

# A Review on Encapsulation of Oils

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## Abstract

Heat, oxygen, moisture and light are the main causes of oxidation reactions in lipid containing foods. In particular, lipid oxidation is a major problem for unsaturated lipids. Until recent years, natural or synthetic antioxidants have been widely used in oils to retard oxidative deteriorations. Nowadays, encapsulation of oils like other sensitive materials such as vitamins, colorants, phenolic compounds or probiotic bacteria by various techniques have become increasingly popular as a promising preservation method. On the other hand, encapsulation improves handling properties of oils as well as protecting oils against oxidation. Spray drying is the most preferred encapsulation technique due to its lower operating costs and simplicity. Freeze-drying, coacervation and emulsification are the other well-known encapsulation methods. However, process parameters of these methods have extremely important effect on storage stability of encapsulated oils. As for encapsulated oils by drying, also the characteristics of powder products are greatly influenced by process variables. Many studies have been carried out to optimize process factors for encapsulation of oils with a maximum efficiency. In this review, common practices used for oil encapsulation and the oxidative stability of encapsulated oils are discussed in detail. Furthermore, effects of environmental conditions on storage stability of encapsulated oils during storage are also reviewed.

**Keywords**— Encapsulation, freeze drying, oil, oxidation, spray drying

## 1 Introduction

Encapsulation is the coating of sensitive solid, liquid or gas ingredients such as flavours, enzymes, microorganisms, vitamins, minerals, colorants and lipids, known as core material, with a protective layer which is called wall material [1,2]. The most common wall materials can be divided into three main groups:

### 1) Carbohydrates

- a. Plant based carbohydrates such as maltodextrin, starch, cellulose, gum arabic, mesquite gum, guar gum, galactomannans, cyclodextrin, pectin
- b. Marine based carbohydrates such as carrageenan and alginate
- c. Microbial or animal based carbohydrates

such as xanthan, gellan, dextran, chitosan

### 2) Proteins

- a. Plant based proteins such as soy protein, pea protein, barley protein, zein, gluten
- b. Animal based proteins such as casein, whey protein, gelatine

### 3) Lipids and waxes such as milk fat, phospholipid, beeswax and carnauba wax [3–5].

The selection of suitable wall materials depends on the properties of core material and final capsules [6].

An optimal wall material should have the following characteristics:

- a) Be food – grade
- b) Be cheap
- c) Have low viscosity at high solid contents
- d) Have good emulsifying properties

- e) Have good emulsion stability
- f) Have the ability of holding core material in its structure without any reactivity during processing or storage
- g) Controllable release of core material
- h) Protect core material from any environmental conditions, particularly oxygen, humidity and light [6–8].

The powder products obtained by encapsulation are called by different names according to size of particles. These are macrocapsules (> 5000 µm), microcapsules (1 – 5000 µm) and nanocapsules (< 1 µm) [9]. The purposes of encapsulation are as follows:

- a) Protection of core material from environmental factors (oxygen, temperature, light, moisture, pH etc.)
- b) Controlled release of core materials
- c) Masking of undesired odours
- d) Improvement of handling and flow properties of core materials [10].

In addition to these, the main aim of encapsulation of oils and lipids is to prevent oxidation and so to extend the shelf-life of these products [11]. Various techniques may be used for encapsulation. These are summarized in Table 1.

**Table 1.** Particle sizes of capsules produced by different encapsulation methods [12]

Encapsulation Technology	Particle size of capsules (µm)
Spray drying	10 – 400
Fluid bed coating	5 – 5000
Spray chilling / cooling	20 – 200
Melt injection	200 – 2000
Melt extrusion	300 – 5000
Emulsification	0.2 – 5000
Emulsions with multilayers	0.2 – 5000
Coacervation	10 – 800
Microspheres produced by extrusion or dropping	200 – 5000
Microspheres produced by emulsification	10 – 1000
Co – extrusion	150 – 8000
Inclusion complexation	0.001 – 0.01

Liposome entrapment	10 – 1000
Rapid expansion of super-critical fluids	10 – 400
Freeze or vacuum drying	20 – 5000

The selection of proper encapsulation method depends on certain parameters: mean particle size, physicochemical characteristics of core and wall materials, application of encapsulated material, release system of capsules, commercial production capacity and cost [13]. Meanwhile, nanoencapsulation methods are more complicated than the microencapsulation ones [14].

Oils are generally encapsulated to protect their beneficial compounds, to extend shelf – lives of them and to mask unacceptable odours. In this paper, various oil encapsulation techniques and numerous examples will be reviewed.

## 2 Spray drying

One of the oldest and most widely used food encapsulation technique is spray drying [15]. It is cost – effective, modifiable depending on encapsulation matrix, compatible with other processing equipment [6]. Moreover, heat – sensitive materials can be processed with negligible damages [16]. Due to these advantages, spray drying is commonly preferred for oil encapsulation. Nonetheless, one should keep in mind that high drying temperatures of spray dryer may trigger oxidation reactions in oils [17].

Formation of stable emulsion is the critical step for encapsulation by spray drying. Insufficient emulsification causes larger droplets and instability of emulsion and so lower encapsulation efficiency [18]. Smaller oil globules are better encased in wall matrix and attractive forces between the globules decrease. Hence, more stable emulsions are obtained [19,20]. Likewise, solid content has a direct effect on emulsion droplet size. In other words, when the total solid content of emulsion increases, the sizes of emulsion droplets decrease [21]. This impact may be related to increased emulsion viscosity or sufficient amount of wall materials solution to surround the droplets effectively [22]. However, high viscosity of emulsion is desired to a certain extent because it may lead to

blockage problems in spray dryer [23]. Processing and atomization of emulsions with viscosities higher than 200 mPa in spray dryer is extremely harder [24]. Gharsallaoui et al. [25] reported that the diameter of initial emulsion droplet should be between 1 – 100  $\mu\text{m}$ . Larger emulsion droplets may be broken during atomization step of spray drying and this causes high amounts of non – encapsulated surface oils [26]. According to Goula and Adamopoulos [27], as the droplet size of emulsion decreased, the encapsulation efficiency (EE) of pomegranate seed oil increased.

Emulsion droplet size is mainly influenced by emulsion homogenization pressure. Hogan et al. [28] concluded that the volume average diameter of droplets decreased from 0.93 to 0.38  $\mu\text{m}$ , when the homogenization pressure increased from 10 to 50 MPa. Similarly, Holgado et al. [29] obtained smaller and more uniform oil droplets with increased homogenization pressure. Furthermore, Takeungwongtrakul et al. [30] reported that the emulsions prepared with higher homogenizing pressure were more stable during 14 – day storage. In recent years, some homogenizers such as high – intensity ultrasound, microfluidizers or membrane homogenizers are used to produce emulsions having smaller droplets [31]. Silva et al. [32] reported that droplet mean diameter in emulsion ranged between 2.02 – 2.33  $\mu\text{m}$  for only Ultra – Turrax homogenization; while droplet mean diameter ranged between 1.05 – 1.51  $\mu\text{m}$  for high pressure homogenization after Ultra – Turrax. In addition, Tontul and Topuz [33] stated that they obtained flaxseed oil droplets with mean particle size that is one-tenth of the size of flaxseed oil droplets produced by Carneiro et al. [34]. The main reason of this dramatic difference is just emulsification process. Tontul and Topuz [33] preferred ultrasonic emulsification, whereas Carneiro et al. [34] used conventional emulsification. Emulsification method also influences encapsulation efficiency and size of particles in oil encapsulation by spray drying. According to Koç et al. [35] ultrasonic homogenization caused to lower EE of extra virgin olive oil and smaller particles than rotor – stator homogenizator.

On the other side, the composition of emulsion is another key player for emulsion stability. Therefore,

wall materials should have emulsifying properties. As mentioned before, carbohydrates, proteins or combination of these are generally used as wall materials for oil encapsulation. The major functions of carbohydrates in encapsulation are promotion of drying properties of wall matrix by increasing dry crust formation over the drying droplets [36]. Minemoto et al. [37] found that the emulsions prepared with gum arabic is more stable than the emulsions with maltodextrin. They clarified this situation by the chemical structures of these wall materials. Gum arabic acts as an emulsifier due to its proteinaceous matter [38]. However, maltodextrin, a digestion product of starch, is just composed of D – glucose units [39]. In addition, increase in dextrose equivalent (DE) of carbohydrates has protective effect against oxidation [36]. This is because EE increases, as DE values of carbohydrates increase. In a study carried out by Hogan et al. [40] EE of soy oil increased from 0% to 92%, when maize starch with DE 50 was used instead of maize starch with DE 0.

To improve emulsifying characteristics, generally, carbohydrates are used with proteins in oil encapsulation [41]. Nevertheless, the stability of emulsion stabilized by proteins is influenced by pH of emulsion. Specifically, the emulsifying capacity of proteins is minimum at their isoelectric point [42]. Additionally, in some cases, antagonistic effects may be observed between proteins and carbohydrates for EE. Tontul and Topuz [43] found that sodium caseinate and gum arabic combination had the lowest EE for flaxseed oil, whereas whey protein concentrate increased the EE of gum arabic. Similarly, Goyal et al. [44] reported that whey protein concentrate – lactose combination as wall material resulted in higher encapsulation efficiency of flaxseed oil than sodium caseinate – lactose combination. The authors explained this by the formation of more Maillard reaction products in whey protein concentrate mixtures as a result of higher lysine content of whey protein concentrate [44]. Protein – carbohydrate compounds occurred in Maillard reaction have better emulsifying characteristics than proteins [45].

In general, protection of oils against oxidation is closely related to EE. Higher efficiency leads to better protection. In addition to this, the type of wall mate-

rial is also important. Minemoto et al. [37] reported that oxidation of linoleic acid encapsulated with gum arabic was slower than encapsulated with maltodextrins. The authors stated that smaller droplet size of emulsions containing gum arabic caused a decrease in the rate of oxidation reactions [37]. Although its lower EE, flaxseed oil encapsulated with whey protein concentrate had lower peroxide values than those encapsulated with gum arabic [46]. A similar trend was also observed by Gallardo et al. [47]. This can be a result of antioxidant activity of whey proteins [48]. Nonetheless, it should be kept in mind that, when low molecular weight carbohydrates such as maltodextrins or saccharose are used in microencapsulation, they may cause caking, structural collapsing and recrystallization of amorphous carbohydrate matrix during storage [49]. Caking and recrystallization lead to leakage of core material from microcapsules [50]. Microcapsules should be stored at low temperatures (below  $T_g$  of powders) under controlled relative humidity (RH) conditions to prevent caking [51].

The size, shape and smoothness of spray – dried particles are affected by dry matter content of emulsion. Turchiuli et al. [52] found that average diameter of particles increased from 18 to 85  $\mu\text{m}$ , when total solid content of emulsion increased from 30% to 50%. Moreover, Sahin-Nadeem and Özen [53] determined that incorporation of whey proteins to carbohydrate based wall material mixture increased the smoothness of particles' surfaces for pomegranate seed oil encapsulation. On the contrary, Botrel et al. [54] found that partial substitution of whey protein isolate by inulin improved the viscoelastic characteristics of wall matrix and these particles, which contained fish oil as core material, displayed smoother surfaces. However, in general, microcapsules including polysaccharide based wall materials have remarkable surface dents, while whey protein based particles display smoother surfaces [55]. Microcapsules with less porosity have fewer spaces among particles that hinder oxidation reactions [56]. Nijdam and Langrish [57] proposed high temperatures of spray dryer cause melting of fat found within the walls and this melted fat moves through the pores. As a result, in general, fat/oil is concentrated close to the surface of powders. Another key element affect-

ing microcapsule's morphology is inlet temperature of spray dryer. According to Wang et al. [58] amorphous microcapsules were produced, when the inlet temperature of spray dryer was 180°C. On the contrary, spherical microcapsules with more homogeneous sizes were obtained, when the temperature decreased to 150°C. Spray drying of emulsions at lower temperatures than 150°C caused the insufficient drying of microparticles [58]. Eventually, adequately dried microcapsules having spherical outer surfaces without ruptures are critical for reduced permeability of gases and better protection of core material [59].

Water solubility index (WSI) is a significant criterion for dried powder products to enhance their integration to other foods. It has been proved that powders in crystalline state have lower interaction with water, while amorphous solids contain polar energetic sites having hydrogen bonding capacity with water molecules [60]. In a study dealing with encapsulation of annatto seed oil with whey protein isolate or modified starch by spray drying or freeze drying techniques, it was reported that all microparticles, independent from the type of wall material and drying method used, exhibited amorphous state [61]. Amorphous powders have higher solubility and are more hygroscopic [54]. This parameter is also affected by concentration of oil, in other words "oil load". As the amount of oil increases, the hydrophobicity of powder product increases. However, this leads to decrease of interaction with water and so, the solubility decreases [62]. On the other side, small and polar wall materials with hydrophilic properties such as inulin improve the solubility of spray dried oil capsules [63].

Moisture content and water activity ( $a_w$ ) are crucial factors for storage stability of powder products. The moisture contents of spray dried oil capsules are close to the maximum moisture range, 3 – 4%, for many powder food products [64]. Rocchia et al. [65] suggested that drying air inlet temperature, atomization air flow rate and pump setting were the main factors on the moisture content of spray dried powders. Furthermore, Laohasongkram et al. [66] reported that an increase in feeding rate caused an increase in moisture contents of spray dried Macadamia oil

powders. On the other hand, homogenization pressure also alters moisture content and  $a_w$  of encapsulated powders. Ixtaina et al. [67] determined that microcapsules with higher moisture content and  $a_w$  were obtained when homogenization pressure decreased from 600 bar to 400 bar. Lipid oxidation is usually minimum at  $a_w$  range of 0.20 – 0.40 for many food products [68]. Additionally, Carvalho et al. [69] concluded that spray – dried coffee oil microparticles having  $a_w$  below or equal to 0.20 exhibited lower oxidative stability.

Temperature and glass transition temperature ( $T_g$ ) are the other key elements, which may influence oxidation of encapsulated lipids during processing or storage [70]. For instance, oxidation of encapsulated coffee oil accelerated at 60°C compared to 40°C [21]. Correspondingly, Stapelfeldt et al. [71] observed a tenfold increase in oxidation products when storage temperature increased by 10°C. On the other side, Aghbashlo et al. [72] determined that wall materials with lower  $T_g$  increased encapsulation efficiency of spray dried fish oil capsules by means of stimulating formation of crust. To the best of our knowledge, improved encapsulation efficiency contributes to increased oxidative stability of encapsulated oils. Glassy state of microcapsules, which occurs during drying of food matrices containing protein or carbohydrates, partially preserve the microcapsule against lipid oxidation [73]. To maintain the glassy state of wall materials, spray drying should be carried out below  $T_g$  of wall materials [20]. Above  $T_g$ , amorphous materials start to crystallize and crystalline substances hold less water than its amorphous state. In a closed system, this leads to increase of  $a_w$  [74]. As a consequence, peroxide values of encapsulated oils within wall matrices containing lower  $T_g$  increase rapidly and agglomeration of microparticles occurs [75]. Also, RH influences the oxidation rate of spray dried oil powders [76]. Fang et al. [77] reported that linoleic acid encapsulated by spray drying oxidized rapidly at higher RH. Similarly, lipid oxidation of encapsulated fish oil increased at higher RH conditions due to crystallization of trehalose, which was used as wall material [49].

A great number of published works dealt with extensively oxidative changes in encapsulated oils during

processing and storage. It is believed that oxidation reactions begin in the homogenization step [78]. Due to breakdown of droplets and shear forces during homogenization, oxygen is easily distributed within emulsion and hence, the rate of oxidation increases [79]. Aghbashlo et al. [80] reported higher peroxide values in spray dried encapsulated fish oils as the drying air temperature, aspirator and peristaltic pump rates increased. Wang et al. [81] explained the reason of high peroxide values of encapsulated fish oil at the early stage of storage by rapid film formation of emulsions with high protein content. According to authors, probably the evaporation resistance was increased by film and this led to rapid temperature rise of particles during drying. Consequently, peroxides formed due to high temperature of particles [81]. In contrast to results obtained by Wang et al. [81], Martinez et al. [82] reported that the amount of lipid oxidation products in encapsulated walnut and chia oils did not increase just after spray drying. However, while encapsulation by spray drying protected the walnut oil from oxidative deterioration, it accelerated oxidative damage of encapsulated chia oil. The authors attributed this difference to different unsaturation degree of these oils [82]. Oxidation of encapsulated oil during storage may damage other components of oils such as carotenes and cholesterol as well [83,84].

Oxidation reactions occur in non-encapsulated (surface) oil [85]. Ahn et al. [86] reported peroxide value (PV) of spray – dried microencapsulated sunflower oil with lower EE (70.20%) was 15.20 meq/kg, whereas it was 8.70 meq/kg for microencapsulated sunflower oil with the highest EE (96.60%). PVs of oil capsules are negatively affected by any increase in aqueous phase of oil-in-water emulsion [87]. Oxidation reactions cause formation of compounds, which leads to off – flavour in foods. Kolanowski et al. [88] concluded that microencapsulation by spray drying stimulated undesirable sensory changes in fish oil with the existence of oxygen. Keogh and O’Kennedy [89] showed that sour off – flavour occurred earlier in spray – dried milk fat powders with higher surface fat (49.40%) compared to milk fat powders with lower surface fat. Same authors, in an another study, revealed that development of off-flavour in spray – dried fish oil microcapsules increased as homogeni-

zation pressure of emulsion increased [90]. To prevent sensory deteriorations in encapsulated oils due to oxidation, lipophilic antioxidants such as  $\alpha$ -tocopherol can be used effectively [91]. In addition to oxidation, surface oil may favour caking or agglomeration of microparticles [92].

In summary, spray drying is a cost effective oil encapsulation method. However, process conditions should be optimized to prevent undesirable changes, such as oxidation, in oil. Some antioxidants may be used for this purpose.

### 3 Freeze drying

Freeze drying, which is more commonly known as lyophilisation, is composed of three principal stages. Firstly, the product is frozen and then, ice is sublimed from the solid state under vacuum (sublimation step). Finally, unfrozen (bound) water is removed by evaporation under reduced pressure (desorption step) [93,94]. The advantages of freeze drying in terms of oil encapsulation can be outlined as follows:

- a) Decreased deterioration of heat – sensitive compounds (unsaturated fatty acids, tocopherols etc.)
- b) Controllable moisture of end products
- c) Easier reconstitution of freeze dried oil microcapsules [95].

Despite of these advantages, high operation costs, long processing time and open porous structure of the end product limit the application of this technology in food industry [96]. Furthermore, encapsulation efficiency by freeze drying is not as high as encapsulation by spray drying.

Freezing rate in an important parameter in freeze dried oil capsules. Heinzelmann et al. [97] reported that slow freezing rate (at  $-20^{\circ}\text{C}$ ) increased the shelf-life of fish oil microcapsules. Similarly, in an another study, Heinzelmann et al. [98] determined that slowly-frozen fish oil microcapsules had higher microencapsulation efficiency compared to fast-frozen samples. Lower cooling temperatures cause movement of dispersed oil droplets to the continuous phase and so, these oil droplets aggregates [99]. Encapsulation

efficiency is negatively affected by emulsion breakdown.

Encapsulation efficiency, which influences the oxidative stability of encapsulated oils, partially depends on wall matrix components. For instance,  $\gamma$ -cyclodextrin was found to be more effective compared to  $\beta$ -cyclodextrin in terms of improving stability of emulsion and so, encapsulation efficiency [100]. Differently from this, Koç et al. [101] stated that, although its lower encapsulation efficiency, freeze dried fish oil capsules containing pullulan had better oxidative stability than lactose containing capsules due to pullulan's capability of forming strong films with minimum oxygen permeability. Silva and Meireles [102] reported that encapsulation efficiency of annatto seed oil decreased as the degree of polymerization of inulin, the wall material, decreased. Similarly, Zhang et al. [103] determined that encapsulation efficiency of freeze dried fish oil decreased when degree of hydrolysis of soy protein isolate – maltodextrin conjugates increased. This may be due to insufficient strength of wall matrix with shorter chain length to cover the oil completely. Parallel results were obtained by Karaca et al. [104], who reported lower EE in freeze dried flaxseed oil encapsulated with maltodextrin with higher DE than maltodextrin with lower DE. On the contrary to these studies, Turasan et al. [105] showed that rosemary essential oil capsules containing maltodextrin with low DE (4 – 7) as wall material had higher storage stability due to its higher  $T_g$  compared to capsules with higher DE (13 – 17) maltodextrin.  $T_g$  of food matrices can be increased by addition of compounds having high molecular weight [106]. This might explain why protein based wall materials protect the oil capsules from oxidation better than carbohydrate based wall materials although they have lower encapsulation efficiency [107,108].

As in the case of spray drying,  $T_g$  is an influential aspect for freeze dried products. Imamura et al. [109] declared that a great majority of linoleic acid methyl ester were lost in the early step of freeze drying, in which sugar units were entirely hydrated in the amorphous phase between ice crystals. After drying, controlling of humidity and temperature of storage environment is extremely significant for mainte-

nance of quality of freeze dried products. In dried product, collapsing appears in case of change in moisture content or increase of temperature due to transformation of glassy amorphous state of encapsulated oil into rubbery state at  $T_g$  [110]. Occurrence of collapse may trigger the oxidation by increasing the exposure area of oil to oxygen [111]. Owing to extremely low drying temperatures used during freeze drying, the temperature of dried product never surpasses its  $T_g$  and hence, oil capsules keep their glassy state throughout drying and storage [112]. Freeze dried microparticles are generally have porous and irregular structures. On the other hand, porosity of matrix contributes to oxygen diffusion through microcapsules, and this may affect the storage life of product [61]. The holes observed via scanning electron microscope in the lyophilized products were associated with water that was removed during lyophilisation [113].

Oxidation stability of encapsulated oils is also altered by RH. Oxidation of surface oil decelerated when RH increased from 0% to 32% [114]. They attributed this result to bonding of hydrogen, a fraction of hydroperoxides that are generated in propagation phase of oxidation, by moisture. So, further oxidation reactions, in which hydroperoxides are used as substrates, are prevented. However, Minemoto et al. [115] explained this situation by hydrophobic character of oxygen. Under intermediate RH conditions, water is adsorbed by two or more layers of wall material and the diffusion of hydrophobic oxygen through these layers becomes difficult. On the other side, Velasco et al. [116] suggested that surface oil oxidizes likewise bulk oil (continuous phase), while encapsulated oil follows a pattern similar to mixtures having different oxidation levels. This variation is due to different oxidation degrees of oil droplets encased in a wall matrix [117].

Antioxidants can be added to emulsions to retard lipid oxidation. Velasco et al. [118] reported that polar antioxidants, in other words hydrophilic antioxidants, were less effective in oil-in-water emulsion system than lipophilic antioxidants. Since polar antioxidants are particularly situated in the aqueous phase of emulsions, they cannot preserve oil phase from oxidation. Similar to this manner, lipophilic

radicals boosted the oxidation of freeze dried encapsulated rapeseed oil, whereas hydrophilic radicals were found to be ineffective [119].

In conclusion, in addition to studies mentioned above, Avramenko et al. [120], Dzondo-Gadet et al. [121] and Minemoto et al. [122] also suggested the protective effect of freeze drying encapsulation process on lipid oxidation.

#### 4 Coacervation

The phase separation of the liquid phase of an aqueous solution of hydrocolloids under controlled conditions (adjusting by changing pH or temperature of solution and/or adding micro ions) and deposition of the resulting phase, known as coacervate, on the surface of core material is called "coacervation" [123]. The liquid – liquid phase separation is named as "coacervation", whereas solid – liquid phase separation is named as "precipitation" [124]. The fundamental stages of coacervation are as follows: 1) core material is emulsified within an aqueous solution consisted of two polymers at above gelling temperature of protein and above isoelectric pH of protein; 2) immiscible phases are formed; 3) liquid polymer(s) is/are deposited around the core material; 4) microcapsules are stabilized via cross – linking agents various drying processes such as spray drying or freeze drying [125,126].

There exist two types of coacervation: simple coacervation that includes only one polymer and complex coacervation, which consists of two immiscible liquid phases with oppositely charged ions. Complex coacervates are generally preferred for encapsulation of bioactive food components [12,127]. Principally, a protein fragment such as gelatine or whey protein and a carbohydrate fragment such as cellulose, gum arabic, chitosan or carrageenan are used as biopolymers in coacervation technique [128]. The formation of protein/carbohydrate complexes and coacervates depends on many factors: ionic strength, pH and temperature of solution; molecular weights, total concentration and charge density of biopolymers; ratio of protein to carbohydrate; pressure and stirring [129,130]. Mononuclear or multinuclear capsules can be produced by coacervation. In mononuclear capsule, core material is enclosed by coacervates;

while multinuclear capsule involves emulsified two separate cores [131]. Mononuclear capsule is a reservoir type system and delivers core material entirely even when only a part of wall material is damaged. Contrastingly, multinuclear capsule has a matrix type system and delivers the core material slowly even when wall material is fully damaged [132,133]. Due to these reasons, formation of multinuclear capsules is desired to extend the shelf – life of sensitive food components. The superiority of coacervation over the other oil encapsulation techniques is to produce powders with less surface oil even at higher oil (core material) loading levels [134].

Studies in the literature revealed that concentration and composition of wall materials, stirring speed and pH should be optimized to obtain spherical multinuclear capsules [135–137]. However, undoubtedly, pH is the most important factor in coacervation method. Suitable pH range depends on the types and the charges of biopolymers. While Weinbreck et al. [138] determined that a smooth whey protein – gum arabic coacervate layer formed at pH 4, pH 5 was selected as the best operation point for chitosan – gelatine (B) coacervates [139]. Secondly, stirring rate and homogenization speed play an important role. Aziz et al. [140] reported that a reduction in dimensions of microcapsules and an increase in the ratio of mononuclear microcapsules occurred, when the stirring speed increased. The main reason of increase in the amount of mononuclear capsules is the inadequate contact time between oil droplets due to high turbulence level caused by higher stirring rate [141]. If there is enough contact time, oil droplets can approach each other and two or more oil droplets may be combined in one capsule (multinuclear type). All of these parameters should be adjusted for coacervates with required characteristics.

Microencapsulation by coacervation, like the other techniques, contributes to the protection of oils against undesirable changes during storage. Throughout a 40–day storage in cold water, only 7% of peppermint oil was delivered from coacervate microcapsules, which consisted of gelatine and gum arabic [142]. On the other hand, Wang et al. [143] obtained freeze dried tuna oil capsules with extremely high EE of 98.56 % by applying gelatine – sodium

hexametaphosphate coacervates. These tuna oil microcapsules displayed more than 2–fold oxidation stability compared to bulk oil. Similar results were received by Siow and Ong [144], Martins [145] and Eratte et al. [146].

To the best of our knowledge, up to today, lemon and orange oil [138]; fish oil [147–149]; peppermint oil [150,151]; thyme oil [152]; sunflower oil [153]; cinnamon oil [154]; coriander oil [155]; lavender oil [156]; coffee oil [157]; flaxseed oil [158], olive oil [159]; palm oil [160]; citronella oil [139] and kiwi fruit seed oil [161] were successfully microencapsulated by coacervation technique. However, it should be noted that the composition of every oil substantially varies and this difference majorly affects the efficiency of coacervation method [162,163].

## 5 Other Methods

In addition to common microencapsulation techniques explained above, there are limited oil encapsulation studies including single layer emulsions [164,165]; multilayer emulsions [166,167]; molecular inclusion [168–170]; use of particles from gas-saturated solution (PGSS) process [171]; use of macroscopic beads [172] and miniemulsification–solvent evaporation technique [173]. In particular, new and expensive methods are evaluated to preserve volatile compounds of essential oils.

## 6 Conclusion

Various encapsulation techniques, mainly spray drying, freeze drying and coacervation, have been successfully utilized to preserve oils from oxidative deterioration. Mostly, the priority of encapsulation studies is to increase EE of oils. However, EE is affected by a combination of numerous process factors. These factors include equipment parameters, characteristics of wall matrix and oils' properties. The knowledge about impacts of all of these variables on encapsulation of oils has been increased in recent years. Nonetheless, the adaptation of these laboratory based results into industrial scale production of oil capsules has not been made. Further studies should concentrate on optimization of industrial scale encapsulation processes. Moreover, novel wall materials and encapsulation techniques should be investi-



gated to obtain oil capsules with higher EE and lower production costs.

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