive oil	Sodium alginate	Homogenization: 5000 rpm Time: 15 min	64.10-87.76 Bera <i>et al.</i> (2015b)
Emulsion gelation	Rapeseed oil	Pea protein isolate (PPI) and glucose syrup	Ultra-turrax: 21.500 rpm Time: 90 s	94.47-95.60 Tamm <i>et al.</i> (2016)
Emulsion gelation	Sunflower oil	Sodium alginate	Stirring: 6000 rpm Time: 40 min	81.92-95.39 Bera <i>et al.</i> (2009)
Extrusion	Sunflower oil	Alginate and shellac	Nozzle diameter: 0.3 mm Feed flow rate: 30-90 mL/h	63.2-98.7 Morales <i>et al.</i> (2017)
Supercritical carbon dioxide spray unit	Coconut oil	Maltodextrin (md) and sodium caseinate (NaCas)	CO ₂ flow rate: 150L/h Pressure: 12,14 and 16 MPa Temperature: 40,50 and 60°C Emulsion flow rate: 3-5 mL/min	72.7-80.2 Hee <i>et al.</i> (2017)
Vibrating nozzle extrusion or fluid bed	Flaxseed oil	Pectin	Nozzle diameter: 450-1000 µm Frequency: 180-350 Hz Pressure: 553-664 mbar Amplitude: 4-7 Electrode: 1500 V	95.27-97.97 Menin <i>et al.</i> (2018)

PGSS process. Particles from gas-saturated solutions (PGSS) is the point of interest as particles formed by supercritical carbon dioxide. Solubility of SC-CO₂ in fats, oils and biopolymers is the base of successful encapsulation using PGSS (Haq and Chun 2018). PGSS process is demonstrated as the suitable method for processing the aqueous solution. The process comprises of two major steps: carbon dioxide comes in contact with solute at high pressure, nearly 10-15 MPa and high temperature (around 373-393 K) in static mixer. The biphasic mixture is expanded in spray tower. The atomization is improved by the expansion that release the dissolved gas into liquid phase. The temperature is reduced extremely fast due to Joule-Thomson effect that forms the small size particles, usually in micron size and distributes the size in narrow range (Martin *et al.*, 2010). The PGSS drying allows to dry aqueous solutions into powders that avoids thermal degradation of the product because of shorter residence time in the static mixer that is the only part of higher temperature (Meterc *et al.*, 2008). It is the vital process for solvent free final products in the food and pharmaceutical sector. Moreover, efficient morphology of the particle in terms of sphericity, solidity, distortion and agglomeration can be achieved by PGSS process. The variation in density and viscosity of SC-CO₂ alters due to pressure and temperature that affects the encapsulation yield. Also, nozzle size influences the encapsulation process (Haq and Chun 2018).

Klettenhammer *et al.* (2022) encapsulated linseed oil enriched with carrot pomace extracts using PGSS process and found best efficiency, powder density and flowability at 10 MPa pressure. Melgosa *et al.* (2019) compared PGSS, spray drying and freeze drying methods to encapsulate omega-3. Higher encapsulation efficiency (~98%) with spherical morphology was

obtained for PGSS. At lower temperature, PGSS dried particle were noticed for cold-crystallization which may correspond to transmit the heat to the oil offered by shell. Particle size analysis also showed the narrow span for PGSS process. PGSS process have a potential to encapsulate the oil for the production of an enhanced ingredient to be further tested in food, cosmetic and pharmaceutical applications (Ndayishimiye *et al.*, 2019).

Other techniques. Several other techniques (Table 3) are used to encapsulate the oils in two different structures: beads and capsules. The beads of lipophilic drugs in sunflower oil was prepared using emulsification or internal gelation by Ribeiro *et al.* (1999). Beristain *et al.* (1996) encapsulated orange peel oil by cocrystallization method. Parameters such as hydrophile-lipophile balance, incorporation of emulsifier and dispersed phase morphology of sunflower oil beads obtained by extrusion was correlated (Yilmaz *et al.*, 2001). Aliabadi *et al.* (2007) optimized the co-solvent evaporation process for the encapsulation of hydrophobic drugs in polymeric micelles. Castor oil was encapsulated in agar by emulsification (Miyazawa *et al.*, 2000). Palm oil was encapsulated by ion gelation method to produce beads of oil in Ca-alginate with high oil loading (Chan, 2011). Docosahexaenoic acid (DHA) was encapsulated in dodecyl succinic anhydride-esterified agarose (DSAG) by spray-chilling process, similar to spray drying with a difference of removal of energy instead of application of heat energy (Xiao *et al.*, 2020). The existing methods provide potential efficiency to encapsulate vegetable oils individually or in combination of other compounds to achieve the desired properties and widen the applications of bioactive compounds.

EFFECT OF ENCAPSULATION ON OXIDATIVE STABILITY OF VEGETABLE OILS

Vegetable oils have higher unsaturated fatty acids, especially PUFA (polyunsaturated fatty acids), which are susceptible to lipid oxidation. In addition to PUFA, compounds such as, vitamin E, carotenoids, flavonoids and quinones are easily degraded or unstable when exposed to environmental factors. The major factors responsible for degradation of oils are oxygen, light and heat. Oxidation of fatty acids results in unwanted organoleptic properties and degradation of nutritional quality of the product. Lipid oxidation has an adverse effect on health as it produces free radicals. Major primary oxidation product, peroxides have higher reactive ability and break down to free radicals that promote oxidation reactions. They also propagate auto-oxidation, which is responsible for ketones and aldehydes production; secondary lipid oxidation products. Oxidation is described in three phase: 1) induction 2) propagation and 3) terminal phase. Encapsulation is the process to protect the vegetable oils from oxidation. Process parameters which affect the stability of the encapsulated vegetable oils are further reviewed in this section.

Durum wheat (*Triticum durum* Desf.) germ oil was encapsulated in sodium alginate which improved its storage stability over the storage of 30 days at 4°C temperature (Durante *et al.*, 2012). High oleic sunflower oil (MEHS) was microencapsulated in dextrin with milk protein isolate (MPI), soy lecithin and sodium triphosphate emulsifier as a supplements (Ahn *et al.*, 2008). The higher peroxide value was observed at lower encapsulation efficiency after 30 days of storage for MEHS. Karaca *et al.* (2013a) studied the oxidative stability of free and encapsulated flaxseed oil. The peroxide value (PV) was observed 5.73 and 6.31 to 6.80 meq of O₂/kg before encapsulation and after encapsulation, respectively. The increase in PV was due to contact with oxygen during emulsification and spray drying. The peroxide value for maltodextrin-chickpea protein isolate (CPI) encapsulated flaxseed oil (7.31 meq of O₂/kg) and maltodextrin-lentil protein isolate (LPI) encapsulated flaxseed oil (6.86 meq of O₂/kg) was significantly lower as compared to free flaxseed oil (12.91 meq of O₂/kg) after 25 days storage at room temperature. Sunflower oil encapsulated in trehalose and whey protein isolate/sodium caseinate showed higher oxidative stability than non-encapsulated oil (Domian *et al.*, 2014). The peroxide value of free flaxseed oil was increased at higher rate as compared to encapsulated flaxseed oil after six month of storage (Goyal *et al.*, 2015). Oxidative stability of encapsulated coffee oil was investigated and reported that higher temperature showed higher peroxide value of un-encapsulated oil as compared to encapsulated coffee oil (Getachew and Chun 2016). The oxidative stability of pea protein isolate (PPI)-pectin based spray dried encapsulates of PUFA rich oil was observed higher as compared to absence of pectin in wall material (Aberkane *et al.*, 2014).

Icyer *et al.* (2016) observed that the peroxide value was slightly increased after encapsulation of fig oil (7.36 meq O₂/kg oil) in gun Arabic and maltodextrin as compared to liquid fig oil (7.17 meq O₂/kg oil). The oxidative deterioration of flaxseed oil powder was observed higher as compared to sunflower oil powder, due to fatty acid composition (Shivakumar *et al.*, 2012). The peroxide value was increased for both the powders at ambient temperature as well as 38°C up to 3 weeks of storage. Fresh rapeseed oil was not detected with hydrogen peroxide, while encapsulation of the rapeseed oil in pea protein isolate (PPI) and pea protein hydrolysates (PPH) showed the increase in hydrogen peroxide, 2.6 mmol/kg oil due to emulsification, atomization and drying. The storage at 20°C and 33% RH revealed that the increase in the hydrogen peroxide content was observed higher in PPI based encapsulates as compared to PPH encapsulated rapeseed oil (Tamm *et al.*, 2016). After accelerated condition of storage (45°C for 8 weeks), the peroxide value of docosahexaenoic acid (DHA)-rich microencapsulates remained one ninth as compared to unencapsulated oil which revealed the promising oxidative stability of DHA-rich microencapsulated oil (Chen *et al.*, 2016). The peroxide value (PV) of flaxseed oil was 0.16 meq/kg, which increased to 0.34 meq/kg and 1.10 meq/kg for storage of 60 days at 4°C and 25°C temperature, respectively. Whereas, the PV of encapsulated flaxseed oil was observed 0.25-0.30 meq/kg and 0.37-0.62 meq/kg for 60 days storage at 4°C and 25°C temperature, respectively.

The powder form of encapsulated flaxseed oil showed higher stability against oxidative deterioration of oil. The storage temperature also affected the oxidative stability of flaxseed oil encapsulates. Higher temperature favours the development of peroxides in the oil (Shahid *et al.*, 2020). Flaxseed oil was encapsulated in flaxseed protein isolate (FPI) complexes with flaxseed polyphenol (FPP) or flaxseed gum by coacervation showed higher oxidative stability as compared to bulk oil storage of 4 weeks (Pham *et al.*, 2020). During the storage of encapsulated flaxseed oil, the peroxide value of encapsulates increased from 3.0 to 5.3 meq O₂/kg (1.7 time) while higher oxidation rate was recorded for free oil (2.4 times). However, higher value of peroxide (3.0 meq O₂/kg) after encapsulation of bulk flaxseed oil (1.8 meq O₂/kg) was observed by Mohseni and Goli (2019). Calvo *et al.* (2012) studied the storage of encapsulated olive oil in heating chambers at 30°C for 4 months. It was concluded that protein constituents in wall matrix enhanced the shelf life of olive oil. Oil quality was unalterable up to 9 to 11 months. The peroxide value of sesame seed oil before microencapsulation (5.09 meq/kg) was lower than spray dried microcapsules M1; Core: wall: 1:1 (5.13 meq/kg) and M2; Core: wall: 1:2 (5.19 meq/kg). For temperatures of 25, 35 and 40°C, free sesame oil showed higher peroxide as compared to encapsulated sesame oil over six weeks storage (Alpizar-Reyes *et al.*, 2020).

Though, the peroxide value of encapsulated oils was higher after encapsulation due to exposure to the oxygen and/or atmosphere during processing and handling, the stability of encapsulated vegetable oils against oxidative deterioration was higher during storage as compared to unencapsulated bulk oil. Storage conditions also affect the formation of the peroxides in the oils. Higher temperature leads to higher rate of peroxide formation. The matrix of wall affected in terms of porosity. Higher the porous structure, more the oxygen get chance to enter the capsules and deteriorate the quality of oils. Multi-compounds wall matrix resulted with significance in stability along with the different proportion of components. Thus, the higher oxidative stability of vegetable oils could be achieved by encapsulating the oils in appropriate wall matrix and at proper storage conditions.

APPLICATIONS OF ENCAPSULATED VEGETABLE OIL POWDER

An awareness regarding benefits of having functional food in diet is increasing as same as the recent century goes ahead. The foods containing bioactive compounds have a specified function that add the nutritive value to the food which facilitates health benefits. The foods have positive effect on health beyond their native food nutrition. Vegetable oils are one such compound that alters the functional properties. The use of pure form of vegetable oils in foods have several limitations such as, handling problems, sensitive to temperature and oxygen, uncontrolled release, lower solubility, undesirable sensory attributes, poor bio-accessibility and bio availability (Rodrigues *et al.*, 2016; Vinod *et al.*, 2021; Aruna *et al.*, 2022). In order to retain its quality up to the desired destination, the process now-a-days used to convert it in the different forms with the help of encapsulation. Encapsulation provides a carrier for vegetable oils to match with other food formulations. The use of encapsulated vegetable oil overcome the limitation of pure form application into the foods. In addition, vegetable oil encapsulates provides an alternative to non-vegetarian food as a fat replacer (Delshadi *et al.*, 2020).

Atta *et al.* (2020) incorporated encapsulated rice bran oil (2, 4 and 6%) and rice bran oil (2%) into yogurt. Increase in titratable acidity and decrease in the pH value of the yogurt were observed during storage at 5°C up to 14 days due to metabolic activity of yogurt starters. The water holding capacity was also observed increased which favors the quality of yogurt. The organoleptic properties of fortified yogurt was well retained. The soup powder was enriched with omega-3 rich linseed oil encapsulates by Rubilar *et al.* (2012). The enriched soup reported with similar energy and carbohydrates and increased protein (24-36% higher) and fat (25% higher) compared to the available soup. Gallardo *et al.* (2013) fortified bread with the encapsulated linseed oil. The bread was similar in appearance to control bread. Fortification may lower the shelf life due to oxidation of sensitive compound

present in bread. The Labneh cheese was prepared with replacement of milk fat by free and encapsulated wheat germ oil (Soliman *et al.*, 2019). Comparable quality and composition with the control was achieved in Labneh incorporated with wheat germ oil encapsulates. Free oil imparted yellow colour and slight oily taste to the Labneh, when encapsulation masked the colour and taste of oil and resulted comparable characteristics with control.

Omega-3 rich chia oil was encapsulated in chitosan and incorporated in butter at the concentrations of 2, 4, 6 and 8% (Ullah *et al.*, 2020). The stored samples (90 days at -10°C) was observed with higher retention of colour, better flavour and texture as compared to the control (without encapsulates). Omega-3 fatty acids concentrations of butter can be increased up to 8% with the chia oil microcapsules with reasonable oxidative stability and almost no effect on the sensory parameters of butter. Ice-cream was fortified at 3, 4 and 5% of encapsulated flaxseed oil (Gowda *et al.*, 2018). Around 45% of recommended daily allowance (1.4g of alpha-linolenic acid/day) was served by the freshly prepared functional ice cream prepared with flaxseed oil encapsulates. The ice cream can be fortified up to the 4% level of encapsulated flaxseed oil. Bread was enriched with encapsulated flaxseed oil and reported that the rheological properties, firmness and density was increased and lightness was decreased as compared to control (Beikzadeh *et al.*, 2020). Encapsulates containing bread showed higher alpha-linoleic acid content and lower peroxide index as compared to oil containing bread. Sensory properties were also well preserved.

Mayonnaise was formulated with incorporation of microencapsulated chia seeds, pumpkin seeds or baru oils by Rojas *et al.* (2019). Viscosity of the Mayonnaise was increased with chia and pumpkin seed oils encapsulates. Texture was not affected with incorporation. Sensory analysis revealed that Mayonnaise can be successfully produced by enrichment up to 5% (w/w) the microencapsulated oils. Sensory characteristics of milk was not significantly changed after incorporation of microencapsulated flaxseed oil powder (Goyal *et al.*, 2015). Rutz *et al.* (2017) incorporated carotenoid rich palm oil encapsulates into yogurt and bread. Difference was observed in the carotenoid release behavior for before and after application in the food systems. The release was lower after application in food and the carotenoids did not degrade after release in food. The delayed nutrient release was demonstrated in yogurt fortified with soyabean oil microencapsulated in soy protein isolate (SPI) and zein complex (Chen *et al.*, 2010). The olive oil and lemon juice encapsulated with the help of freeze drying was successfully used as instant salad sauce (Silva *et al.*, 2013).

Dahi (Indian yogurt) fortified with 2% level of microencapsulated flaxseed oil powder was observed comparable to control for permissible limit of peroxide value with serving potential of omega-3 fatty acids

(Goyal *et al.*, 2016). Fat of the burger was successfully replaced with the help of chia and linseed oil encapsulates obtained by ionic gelation in order to prepare low-fat burger with a healthy ω -6/ ω -3 PUFA ratio without affecting the technological and sensory properties (Heck *et al.*, 2017). Thus, there is a wide range of food products, especially dairy and bakery products could be enriched with encapsulated vegetable oils. Enriched foods carries higher PUFAs as compared to base nutrition with quality oils. Encapsulation of vegetable oils helps in serving recommended daily allowances (RDA) of essential fatty acids in human diet. Further, encapsulated vegetable oils have a potential to replace the fat in food industries.

CONCLUSIONS

Higher demand of vegetable oils as a source of PUFA was observed due to their health benefits and higher number of vegan and vegetarian diet. The susceptibility of PUFA rich vegetable oils to oxidative degradation has questioned the technologists for its preservation through encapsulation. The liquid form of oil can successfully be entrapped into a gel or powder matrix in various ways such as spray drying, freeze drying, microwave drying, coacervation, PGSS process and other innovative and novel techniques. Selection of wall materials which are majorly combination of polysaccharides and proteins is prominent step in the encapsulation process. Encapsulation of the PUFA rich vegetable oils would be a promising approach to increase the oxidative stability for their processing and storage. Further, the composition of fatty acids has not been affected significantly by encapsulation. The encapsulated oil particles have a wide applications especially in food industries like, dairy, bakery and meat where the replacement of fat or enrichment of beneficial fatty acids are the major concern. Encapsulates were acceptable by consumers for flavour and taste attributes on incorporation in food products. The vegetable oil encapsulates are biodegradable, pollution free, environment friendly and favor controlled release.

FUTURE SCOPE

Future research should be more focused on the advancement of existing techniques that promise higher efficiency of process and quality of final product. The gap in formation of encapsulated vegetable oils at large scale can be improved with advanced scientific works in research and development. Wall materials and technologies should be investigated for lower cost of application. Studies on biomaterial behaviour science, physical chemistry and biophysics regarding encapsulation of vegetable oils are needed to understand the interaction of oils in food, bioavailability of oil and nutritional impact for processing and consumption.

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Conflict of Interest. None

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