

# Microencapsulation of Flaxseed Oil—State of Art

## Authors:

Asma Yakdhane, Sabrine Labidi, Donia Chaabane, Anita Tolnay, Arijit Nath, András Koris, Gyula Vatai

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## Abstract:

Microencapsulation is a well-known technology for the lipid delivery system. It prevents the oxidation of fatty acids and maintains the quality of lipid after extraction from oil seed and processing. In flaxseed oil, the amount of  $\omega$ -3 and  $\omega$ -6 polyunsaturated fatty acids are 39.90% and 60.42% and 12.25% and 17.44%, respectively. A comprehensive review article on the microencapsulation of flaxseed oil has not been published yet. Realizing the great advantages of flaxseed oil, information about different technologies related to the microencapsulation of flaxseed oil and their characteristics are discussed in a comprehensive way, in this review article. To prepare the microcapsule of flaxseed oil, an emulsion of oil-water is performed along with a wall material (matrix), followed by drying with a spray-dryer or freeze-dryer. Different matrices, such as plant and animal-based proteins, maltodextrin, gum Arabic, and modified starch are used for the encapsulation of flaxseed oil. In some cases, emulsifiers, such as Tween 80 and soya lecithin are used to prepare flaxseed oil microcapsules. Physico-chemical and bio-chemical characteristics of flaxseed oil microcapsules depend on process parameters, ratio of oil and matrix, and characteristics of the matrix. As an example, the size of the microcapsule, prepared with spray-drying and freeze-drying ranges between 10 $\mu$ m and 20 $\mu$ m, respectively. It may be considered that the comprehensive information on the encapsulation of flaxseed oil will boost the development of functional foods and biopharmaceuticals.

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Review

# Microencapsulation of Flaxseed Oil—State of Art

Asma Yakdhane <sup>1,\*</sup>, Sabine Labidi <sup>2</sup>, Donia Chaabane <sup>1</sup>, Anita Tolnay <sup>3</sup>, Arijit Nath <sup>1</sup>, András Koris <sup>1</sup>  
and Gyula Vatai <sup>1,\*</sup>

<sup>1</sup> Department of Food Engineering, Faculty of Food Science, Szent István University, Ménési st 44, HU-1118 Budapest, Hungary; Chaabane.Donia@hallgato.uni-szie.hu (D.C.); Arijit.Nath@etk.szie.hu (A.N.); Koris.Andras@etk.szie.hu (A.K.)

<sup>2</sup> Department of Food Microbiology and Biotechnology, Faculty of Food Science, Szent István University, Budapest, Somlói st 14–16, HU-1118 Budapest, Hungary; Labidi\_sabrina@yahoo.fr

<sup>3</sup> Institute of Supply Chain Management, Marketing and Tourism, Faculty of Economics and Social Sciences, Szent István University, Páter Károly St. 1, H-2100 Gödöllő, Hungary; tolnay.anita@labex.hu

\* Correspondence: Yakdhane.Aasma@hallgato.uni-szie.hu (A.Y.); Vatai.Gyula@etk.szie.hu (G.V.); Tel.: +36-1-305-7110 (A.Y.); +36-1-305-7115 (G.V.)

**Abstract:** Microencapsulation is a well-known technology for the lipid delivery system. It prevents the oxidation of fatty acids and maintains the quality of lipid after extraction from oil seed and processing. In flaxseed oil, the amount of  $\omega$ -3 and  $\omega$ -6 polyunsaturated fatty acids are 39.90–60.42% and 12.25–17.44%, respectively. A comprehensive review article on the microencapsulation of flaxseed oil has not been published yet. Realizing the great advantages of flaxseed oil, information about different technologies related to the microencapsulation of flaxseed oil and their characteristics are discussed in a comprehensive way, in this review article. To prepare the microcapsule of flaxseed oil, an emulsion of oil-water is performed along with a wall material (matrix), followed by drying with a spray-dryer or freeze-dryer. Different matrices, such as plant and animal-based proteins, maltodextrin, gum Arabic, and modified starch are used for the encapsulation of flaxseed oil. In some cases, emulsifiers, such as Tween 80 and soya lecithin are used to prepare flaxseed oil microcapsules. Physico-chemical and bio-chemical characteristics of flaxseed oil microcapsules depend on process parameters, ratio of oil and matrix, and characteristics of the matrix. As an example, the size of the microcapsule, prepared with spray-drying and freeze-drying ranges between 10–400 and 20–5000  $\mu$ m, respectively. It may be considered that the comprehensive information on the encapsulation of flaxseed oil will boost the development of functional foods and biopharmaceuticals.

**Keywords:** flaxseed oil; microencapsulation; emulsion; matrix; emulsifier; spray-drying; freeze-drying; characterization of microcapsule



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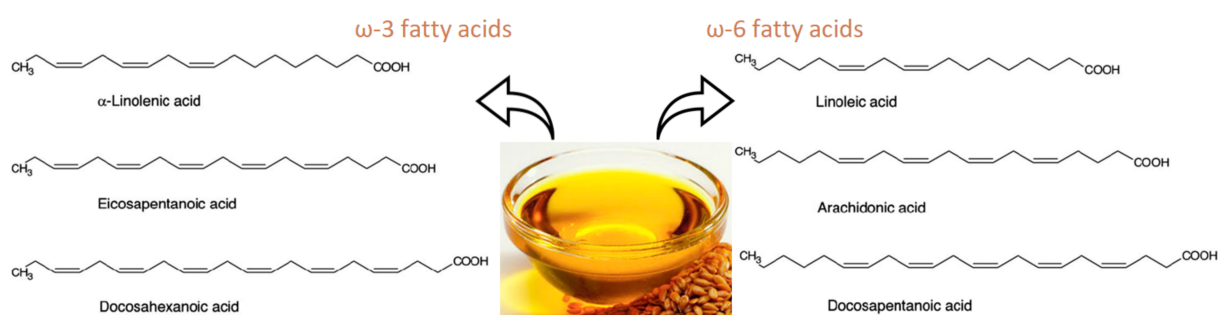
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## 1. Introduction

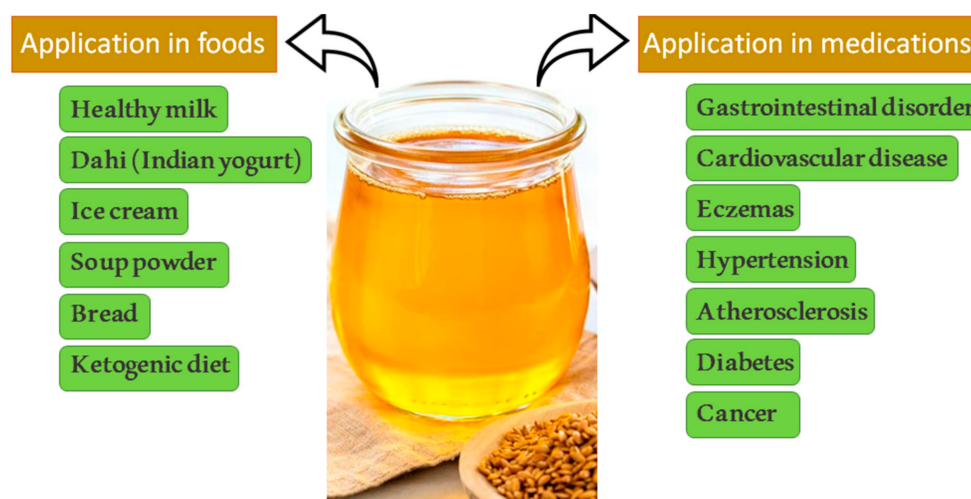
Flaxseed (latin name *Linum usitatissimum*) is an abundant source of polyunsaturated fatty acids, short chain polyunsaturated fatty acids [1], soluble and insoluble fibers [2], phytoestrogenic lignans, antioxidants [3], and proteins [4]. In flaxseed, the total amount of fat is quite high (41% weight basis) compared to the carbohydrate (29% weight basis) and protein (20% weight basis). In flaxseed oil, the contents of palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1),  $\alpha$ -linolenic acid (C18:3) ( $\omega$ -3), and linoleic acid (C18:2) ( $\omega$ -6) are 4.90–8.00%, 2.24–4.59%, 13.44–19.39%, 39.90–60.42%, and 12.25–17.44%, respectively [5]. Different types of  $\omega$ -3 and  $\omega$ -6 fatty acids present in flaxseed oil are represented in Figure 1.

Polyunsaturated essential fatty acids, such as  $\omega$ -3 and  $\omega$ -6 fatty acids are characterized by the presence of a double bond in three and six atoms, respectively away from the terminal methyl group in their chemical structure [1]. Both  $\omega$ -3 and  $\omega$ -6 fatty acids are important for cell membrane development and precursor molecules of many physiological elements, which are involved in controlling inflammatory reactions, blood pressure, and mortal cardiac diseases. Additionally,  $\omega$ -3 fatty acids reduce the risk of diabetes and certain

types of cancer. Eicosapentaenoic and docosahexaenoic acids can be synthesised from the  $\alpha$ -linolenic acid [6]. In many foods, the addition of  $\omega$ -3 fatty acid maintains the ratio of  $\omega$ -3 fatty acid to  $\omega$ -6 fatty acid [7]. Clinically, it has been proven that the ratio of  $\omega$ -6 fatty acid to  $\omega$ -3 fatty acid in a diet 4:1 or less is beneficial for health. Unfortunately, in many diets, this ratio ranges between 10:1 and 50:1 [8]. Therefore, flaxseed oil is popularly used in food and biopharmaceutical industries. In the food industry, flaxseed oil was used to prepare dahi (Indian yogurt) [9], healthy milk [10], ice cream [11], soup powder [12], and bread [13]. Presently, its application to develop the ketogenic diet has gained lots of attention [14–16]. In the biopharmaceutical industry, flaxseed oil is used to prepare formulations for the treatment and prevention of gastrointestinal disorders, cardiovascular disease, eczemas, hypertension, atherosclerosis, diabetes, and cancer [5]. The application of flaxseed oil in different food matrixes and biopharmaceuticals for the prevention of different diseases are represented in Figure 2.



**Figure 1.** Different types of  $\omega$ -3 and  $\omega$ -6 fatty acids present in flaxseed oil (self-developed, the concept was adopted from [5]).



**Figure 2.** Applications of flaxseed oil in different food matrixes and biopharmaceuticals for the prevention of different diseases (self-developed, the concept was adopted from [5,9–13]).

Biological activities of flaxseed oil against various autoimmune and chronic inflammatory diseases are associated with several mechanisms, such as (a) modifications in cell membrane lipid composition, (b) expression of genetic activity, (c) cellular metabolism and, (d) signal transduction [17]. Furthermore, orbitides or cyclolinopeptides with an antitumor activity are abundant in flaxseed oil [18]. The micronutrient tocopherol present in flaxseed oil acts as an antioxidant and suppresses the activity of reactive oxygen species. Even though flaxseed oil is rich in antioxidants, after its extraction from the seed and purification, fatty acids in flaxseed oil are easily oxidized. Oxidized fatty acids change the organoleptic prop-

erty of food and deteriorate the nutritional status [19–21]. Therefore, the microencapsulation of flaxseed oil is a prerequisite that ensures its stability in the food matrix.

However, several biological activities of flaxseed oil are reported [14] and its industrial production is still limited. It might be due to the lack of technologically needed information. Considering the great potentiality of flaxseed oil as well as the microencapsulation technology, some laboratory-scale investigations were performed by several research groups. In this review article, information about different technologies on the microencapsulation of flaxseed oil and biochemical characteristics of the microcapsule are discussed in a comprehensive way.

## 2. Microencapsulation

Microencapsulation has been explored in order to satisfy the increasing expectation of developing food ingredients with complex properties and functional values. It is an emerging technology which has been receiving interest in food and biopharmaceutical industries. It is used to protect encapsulated bioactive compounds and control their release. In the microencapsulation technology, small droplets of liquid or solid particles are coated within a thin film, known as a wall material or matrix [22,23]. For the microencapsulation of food-grade bioactive compounds, different techniques have been adopted and they can be classified into three distinctive categories. Those include (a) chemical methods: Entrapping the bioactive compound within the polymerized matrix, (b) physical methods: Spray-drying, spray coating, freeze-drying, and supercritical encapsulation processes, and (c) physico-chemical methods: Complex coacervation, entrapment within the nanostructured lipid matrix, ionotropic gelation, and molecular inclusion [24]. For the microencapsulation of flaxseed oil, two major steps are: Preparation of flaxseed oil emulsion with an aqueous solution of the matrix and subsequently, spray-drying or freeze-drying.

### 2.1. Emulsification

The emulsion preparation plays a key role in the encapsulation efficiency. An emulsion is a mixture of two or more immiscible liquids [25]. The emulsion stability is controlled by many factors. Flocculation, a reversible aggregation of droplets and coalescence, an irreversible fusion of droplets are two main types of emulsion instabilities. The emulsifier can make a bridge between polar and non-polar components, and provides stability in the emulsion [26]. To prepare the emulsion, two different types of technologies can be adopted. Those are (a) high energy consuming technologies using mechanical devices to mix up the water and oil phase, such as (i) ultrasound generator and (ii) high pressure homogenizers, as well as (b) low energy consuming technologies, such as (i) phase inversion temperature, (ii) membrane emulsification, and (iii) spontaneous emulsification of two immiscible liquids without any significant external thermal or mechanical energy [27]. In Table 1, a summary of different emulsification technologies is provided. The emulsion stability, droplets size, and their distribution are considerably affected by the adopted technologies [28]. It has been reported that the fine emulsion increases the organoleptic properties of microcapsules [29].

**Table 1.** Summary of different emulsification technologies.

Emulsification Techniques	Description	Reference
High-Energy Consuming Methods	Ultrasound generator	Due to the ultrasound (physical shear force), fine droplets are created. At a certain range of sound, a source pressure amplitude cavitation takes place and emulsification of the immiscible liquids occurs. [30]
	High pressure homogenizer	In a homogenizer, with the help of a pump the liquid is pressed with high pressure to a narrow channel, which offers shear force on immiscible liquids. It creates cavitation and leads to emulsion with a small droplet size. [31]

Table 1. Cont.

Emulsification Techniques	Description	Reference
Low-Energy Techniques	Phase inversion temperature	Due to the change in factors, such as temperature or pH, the activity of the emulsifier in terms of its hydrophilic—lipophilic balance is affected. It helps create the emulsion. [32]
	Membrane emulsification	Membrane emulsification is performed with the porous membrane. Hydrophilic liquid (oil) in a dispersed phase is pressed through the membrane pores to a continuous phase, generally hydrophilic liquid and the emulsion is formed in a continuous phase. [33]
	Spontaneous emulsification	In spontaneous emulsification, the immiscible liquids, such as oil and water along with the emulsifier create the emulsion without an external energy source. [34,35]

## 2.2. Spray-Drying

Spray-drying is a commonly used technology to prepare the encapsulation of vegetable oil [36]. In the spray-drying process, due to high heat, the water content is evaporated. Subsequently, the phase of the matrix is altered and solidification of the matrix takes place. Oil droplets are encapsulated within the molten matrix in a non-homogeneous way and the size of the microcapsule ranges between 10 to 400  $\mu\text{m}$  depending on the initial parameters [37]. Compared to other microencapsulation technologies, spray-drying is simple, may operate with a continuous mode, and has a low production cost. On the other hand, the disadvantages of spray-drying are (a) the availability of water-soluble matrixes is limited and (b) loss of heat energy [38]. In Figure 3, the process flow diagram of the spray-drying technology is represented.

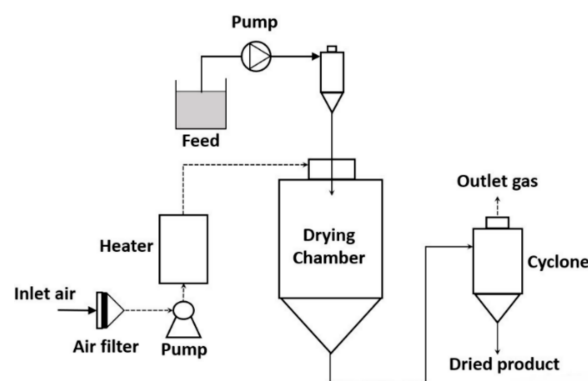


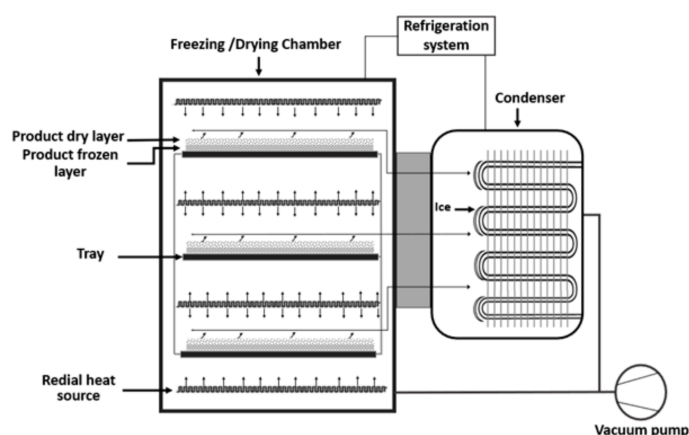
Figure 3. Schematic diagram of the spray-drying process (self-developed, the concept was adopted from [39,40]).

To obtain an optimum encapsulation efficiency with a minimum amount of oil on the surface of the matrix and maximum retention of the active compound, the composition of the emulsion, technology to prepare the emulsion, and parameters of the spray-drying process are taken into consideration [38,41,42]. The composition of the emulsion, size of droplets, and the viscosity of emulsion influence the quality of the spray-dried product. In the emulsion, the ratio of oil and matrix affects the stability of emulsion and encapsulation efficiency. It also affects the physical and biochemical properties of the spray-dried product [43,44]. The lower oil content and higher matrix to oil ratio lead to a smaller droplet of oil bodies in the emulsion. In this case, the amount of oil on the surface of

the matrix is reduced and encapsulation efficiency is increased. In the emulsion, viscosity is directly proportional to the droplet size and inversely proportional to the emulsion stability. These two conditions affect the encapsulation efficiency. The type of atomizer and its operating parameters are important to prepare the microcapsule since they influence the particle size. Among the existing atomizers used for breaking the bulk feed into a smaller droplet, the centrifugal wheel atomizer and spray pressure nozzle are the most used for the encapsulation of oil [40,43]. In the spray-drying process, the particle size of the product is increased with the increase in the emulsion flow. In the case of the spray pressure nozzle, the particle size of the spray-dried product is increased with the increase in the nozzle orifice diameter and decrease in the atomization pressure. In the case of the centrifugal wheel atomizer, an increase in the wheel diameter and speed provides a smaller size of the particle [43]. Furthermore, drying parameters provide the desired quality of the final product. The major drying parameters are the drying air flow, and inlet and outlet temperatures [43,44]. A high inlet temperature in the spray-drying process may lead to deterioration of the encapsulated active compound and an imbalanced evaporation of water from the matrix, which affects the encapsulation efficiency. On the other hand, a higher water content in the encapsulated product and agglomeration of the microcapsule may take place at a very low inlet temperature in the spray-drying process. Furthermore, the outlet temperature in the spray-drying process influences the stability of the microcapsule and retention of the encapsulated product. However, the outlet temperature in the spray-drying process cannot be regulated in a direct way, it can be monitored in an indirect way by controlling the solid content in the feed, inlet temperature, and feed flow rate. The spray air flow rate affects the quality of the final product. It controls the stickiness of the dried particles, deposited onto the wall of the drying chamber [43].

### 2.3. Freeze-Drying

Freeze-drying is also known as the lyophilization process. It is used for the dehydration of high temperature sensitive bioactive compounds, including flaxseed oil and aromas. During the freeze-drying process, a reduction of the surrounding pressure, heating of the emulsion, and sublimation of the frozen water in the material take place [39]. After crystallization of water in the emulsion, sublimation of water takes place at a minimal temperature. It promotes the transformation of water from a solid phase to vapor, directly [45]. In Figure 4, the process flow diagram of the freeze-drying technology is represented.



**Figure 4.** Schematic diagram of the freeze-drying process (self-developed, the concept was adopted from [39,46]). Reproduced with permission from [46]. Copyright Elsevier, 2016.

It is considered an expensive drying technology compared to the spray-drying process [47]. Furthermore, freeze-drying is a time occupying process, which consumes high energy. Due to the use of low temperature in the freeze-drying process, there is no thermal deterioration in the bioactive compound and less degradation in the heat-sensitive product.

The moisture content in the final product is controlled by freeze-drying. Therefore, this technology provides a better quality of the product with a remarkable preservation of sensory properties of food ingredients [39,45,48]. Similar to spray-drying, the properties of the emulsion, characteristics of the matrix, and ratio of the matrix and oil are major factors to ensure the quality of the freeze-dried product. Maltodextrin, gum Arabic, and protein are commonly used as a wall material or matrix for microencapsulation through freeze-drying [40]. The freezing rate can affect the morphology of the microcapsule. A faster rate of freezing of the emulsion can lead to aggregation of the freeze-drying products. In the freeze-drying process, the system pressure and temperature influence the properties of the microcapsule. Furthermore, the operational time of drying is important to achieve a stable moisture content in the microcapsule and the stability of the final product [44,45]. Depending on the process parameters, a particle size of the freeze-dried product remains between 20 to 5000  $\mu\text{m}$  [45].

#### 2.4. Matrix (Wall Material)

For the microencapsulation of flaxseed oil, a selection of the suitable matrix, accepted in the food industry has a great importance. The matrix provides the desired stability of the encapsulated product, increases the encapsulation efficiency, and controls the release of the encapsulated item into the environment. Furthermore, the matrix provides unique physico-chemical and bio-chemical characteristics of the microcapsule [43,49]. The inexpensive wall material may reduce the cost of the process [49]. The water soluble wall material is preferable for the encapsulation of flaxseed oil. In the case of spray-drying, the wall material should be soluble in an aqueous medium to shield the encapsulated material from the external environment [40]. The drying characteristics of the matrix influence the moisture content of microcapsules. If the matrix has a chance to dry with high temperature at a minimal time, the moisture content in the microcapsule is reduced. Therefore, the encapsulated item has less chance to be contaminated with water [43,49]. An aqueous solution of the selected wall material is needed to have low viscosity with a high concentration of the solid. It helps obtain a fine microcapsule (lower particle size) and control the release of the encapsulated product. The Newtonian behavior of the emulsion is desirable for spray-drying, a continuous industrial drying process. In the case of spray-drying, previous knowledges about the glass transition temperature of the oil and matrix are a prerequisite to ensure the stability of the microcapsule, and avoid the stickiness of the obtained powder in the spray-drying chamber [43]. In Table 2, the biochemical characteristics of different matrixes along with their advantages and disadvantages for preparing the microcapsule are represented.

**Table 2.** Characteristics of the matrix used for the microencapsulation of flaxseed oil.

Matrix	Source	Characteristics	Advantages	Disadvantages	Reference
Gum Arabic	Extracted from <i>Acacia senegal</i> (L.) or <i>Acacia seyal</i> (L.).	<ol style="list-style-type: none"> <li>1. It is a mixture of polysaccharides, oligosaccharides, and glycoproteins.</li> <li>2. Hydrolysis of polysaccharides produce arabinose, galactose, rhamnose, and glucuronic acid.</li> <li>3. It is soluble in water.</li> </ol>	<ol style="list-style-type: none"> <li>1. Well accepted film-forming ability.</li> <li>2. It has an emulsifying property due to the presence of protein.</li> <li>3. Low viscosity in aqueous solution.</li> <li>4. Stable in aqueous emulsion.</li> <li>5. High solubility in aqueous solution.</li> <li>6. Good retention of flavour.</li> </ol>	<ol style="list-style-type: none"> <li>1. Expensive.</li> <li>2. Variable availability and quality.</li> <li>3. Limited potentiality to prevent oxidation of the encapsulated item.</li> </ol>	[40,43]
Maltodextrin	Enzymatically derived from corn ( <i>Zea mays</i> ), potato ( <i>Solanum tuberosum</i> L.), rice ( <i>Oryza sativa</i> ), and wheat ( <i>Triticum aestivum</i> L.) starches.	Maltodextrin consists of D-glucose, linked with $\alpha(1\rightarrow4)$ glycosidic bond. Maltodextrin can be of variable length according to the degree of polymerization. Typically, it varies from 3 to 17 glucose units. Maltodextrins are classified according to the dextrose equivalent. The higher value of dextrose equivalent signifies a shorter glucose chain, higher solubility, higher sweetness, and lower heat resistance.	<ol style="list-style-type: none"> <li>1. Low cost.</li> <li>2. High potentiality to prevent oxidation of the encapsulated item.</li> <li>3. Easily digestible in the intestine.</li> <li>4. Highly soluble in water.</li> <li>5. Low viscosity with a high solid content in the emulsion.</li> <li>6. Heat resistance.</li> </ol>	<ol style="list-style-type: none"> <li>1. Poor emulsifying property.</li> <li>2. Poor flavour retention.</li> <li>3. Sometimes offer allergenic activity.</li> </ol>	[43]
Modified starch	Native starch is collected from corn ( <i>Zea mays</i> ), potato ( <i>Solanum tuberosum</i> L.), rice ( <i>Oryza sativa</i> ), and wheat ( <i>Triticum aestivum</i> L.) starches.	It is prepared by physical, enzymatic, or chemical treatment of native starch, which changes according to the property of the native starch.	<ol style="list-style-type: none"> <li>1. Well soluble in water.</li> <li>2. Low viscosity.</li> <li>3. Excellent volatile compound retention.</li> <li>4. Excellent emulsifying property.</li> <li>5. Provide stability in emulsion.</li> <li>6. Heat stable.</li> <li>7. Odourless and tasteless.</li> <li>8. Low cost.</li> </ol>	Provide allergenicity to food due to the presence of gluten.	[37,40,43]
Methyl cellulose	Methyl cellulose is not present in the plant cell wall. After the collection of natural cellulose from the plant cell wall, methyl cellulose is produced by the heat treatment of native cellulose with a sodium hydroxide solution and treating with methyl chloride.	Different types of methyl cellulose are produced by the substitution of different numbers of the hydroxyl group. It has an amphiphilic property.	<ol style="list-style-type: none"> <li>1. Stable viscosity over a wide range of pH (pH 3–11).</li> <li>2. Heat stable.</li> <li>3. Odourless and tasteless.</li> <li>4. High emulsifying property due to its amphiphilic structure.</li> <li>5. Satisfactory film-forming ability.</li> </ol>	Low solubility with a higher degree of polymerization.	[37,50]



Table 2. Cont.

Matrix	Source	Characteristics	Advantages	Disadvantages	Reference
Whey protein	Dairy milk	<p>1. Whey protein is a mixture of <math>\alpha</math>-lactalbumin (molecular weight: 14.2 kDa, isoelectric point: 4.2), <math>\beta</math>-globulin (molecular weight: 18.3 kDa, isoelectric point: 5.2–5.4), serum albumin (molecular weight: 66 kDa, isoelectric point: 4.9–5.1), lactoperoxidase (molecular weight: 78 kDa, isoelectric point: 9.6), lactoferrin (molecular weight: 78 kDa, isoelectric point: 8), immunoglobulin G (molecular weight: 150 kDa, isoelectric point: 6.5–9.5), immunoglobulin A (molecular weight: 320 kDa, isoelectric point: 4.5–6.5), and immunoglobulin M (molecular weight: 900 kDa, isoelectric point: 4.5–6.5).</p> <p>2. All whey proteins may denature with the heat treatment <math>\sim 70</math> °C for 20 min, but do not aggregate due to renneting or acidification of milk.</p>	<ol style="list-style-type: none"> <li>1. High solubility in aqueous solution.</li> <li>2. Satisfactory film-forming ability.</li> <li>3. Efficient to protect from oxidation.</li> <li>4. Good emulsifying property due to its amphiphilic structure.</li> </ol>	<ol style="list-style-type: none"> <li>1. Coagulate at lower pH of the emulsion.</li> <li>2. Heat sensitive.</li> <li>3. Provide allergenicity to food.</li> </ol>	[43]
Sodium caseinate	Dairy milk	<p>Casein is a phospho protein. Different types of casein proteins, such as <math>\alpha_{s1}</math>-casein, <math>\alpha_{s2}</math>-casein, <math>\beta</math>-casein, and <math>\kappa</math>-casein are present in the casein fraction of milk. Casein is produced by the neutralisation of acid precipitated casein with sodium hydroxide.</p>	<ol style="list-style-type: none"> <li>1. Highly soluble in aqueous solution.</li> <li>2. Good film-forming ability.</li> <li>3. High denaturation temperature.</li> <li>4. Good emulsifying property due to the presence of hydrophilic and hydrophobic amino acids in the protein structure.</li> </ol>	<ol style="list-style-type: none"> <li>1. Coagulate at a lower pH of the emulsion.</li> <li>2. Provide allergenicity to food.</li> </ol>	[43]
Vegetable proteins, such as lentil, chickpea, flaxseed, soy, pea proteins, etc.	<p>Proteins from lentil (<i>Lens culinaris</i>), chickpea (<i>Cicer arietinum</i>), flaxseed (<i>Linum usitatissimum</i>), soybean (<i>Glycine max</i>), pea (<i>Pisum sativum</i>).</p>	<p>Proteins from different plant sources have a unique amino acid sequence. Due to that, they offer a variety of biochemical activities.</p>	<ol style="list-style-type: none"> <li>1. Inexpensive and available throughout the year.</li> <li>2. Highly soluble in aqueous solution.</li> <li>3. Good film-forming ability.</li> <li>4. Efficient to protect from oxidation.</li> <li>5. Good emulsifying property due to its amphiphilic structure.</li> </ol>	<ol style="list-style-type: none"> <li>1. Coagulate at a lower pH of the emulsion.</li> <li>2. Heat sensitive.</li> <li>3. Some vegetable proteins, such as chickpea and soy-based proteins may provide allergenicity to the food product.</li> </ol>	[37,40,51]

### 2.5. Emulsifier

The emulsifier, also known as “emulgent” and “surface active agents”, has a great influence on the preparation of flaxseed oil emulsion in the aqueous solution of the matrix, prior to the drying process. Emulsifiers are amphiphilic with hydrophilic/polar and hydrophobic/non-polar moieties [52]. The emulsifier reduces the interfacial tension between hydrophilic and hydrophobic compounds and makes them miscible [53]. Furthermore, emulsifiers have an antimicrobial property [54]. During the emulsification process, the hydrophilic group of emulsifier binds with water or the wall material and the hydrophobic group binds with the flaxseed oil. The concentration of the emulsifier and hydrophile–lipophile balance influence the stability of the emulsion, as well as the encapsulation efficiency [23,53,55]. For the preparation of flaxseed oil encapsulation, emulsifier Tween 80 [56] and soya lecithin [13] were used by several researchers.

### 3. Characterization of Microencapsulated Flaxseed Oil

Several physical and biochemical aspects were considered to characterize flaxseed oil microcapsules. The physical and biochemical properties include particle size, particle morphology, colour, moisture content, water activity, oxidative stability, encapsulation efficiency, and the release of bioactive compounds from the matrix [51,57]. These properties of flaxseed oil microcapsule depend on the type of matrix, ratio of oil and matrix or wall component, and type and amount of the emulsifier. Furthermore, the technology of microencapsulation preparation and operational parameters influence the characteristics of the flaxseed oil microcapsule. The most important characteristic of the encapsulation of flaxseed oil is the encapsulation efficiency, which is generally estimated by measuring the surface oil and total entrapped oil. Sometimes, high-performance liquid chromatography (HPLC) is used for this purpose [58]. The mean particle size and their distribution is generally evaluated by the dynamic light scattering analytical instrument [59]. The particle morphology and size are measured by electron microscopy. The moisture content influences the shelf life of the microcapsule and is measured by the evaporation of water. The moisture analyzer is used for this purpose [58,59]. The zeta potential of a microcapsule is a good indicator to understand the stability of the microcapsule in colloid and is measured by the zeta potential analyzer [59]. Fourier transform infrared (FTIR) spectroscopy allows the understanding of the functional groups modification of the oil, matrix, and emulsifier in the microcapsule [59,60]. The oxidation of oil and fat with the time progress is a considerable important factor since the oxidation of oil and fat changes the organoleptic property of food items. The oxidation of the encapsulated oil can be evaluated by determining the peroxide value, oxidation induction period, and thiobarbituric acid reactive substances (TBARS) [58]. An overview of the flaxseed oil microencapsulation process and characterization of the microcapsule in terms of the oil content, particle size, encapsulation efficiency, moisture content, and oxidative stability are represented in Table 3.

**Table 3.** Process conditions for producing the encapsulated flaxseed oil and their biochemical characterization.

Process	Wall Material (Matrix)	Oil Content	Emulsifier	Particle Size	Encapsulation Efficiency (%)	Moisture Content %	Oxidative Stability	References
Bench top spray-dryer	Combination of chickpea protein isolate and maltodextrin	10%	-	16.3–24.0 $\mu\text{m}$	88.72	3.66–4.07	Peroxide value 6.68–7.31 meq active $\text{O}_2/\text{kg}$ for the chickpea protein isolate.	[51]
		15%			86.69			
		20%			83.62			
	Combination of lentil protein isolate and maltodextrin	10%		90.42	3.65–4.12	Peroxide value 6.62–6.86 meq active $\text{O}_2/\text{kg}$ for the lentil protein isolate.		
		15%		87.89				
20%	85.61							
Spray-drying	Combination of whey protein isolate, methyl cellulose, maltodextrin, gum Arabic, and soya lecithin	>20%	Soya lecithin	10–50 $\mu\text{m}$	~90	1.8–3.1	Rancimat induction period after 10 months (h) for gum Arabic + soya lecithin: $5.9 \pm 0.1$ , gum Arabic + maltodextrin + soya lecithin: $2.8 \pm 0.1$ , gum Arabic + maltodextrin + whey protein isolate + soya lecithin: $6.8 \pm 0.3$ .	[13]
Coacervation, Spray-drying, Freeze-drying	Flaxseed gum, Flaxseed protein isolate	Oil-to-wall ratios 1:2, 1:3, and 1:4	-	For liquid microcapsules 90–130 $\mu\text{m}$	Maximum value 87.60 by spray-drying and 67.06 by freeze-drying.	For spray-drying 3.20–3.70 and for freeze-drying 4.18–4.47.	Peroxidase value (meq active $\text{O}_2/\text{kg}$ ) after 30 days are 2.85–5.52 for spray-drying and 3.25–8.72 for freeze-drying.	[61]
Spray-drying	Combination of maltodextrin and gum Arabic	14 and 20%	-	17.6–23.1 $\mu\text{m}$	The highest encapsulation efficiency was achieved with 14% oil. 54.6–90.7.	-	Induction time $2.83 \pm 0.62$ h, oxidative stability index 3.78 h for 14% oil.	[12]
Spray-drying	Combination of maltodextrin, whey protein concentrate, gum Arabic, modified starch, 100 Hi-Cap	20%	-	Droplet diameter: 0.6–26 $\mu\text{m}$	The lowest value obtained for maltodextrin and whey protein concentrate is 62.3–95.7.	1–3%	Peroxidase value (meq peroxide/kg oil) after 4 weeks for gum Arabic + maltodextrin: 138, modified starch + maltodextrin 138, Hi-Cap + maltodextrin: 124, whey protein concentrate + maltodextrin: 107.	[36]
Spray-drying, Freeze-drying	Zein	-	-	-	For spray-drying $93.26 \pm 0.95$ and for freeze-drying $59.63 \pm 0.36$ .	For spray-drying 3.49–5.06 and freeze-drying 4.94–5.33.	-	[62]

Table 3. Cont.

Process	Wall Material (Matrix)	Oil Content	Emulsifier	Particle Size	Encapsulation Efficiency (%)	Moisture Content %	Oxidative Stability	References
Spray-drying	<ul style="list-style-type: none"> <li>• Gum Arabic</li> <li>• Whey protein concentrate</li> <li>• Modified starch</li> <li>• Hi-Cap 100</li> </ul>	10% 20% 30% 40%	-	0.24–180 $\mu\text{m}$	Emulsions prepared with modified starch had the highest encapsulation efficiency, whereas emulsion prepared with whey protein concentrate had lowest encapsulation efficiency. 37–97.	For whey protein concentrate 0.36–0.78, for gum Arabic 0.89–1.74, for modified starch 0.19–0.53; Hi-Cap 100.	Peroxidase value (meq peroxide/kg oil) for modified starch. Hi-Cap 100 is 0.5–1.8, 3.1–4 for gum Arabic and 1.3–2 for whey protein concentrate.	[63]
Freeze-drying	Combination of lentil protein isolates and maltodextrin	10, 20, 30%	-	4.2–6.7 $\mu\text{m}$	Highest encapsulation efficiency ~62.8.	<6.0%	Peroxide value on day 30 for 4.0% native lentil protein isolates + 36% maltodextrin + 10% oil 25.57 and 14.75 meq of active $\text{O}_2$ /kg for free oil and entrapped oils, respectively.	[64]
Spray-drying	Combination of whey proteins concentrate, sodium caseinate, lactose and ascorbyl palmitate	12.5%	-	0.54–70.6 $\mu\text{m}$	86.77–84.51%	3.88–3.98	Peroxide value after 6 months varied from 0.81 to 0.99 meq peroxides/kg.	[65]
Spray-drying	Modified starch	30%	-	0.5–100 $\mu\text{m}$	90.9%	3.5%	Induction period of the microcapsules exceeded 50 h for all times.	[57]
Spray-drying	Combination of gum Arabic, maltodextrin, skimmed milk powder, and Tween 80	8–22%	Tween 80	-	70–86%	3.2–4.8%	Peroxide value varied from 1–1.28 meq/kg.	[56]
Spray-drying	Gum Arabic	10–30%	-	0.1–477 $\mu\text{m}$	51–92%	-	Peroxide value 0.017–0.106 meq peroxide/kg oil.	[66]
Spray dried	Combination of whey protein concentrate, sodium alginate, and maltodextrin	4.5–5%	-	1–10 $\mu\text{m}$	30.69–84.39%	-	Peroxidase value (meq/kg oil) 3.46–6.84.	[67]

Table 3. Cont.

Process	Wall Material (Matrix)	Oil Content	Emulsifier	Particle Size	Encapsulation Efficiency (%)	Moisture Content %	Oxidative Stability	References
Freeze-drying	Combination of whey protein isolate, maltodextrin, and low density sodium alginate	10%	-	-	27.01–95.44%	-	Peroxide value for the emulsion with 20.24 total solids content (g/100 g emulsion) was increased from 1.5 to 46.5 meq/kg oil after freeze-drying.	[68]
Freeze-drying	Combination of tertiary conjugate of gelatin, flaxseed mucilage, and oxidized tannic acid	15, 30, and 50%	-	-	>90%	-	Peroxide value increased from 3.0–5.3 meqO <sub>2</sub> /kg.	[60]
Spray-drying	Combination of maltodextrin and pea protein isolate	20% and 40%	-	Particle size distribution ~24 µm	For 20% oil 35.2–95.6% and for 40% oil 22.3–93.6%.	-	-	[69]
Spray-drying	Combination of maltodextrin and whey protein concentrate	20%	-	5.47–7.09 µm	Ranged between 81.3–95.3%	3.16–4.91% (weight basis)	-	[70]
Spray-drying	Different combinations of soy protein isolate, pea protein isolate, wheat dextrin soluble fiber, and trehalose	35%	-	Mean diameter of particles 18–40 µm	Microcapsules with the protein-trehalose matrix 98–94%. Microcapsules with the protein-soluble fiber matrix 81–62%.	1.5–2.3%	Peroxide value of microencapsulated oil before storage: 1.80–7.90 meqO <sub>2</sub> /kg and after 12 weeks 4–27 meqO <sub>2</sub> /kg.	[71]
Spray-drying	Gum Arabic	10%		Droplets mean diameter 1.854 µm	~92%		Peroxide value ~0.032 meq/kg oil.	[72]
		20%		Droplets mean diameter 2.191 µm	~75%		Peroxide value ~0.036 meq/kg oil.	
		30%		Droplets mean diameter 2.479 µm	~52%		Peroxide value ~0.036 meq/kg oil.	
		40%		Droplets mean diameter 3.464 µm	~40%		Peroxide value ~0.04 meq/kg oil.	

#### 4. Conclusions

The presence of  $\omega$ -3 and  $\omega$ -6 polyunsaturated fatty acids in flaxseed oil is abundant. To protect the polyunsaturated fatty acid from oxidation and to conserve its biochemical importance, the microencapsulated flaxseed oil has been used in the food matrix. To prepare the microcapsule of flaxseed oil, first emulsification and subsequently dehydration have been used. During the preparation of the emulsion, the amount of flaxseed oil, matrix, and emulsifiers have been varied. It is believed that their ratio significantly affects the encapsulation stability and functional properties of the microcapsule. Drying of the emulsion has been performed using the spray-drying or freeze-drying technology. Different unit operations to prepare the flaxseed oil microcapsule, such as mixing of flaxseed oil and an aqueous solution of the matrix, drying temperature, and air flow rate have been varied. It was shown that the mentioned parameters significantly affected the encapsulation efficiency, physico-chemical, and bio-chemical properties of the flaxseed oil microcapsule. Wrapping up all the information, it is perceived that flaxseed oil may receive a high reputation to develop functional foods and ketogenic diet. Furthermore, the flaxseed oil may gain a high status to develop new biopharmaceuticals. Therefore, it is expected that this review may receive attention from different research communities, as well as food and biopharmaceutical industries.

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