Microencapsulation of omega-3 fatty acids: What it is, how it's made, and challenges in food technology

Sri Haryani Anwar

Department of Agricultural Product Technology, Syiah Kuala University,
Banda Aceh 23111, Indonesia. Corresponding author: sri.haryani@unsyiah.ac.id

Abstract. Fatty acids with double bonds beyond the ninth carbon from the carboxyl end are classified as essential for human health, including omega-3 fatty acids: eicosapentaenoic acid (EPA) and docosahexanoic acid (DHA). The main sources of omega-3 fatty acids are fatty fish species such as herring, mackerel, sardine and salmon. Oils from the marine algae *Cryptecondium conchii* are mainly rich in DHA only, while fish oil contains both EPA & DHA. Why is it important to microencapsulate EPA & DHA? Because these fatty acids cannot be synthesized by the human body but have to be obtained through nutrition uptake. The beneficial effects of these fatty acids including lowering cholesterol, decreasing the risk of arrhythmia, lowering the blood pressure, preventing diabetes in pregnancy, and positive effects on joints (relief of arthritis). EPA and DHA also play an important role in early infant nutrition and the imbalance of these fatty acids is believed to cause a variety of diseases. Because of their sensitivity to oxidation, these fatty acids need to be stabilized to protect them from oxidation. In food application, their interaction with other food ingredients needs to be prevented. Attempts to prevent oxidation to allow omega-3 fatty acids to fulfill their functions are not trouble-free. Fish oils in their natural state have a taste and smell that make them less attractive to consumers. Processing technology for masking the smell and taste of fish oil in food faces great challenges. Therefore, to address the problems concerning the susceptibility of fish oil to oxidation and its unpleasant smell, microencapsulation, where the oil is packaged within coating materials, may be used to replace bulk oils. This paper discusses great challenges faced by the scientists to microencapsulate omega 3 fatty acids from fish oil after introducing concise information related to microencapsulation and its advance techniques. As the demand of functional food containing omega-3 is continuously growing, overcoming those challenges mean solving one problem in providing healthy food for the world.

Key words: microencapsulation, fish oil, omega-3 fatty acids, spray granulation, spray drying, freeze drying, fluid bed coating.

What is Microencapsulation?

Microencapsulation is defined as "the technology of packaging solid, liquid, and gaseous materials in matrices or small capsules that release their contents at controlled rates over prolonged periods of time" (Champagne and Fustier, 2007;Thies, 1987). The substance to be encapsulated is called "core", while the microencapsulating agent surrounding the core is defined as "wall". The core is also known as "active agent", and the term "wall" is also referred to "matrix, coating material, or shell". Microcapsules often have a diameter between 3 and 800 microns and contain 10 to 90 wt % core. The shell is designed to prevent diffusion of material from a microcapsule or into a microcapsule (Thies, 2004), to protect the core from deterioration, and to release it under the desired conditions (Young et al., 1993).

The first commercial application of microencapsulation technology began in the late 1930s and 1940s with the development of "carbonless paper" by the National Cash Register (Deasy, 1984). Nowadays, the encapsulation technology has been applied broadly in the food industry to microencapsulate sensitive food ingredients such as flavors, spices, vitamins, carotenoids, and omega-3 oils. The technology is aimed to protect sensitive ingredients from chemical degradation by blocking the direct influence of oxygen, pressure, heat, pH, heavy metals and other influences that may cause or accelerate degradation. In the case of vitamins, the protection is essential to maintain vitamin levels, while flavor encapsulation is important to avoid unwanted off-taste. Microencapsulation of pigments such as β-carotene is necessary to achieve special physical effects such as high colour strength and special colour hue (Runge, 2004).

What are Omega-3 PUFAs and Their Nutritional Benefits

Polyunsaturated fatty acids (PUFAs) are composed of 18 or more carbon atoms and a terminal carboxylate group having two or more double carbon bonds. Classification of these fatty acids is determined by the position of the first double bond, as counted from the methyl terminus. The one with its first double bond at position 3 as counted from the

methyl terminus is called omega-3 PUFA, while the one located at position 6 is omega 6. These fatty acids are also known as *omega-3 (linolenic)* and *omega 6 (linoleic)* (O´Brien, 2004).

The symbol omega (ω) and its synonym *n* is often used to classify PUFAs (Sijtsma and de Swaaf, 2004). Alpha-linolenic acids (18:3 Δ9, 12, 15), eicosapentaenoic acid (EPA, 20:5 Δ5, 8, 11, 14, 17), and docosahexanoic acid (DHA, 22:6 Δ4,7,10,13,16,19) are the most studied PUFAs within this group (Table 1).

Table 1. ω-3 PUFAs, adapted from (Sijtsma and de Swaaf, 2004)

Common name	Systematic name (with all double bonds in *cis*-configuration)	Short name
α-Linolenic acid	Δ9, Δ12, Δ15-Octadecatrienoic acid	ω-3 18:3
	Δ6, Δ9, Δ12, Δ15-Octadecatetraenoic acid	ω-3 18:4
	Δ8, Δ11, Δ14, Δ17-Eicosatetraenoic acid	ω-3 20:4
Eicosapentaenoic acid	Δ5, Δ8, Δ11, Δ14, Δ17-Eicosapentaenoic acid	ω-3 20:5
	Δ7, Δ10, Δ13, Δ16, Δ19-Docosapentaenoic acid	ω-3 22:5
Docosahexaenoic acid	Δ4, Δ7, Δ10, Δ13, Δ16, Δ19-Docosahexaenoic acid	ω-3 22:6
	Δ5, Δ8, Δ11, Δ14, Δ17, Δ20- Tetracosahexaenoic acid	ω-3 24:6

The main sources of omega-3 PUFAs are fatty fish species such as herring, mackerel, sardine and salmon (Keogh et al., 2001). Marine oils, including fish oil, are the complex mixture of fatty acids with varying lengths and degrees of unsaturation (Shahidi and Wanasundara, 1998). A high intake of PUFA is associated with a low incidence of coronary heart disease (CHD) and reduced risk of cancer (Wallace et al., 2000). Docosahexaenoic acid (DHA) is an essential component of the cell membranes of human tissues and accounts for over 60% of the total fatty acids in the rod outer segment in the retina (Giusto et al., 2000). It is also regarded essential for the proper visual and neurological development of infants because of its role as a structural lipid component.

PUFAs have also been claimed to have a broad range of beneficial effects including lowering cholesterol, decreasing the risk of arrhythmia, lowering the blood pressure, preventing diabetes in pregnancy, and beneficial effects on joints (relief of arthritis) (McMurray, 2007). Both omega-3 and omega-6 PUFA are precursors of hormone-like compounds, which are involved in many important biological processes in human body (Trautwein, 2001).

Why do Omega-3 Fatty Acids Need to be Encapsulated?

Fatty acids with double bonds beyond the ninth carbon from the carboxyl end of the compound are classified as essential for human health. These fatty acids are important nutrients, which cannot be synthesized by the human body but have to be obtained through nutrition uptake.

In functional food development, incorporation of PUFAs into food products is dominated by omega-3 fatty acids (α-linolenic acid (ALA) C18:3n-3, eicosapentaenoic acid (EPA) C20:5n-3, docosahexaenoic acid (DHA) C22:6n-3) and omega-6 fatty acids (γ-linolenic acid (GLA) C18:3n-6 and arachidonic acid (AA) C20:4n-6) (Augustin and Sanguansri, 2003). Soybean, canola, flaxseed, hemp, and perilla oils are the major sources of ALA, while GLA is mostly found in evening primrose, blackcurrant and borage oils. Oils from the marine algae *Cryptecondium conchii* are mainly rich in DHA only, while fish oil contains both EPA & DHA (Trautwein, 2001). Although the nutritional values of fish oil are recognized, adequate daily intake is difficult to achieve. Fish consumption is relatively low in many countries, especially consumption of oily fish with high levels of omega-3 PUFAs (Kelly and Keogh, 2000). Because of their sensitivity to oxidation, fish oils need to be stabilized to protect them from oxidation.

Attempts to prevent fish oil oxidation to allow omega-3 fatty acids to fulfill their functions are not trouble-free. In addition, fish oils in their natural state have a taste and smell that make them less attractive to consumers (McMurray, 2007). Processing technology for masking the smell and taste in food systems faces great challenges. Therefore, to address the problems concerning the susceptibility of fish oil to oxidation and its unpleasant smell, microencapsulation, where the oil is packaged within carrier materials, may be used in place of bulk oils.

How to Microencapsulate Food Ingredients?

Thies (2004) classified the encapsulation process as chemical (A) or mechanical (B) processes (Table 2). A chemical process may rely only on the physical phenomena, while in a mechanical process a chemical reaction may actually be involved. Some typical processes used for producing microcapsules for food application are: (A) complex coacervation, polymer-polymer incompatibility and submerged nozzle processes, and (B) spray drying, spray chilling, fluidised bed coaters, liquid extraction, melt extrusion, suspended nozzles, and spinning or rotating discs (Thies, 2004).

Table 2. Classification of encapsulation processes (Thies, 2004)

Type A (chemical) process	Type B (mechanical) process
Complex coacervation	Spray drying
Polymer/polymer incompatibility	Spray chilling
Interfacial polymerization in liquid media	Fluidised bed
	Electrostatic deposition
In-situ polymerization	Centrifugal extrusion
In-liquid drying	Spinning disk or rotational suspension separation
Thermal and ionic gelation in liquid media	Polymerization at liquid/gas or solid/gas interface
Desolvation in liquid media	Pressure extrusion or spraying into solvent extraction batch
	Hot-melt extrusion

In type A process, microcapsules are produced entirely in a liquid-filled stirred tank or tubular reactor. In type B process, microcapsules are formed by spraying droplets of coating materials on a core material being encapsulated, where the liquid droplets are solidified by spraying them into a gas phase. This process also allows the gelling droplets to be sprayed into a liquid bath, or a polymerization reaction can be carried out at solid/gas or liquid/gas interfaces of dispersed particles or droplets. There is no single encapsulation process that is able to produce a full range of capsules needed by the users (Thies, 2004).

The selection of a method depends on economic reasons, sensitivity of the core, size of microcapsule desired, physical and chemical properties of both core and coating, application for the food ingredient and the release mechanism. Microencapsulation processes involve both physical and chemical techniques (Jackson and Lee, 1991). In the following, common methods used to microencapsulate food ingredients and essential oils are described briefly, including: spray drying, freeze drying, fluid bed film coating process and one advance method called spray granulation.

1. Spray drying

Spray drying is the most commonly used encapsulation method in the food industry (Shahidi and Han, 1993). This method uses available equipment, has high production capacities (up to 4,000 kg/h), low process cost (20% of that of freeze drying and 30% of that of vacuum drying), applies a wide choices of carrier solids, and has low effective process temperatures (Reineccius, 2004).

The spray drying process involves: 1) formation of an emulsion or suspension of coating and core material, 2) atomization of the emulsion into a drying chamber containing circulating hot dry air, and 3) evaporation of moisture from the emulsion droplets when in contact with the hot air (Jackson and Lee, 1991). In the food industry, the core is generally a water-immiscible flavour, vitamin, animal fat, fish oil or plant oil. The core is emulsified in an aqueous solution of coating material (Thies, 2004).The carrier is usually hydrated until it

reaches a chosen solids level. The upper limit of infeed solids is the viscosity at which the infeed cannot be adequately atomised, or if the material is not to be dried in the chamber. High infeed solids will produce large particles that may not dry, and thus impinge on the dryer wall, stick to the wall and ultimately burn on (Reineccius, 2004).

To prepare a stable emulsion, the exact calculation of dissolved solid content in the feed is crucial. High solid content in the prepared emulsion significantly increases the core retention by: (1) decreasing the time needed to form a semi-permeable membrane at the surface of the drying particle, (2) increasing emulsion viscosity which prevents the circulation movement inside the droplets and leads to rapid skin formation (Re, 1998).

2. Freeze drying

Freeze drying or lyophilization is an attractive drying method for extending food shelf life (Ma and Arsem, 1982) and was first developed to prevent the flavor and aroma losses that occur when a conventional drying method is used (Dalgleish, 1990). It is the best method for water removal using the lowest drying temperature than any other drying method to obtain highest quality of final products (Ratti, 2008;Heldman and Hartel, 1997). Despite its advantages, freeze drying is known as an expensive drying method, particularly because of high operating and maintenance costs as well as long drying time under continuous vacuum, which increases energy consumption (Ratti, 2008). The cost of freeze drying is twice than that of vacuum belt drying and almost five times than that of spray drying. However, the high cost of freeze drying can be compensated if the products are in high demand (Heldman and Hartel, 1997).

Freeze drying process involves freezing, primary drying, and secondary drying. Freezing is removal of heat that lowers the temperature of foods below 0 °C. At this temperature, the ice crystals begin to form and the solutes present in intra- and extra-cellular fluids become concentrated in the remaining liquid water (Fletcher, 2002). During primary drying, the sublimation front is formed. This front is boundary between the frozen and dried product. The heat must be transferred into the product to this front to accelerate sublimation, and the water vapor must be removed by mass transfer through the dried product (Heldman and Hartel, 1997). The water vapor produced in the sublimation interface is removed through the outer porous layers of the product (Mellor, 1978).

The secondary drying begins when all the ice is sublimed out of the frozen product. For the drying process, heat is added at a slower rate considering that the moisture loss only takes place through diffusion of water molecules out of the freeze-dried matrix (Heldman and Hartel, 1997). At this stage, the bound water, of which the main part is in an unfrozen state, must be dried. In freeze drying, sublimation results from the replacement of the ice layer by air, and therefore the droplets remain entrapped in the matrix. In a series of studies, Heinzelmann et al. (1999; 2000a,b) explored fish oil microencapsulation by freeze drying.

The first investigation reported that the addition of a mixture of antioxidants (ascorbic acid, lecithin, and tocopherol) improved the product shelf life. The second investigation examined several process parameters in the oxidative stability of fish oil powder during storage at 25 ^{o}C. The variables were microencapsulation matrices (sodium caseinate, lactose and maltodextrin), homogenization pressures (1 pass at 10 MPa and 3 passes at 40 MPa), and freezing rates (slow, medium and fast). In the slow freezing process, 50 ml emulsion was frozen in a petri dish at -20 °C for 10 h. The medium freezing process involved pumping the emulsion into a self-conveying scrape cooler (freezing extruder) where the twin screws were covered by a jacket cooled with a refrigerant (-40 °C). In the fast freezing process, the emulsion was dropped into stirred liquid nitrogen using a burette (Heinzelmann et al., 2000).

Although the authors found that low homogenization pressure resulted in larger oil droplet size, lower total oil content, higher free oil content and lowest ME, they concluded a negative correlation between oil globule size or microencapsulation efficiency with the storage stability. The ME increment was inversely related to the freezing rate. The highest ME corresponded to slow freezing rate, followed by medium and fast freezing. The overall results indicate that high ME did not necessarily correlate with high storage stability. Microcapsules prepared from fast freezing exhibited the longest shelf life (12 weeks) (Heinzelmann et al., 2000).

3. Fluid bed film coating

The term film coating refers to the encapsulation technologies that utilise a spray process to deliver film material to a core particle. The technique is based on the use of fluidising air to provide a uniform circulation of particles past an atomizing nozzle. As the atomized coating materials contact the particles, fluidising air evaporates solvent or solidifies coating solids on the particles as part of a developing film. This process is continued until the desired film thickness is achieved (Frey and Hall, 2004).

Two terms have been identified in this field: fluidized bed spray granulation and fluidized bed film coating. The difference is the size of the particles to be coated. In the first process, a solid containing liquid is transformed into granules by atomizing it into seed particles that have the same composition as the dissolved component. The liquid can be a solution, suspension or melt and dried continuously in one step in which the solvent is evaporated thus solid is deposited on the surface of seed particles. Particle growth can take place by two ways: agglomeration or surface layering. The latter is also known as the 'onion skin' layered structure (Link and Schlunder, 1997;Zank et al., 2001).

The main objective of the second process (film coating process) is to form individual particles in which each of the particle is well distributed and uniformly coated (Turton et al., 1999). An existing core has a larger particle size compared to the seed particle in the granulation process. Although the particle size differs from that of the starting particles, the principle steps in granulation and coating process are identical.

In each case, the particle growth is determined by the successful collision between a droplet of the liquid and the seed or core particle. Loeffler (1988) divided particle deposition into two steps: (1) droplet movement to the particle surface, and (2) droplet adhesion on the surface of the particle.

4. Spray granulation

The spray granulation process uses the basic principle of fluidised bed equipment in which the gas passes through the bed material and, at a certain velocity, the bed starts to fluidise. Similar to this principle, a novel apparatus was developed, i.e. a 'spouted bed'. It was originally developed to fluidise larger particles with a high-velocity spout of gas that penetrates to the bed and fluidise the particles upward (Jacob, 2009).

The spouted bed has three distinct regions: the spout, the annulus and the fountain. The apparatus consists of a cylindrical column with a conical base. An orifice is fitted to the conical base through which the spouting fluid is injected. The ascending flow particles inside the chamber are developed by the high fluid flow rate which finally forms a fountain. These fountain particles are directed toward the outer part of the spout and fall into the annular region (Rocha and Taranto, 2009). The spout is the central channel in the system, and in the spout region the particles move in the same direction as the gas flow. Due to the high velocity of the gas, particles move as in a pneumatic conveyor. The peripheral region is the dense region known as the annulus, where the particles move counter- current to the gas. The term fountain is used to describe the mushroom form above the annulus, and in this region the particles move in a decelerated regime subsequently falling into the annulus.

Glatt-ProCell spouted bed

A typical spouted bed manufactured by Glatt GmbH (Germany's leading company producing a wide range of machinery) called ProCell spouted bed. It has a rectangular shape with an extended process chamber. The bottom-end part of the chamber has inclined side walls, called the inside contour, which are supported by two cylinders of the gas throttle shaft positioned on top of a base plate. The two cylinders are separated by a centre profile and form two parallel gaps which functioned to divert the high-velocity fluidized gas entering the chamber. The cylinders are adjustable by rotating them so that the free cross-section area of the gas inlet can be varied. This condition allows control of the gas inlet velocity and its distribution to prevent clogging and a dead zone. The adjustment can be made without interrupting the process (Gryczka et al., 2009).

When the process has started, the fluidizing gas enters the equipment from the two slits horizontally and is diverted upwards by the centre profile. After passing this profile, the two flows are united and form a jet gas and pass the apparatus vertically from the bottom part to the top. The jet gas flow fluidizes seed particles (which have been inserted before

the process started) and bring up the particles. At this time, the liquid (in the form of emulsion, suspension or high-viscosity liquid) can be sprayed in by a nozzle located above the centre profile in the central region. The spraying direction is usually from the bottom to the top (as in the Würster fluid bed), but top spray is also possible. In the upper process chamber, the particles are separated to the sides and transported back to the lower area toward the gas entry zone due to the slope of the inner profile. At this point, the particles fluidized by the core jet into the spout zone and re-circulated (Gryczka et al., 2008).

The regions inside the ProCell spouted bed can be divided into jet zone and back-flow zone. The jet zone is the area above the middle profile where the fluidization gas flows with a high velocity and streams up the particles. The back-flow zone consists of the two zones adjacent to the jet zone.

Gryczka et al. (2009) underlined the advantages of the Glatt-ProCell spouted bed over the conventional fluidized beds. The apparatus is designed to fluidize various forms of particles, including fine, large, and irregular particles, and can be adapted to fluidize particles with a wide range of size distribution. As ProCell characterizes by a low drying temperature and possibility to reduce residence time in the drying chamber, therefore it is suitable for drying heat-sensitive ingredients.

Challenges in Food Technology

Typical fish oil microencapsulation is usually based on the formation of ordinary emulsion in which fish oil droplets are emulsified using combination of matrices and then spray dried or freeze dried to produce microcapsules. The main problem with these microcapsules is that the coating materials are not strong enough to withstand extreme processing conditions such as expose to heat, shear, and acidic environment thus PUFAs are release ahead of time. Moreover, the microcapsules are easily to dissolve during incorporation into food products.

In their latest publications, Anwar et al. (2010) and Anwar and Kunz (2011) underlined factors governing stabilization of fish oil by microencapsulation using four different methods: spray granulation (SG), fluid bed film coating (FC), freeze drying (FD), and spray drying (SD). They concluded that the powder stability against oxidation depends on the total amount of PUFAs contained in the fish oil, and also on the type of fatty acids, i.e., EPA or DHA. The microcapsules with the higher content of DHA oxidized more rapidly and produced more hydroperoxides and propanal. Microcapsules stability containing a high amount of omega-3 (620 mg/g) is governed by the best combination of matrices and type of drying method. The mixture of soybean soluble polysaccharides (SSPS) and modified *waxy* corn starch (OSA-starch) is found superior with respect to stabilization of the microcapsules compared with the other matices.

Spray granulation (SG) is proved to be the best drying process to produce stable microcapsules. Application of high drying temperature is found to be the most critical factor determining product stability. Processing or drying time is less crucial than the exposure to heat treatment. Though SG needs ± 60 min. to produce a desirable size of granules compared to only a few seconds in SD, the results confirm that spray granulation at ± 70 ^{o}C means a lower chances of lipid degradation by autoxidation than spray drying at ± 180 ^{o}C. High drying temperature induced initial development of primary oxidation products as well as hastened rapid degradation of them to become stable secondary oxidation products (Anwar et al., 2010).

The results based on Peroxide Values (PVs) and propanal verified that there is another cause of oxidation other than types of coating material and heat. This factor is the particle microstructure. Though freeze drying (FD) uses no heat or very low drying temperature in its process, oxygen diffusivity onto matrices becomes a rate-limiting factor toward lipid oxidation. The porous, irregular, and flake-like structure of the freeze dried powder accelerates oxidation due to an easy oxygen access into matrices which thus reach the non-encapsulated oil (Anwar and Kunz, 2011).

The superiority of spray granulation (SG) to produce stable microcapsules is to a great extent affected by the particle microstructure i.e., "multiple encapsulations" obtained by this method. Agglomeration of seed particles containing oil droplets in the first stage of process, followed by the envelopment of the seed by the layers growth, and finally the granule surface is coated by very fine particles have create a multi-protection system for

the lipids embedded inside the matrices. In addition, exposure to low-medium heat treatment in SG has maintained minimum formation of free radicals and unstable peroxides. Lower accumulation of oxidation initiators may keep the microcapsules from being affected by oxidation reactions (Anwar et al., 2010;Anwar and Kunz, 2009;Anwar and Kunz, 2011).

Above mentioned results in fish oil microencapsulation are only few examples of exploration in this fields that need to be continued. Since omega-2 fatty acids are fragile and the microencapsulation technology itself is growing very fast, the challenges in this area are enormous. One must take into account the history of how fish oil in liquid phase is produced, transported, and stored. Next is selection of coating materials to be used for microencapsulation. Afterward, formation of stable emulsion must be obtained with or without antioxidants following by drying using appropriate selected-method. Finally storage of dried microcapsules in the best container stored at low temperature with the absent of light is recommended.

One important point that needs to be addressed is that the emulsions and their stability are the basis for microencapsulation of food ingredients. Previous state of the arts in fish oil microencapsulation mainly emphasize to create emulsion containing fish oil to be used only in liquid foods, some are further spray dried or dried by other drying methods but the final particles are easily to dissolve and oxidation of omega-3 fatty acids was detected (Klinkesorn et al., 2005;Shaw et al., 2007). Others used existing dried microcapsules available in the market for direct food enrichment without knowing how they are produced.

In order to produce superior microcapsules the emulsion technology must be understood very well by food technologist. Recently, there have been a number of studies to develop water-in-oil-in-water (W/O/W) emulsions. Nanotechnology is applied to modify the interfacial barriers between water and oil. However the stability of this type of emulsions is difficult to be maintained. Research in this particular field is mainly developing emulsion-based food products which limit its application due to storage and transport cost as well as further food enrichment.

Fish oil microcapsules that are claimed as successful products by researchers in this field are believed to have limitation particularly when they are dissolved into liquid food such as milk, yogurt, mayonnaise, and beverages. The cause of these problems is due to less protection given by the coating materials such as food polymers which are highly soluble in water. These problems have becoming the real challenges in food industries all around the globe and significant investigations in this particular topic are currently underway.

References

Anwar,H., J.Weissbrodt, and B.Kunz. 2010. Microencapsulation of Fish Oil by Spray Granulation and Fluid Bed Film Coating. Journal of Food Science 75:E359-E371.

Anwar,S.H., and B.Kunz. 2009. Proc. XVII International Conference on Bioencapsulation. Groningen, Netherlands.

Anwar,S.H., andB.Kunz. 2011. The influence of drying methods on the stabilization of fish oil microcapsules: Comparison of spray granulation, spray drying, and freeze drying. Journal of Food Engineering 105:367-378.

Augustin,M.A., andL.Sanguansri. 2003. Polyunsaturated fatty acids: delivery, innovation and incorporation into foods. Food Australia 55:294-296.

Champagne,C.P., andP.Fustier. 2007. Microencapsulation for the improved delivery of bioactive compounds into foods. Current Opinion in Biotechnology 18:184-190.

Dalgleish,J.M. 1990. Freeze drying for the food industries. Elsevier Applied Science, New York.

Deasy,P.B. 1984. Microencapsulation and related drug processes. New York: Marcel Dekker, Inc. p 1-60. Marcel Dekker, New York.

Fletcher,J.M. 2002. Freezing. p. 1-416. *In* C.J.K.Henry, and C. Chapman (ed.) Nutrition Handbook for Food Processors. Woodhead Publishing, Cambridge-UK.

Frey,C.R., and H.S.Hall. 2004. Single-core encapsulation: filmcoating. p. 83-100. *In* P.Vilstrup (ed.) Microencapsulation of food ingredients. Leatherhead International Limited, Leatherhead, United Kingdom.

Giusto,N.M., S.J.Pasquare, G.A.Salvador, P.I.Castagnet, M.E.Roque, and M.G.I.de Boschero. 2000. Lipid metabolism in vertebrate retinal rod outer segments. Progress in Lipid Research 39:315-391.

Gryczka,O., S.Heinrich, M.Jacob, N.G.Deen, and J.A.M.Kuipers. 2009. Proc. 9th International Symposium on Agglomeration.

Gryczka,O., S.Heinrich, V.Miteva, N.G.Deen, J.A.M.Kuipers, M.Jacob, and L.Morl. 2008. Characterization of the pneumatic behavior of a novel spouted bed apparatus with two adjustable gas inlets. Chemical Engineering Science 63:791-814.

Heinzelmann,K., K.Franke, J.Velasco, and G.Marquez-Ruiz. 2000. Microencapsulation of fish oil by freeze-drying techniques and influence of process parameters on oxidative stability during storage. European Food Research and Technology 211:234-239.

Heldman,D.R., and R.W.Hartel. 1997. Principles of food processing. Chapman & Hall, New York.

Jackson,L.S., andK.Lee. 1991. Microencapsulation and the Food-Industry. Food Science and Technology-Lebensmittel-Wissenschaft & Technologie 24:289-297.

Jacob,M. 2009. ProCell technology: Modelling and application. Powder Technology 189:332-342.

Kelly,P.M., and M.K.Keogh. 2000. Nutritional studies on dried functional food ingredients containing omega-3 polyunsaturated fatty acids. Rep. Teagasc, End of Project Report, Armis No. 4340. Moorepark, Fermoy, Co. Cork.

Keogh,M.K., B.T.O'Kennedy, J.Kelly, M.A.Auty, P.M.Kelly, A.Fureby, and A.M.Haahr. 2001. Stability to oxidation of spray-dried fish oil powder microencapsulated using milk ingredients. Journal of Food Science 66:217-224.

Klinkesorn,U., P.Sophanodora, P.Chinachoti, D.J.McClements, and E.A.Decker. 2005. Stability of spray-dried tuna oil emulsions encapsulated with two-layered interfacial membranes. Journal of Agricultural and Food Chemistry 53:8365-8371.

Link,K.C., andE.U.Schlunder. 1997. Fluidized bed spray granulation - Investigation of the coating process on a single sphere. Chemical Engineering and Processing 36:443-457.

Ma,Y.H., and H.Arsem. 1982. Low pressure sublimation in combine radiant and microwave freeze drying. *In* A.S.Mujumdar (ed.) Drying'82. McGraw-Hill, New York.

McMurray,I. 2007. Fishy Business. International Food Ingredients (2):23-26.

Mellor,J.D. 1978. Fundamentals of freeze drying. Academic Press, New York.

O´Brien,R.D. 2004. Fats and oils: formulating and processing for applications. CRC Press, New York.

Ratti,C. 2008. Freeze and vacuum drying of foods. p. 225-251. *In* X.D.Chen, and A.S. Mujumdar (ed.) Drying technologies in food processing. Blackwell Publishing, Singapore.

Re,M.I. 1998. Microencapsulation by spray drying. Drying Technology 16:1195-1236.

Reineccius,G.A. 2004. Multiple-core encapsulation: The spray drying of food ingredients. p. 151-185. *In* P.Vilstrup (ed.) Microencapsulation of food ingredients. Leatherhead International Limited, Leatherhead, United Kingdom.

Rocha,S.C.S., and O.P.Taranto. 2009. Advances in spouted bed drying of foods. p. 153-186. *In* C.Ratti (ed.) Advances in food dehydration. CRC Press Taylor & Francis Group, Boca Raton, FL.

Runge,F.E. 2004. Multiple-core encapsulation: encapsulation materials. p. 133-144. *In* P.Vilstrup (ed.) Microencapsulation of food ingredients. Leatherhead International Limited, United Kingdom.

Shahidi,F., andX.Q.Han. 1993. Encapsulation of Food Ingredients. Critical Reviews in Food Science and Nutrition 33:501-547.

Shahidi,F., andU.N.Wanasundara. 1998. Omega-3 fatty acid concentrates: nutritional aspects and production technologies. Trends in Food Science & Technology 9:230-240.

Shaw,L.A., D.J.McClements, and E.A.Decker. 2007. Spray-dried multilayered emulsions as a delivery method for omega-3 fatty acids into food systems. Journal of Agricultural and Food Chemistry 55:3112-3119.

Sijtsma,L., andM.E.de Swaaf. 2004. Biotechnological production and application of the ù-3 polyunsaturated fatty acid docosahexaenoic acid. Appl Microbial Biotechnol 64:146-153.

Thies,C. 1987. Microencapsulation. p. 724-745. *In* H.F.Mark, N.M. Bikales, C.G. Overberge, G. Menges, and J.I. Kroschwitz (ed.) Encyclopedia of polymer science and engineering. John Wiley & Sons, New York.

Thies,C. 2004. Microencapsulation: what it is and purpose. p. 1-30. *In* Vilstrup P (ed.) Microencapsulation of food ingredients. Leatherhead International Limited, Leatherhead, United Kingdom.

Trautwein,E.A. 2001. n-3 Fatty acids - physiological and technical aspects for their use in food. European Journal of Lipid Science and Technology 103:45-55.

Turton,R., G.I.Tardos, and B.J.Ennis. 1999. Fluidized bed coating and granulation. p. 331-434. *In* W.C.Yang (ed.) Fluidization, Solids Handling and Processing. Noyes Publications, Westwood, NJ.

Wallace,J.M.W., A.J.Mccabe, P.J.Robson, M.K.Keogh, C.A.Murray, P.M.Kelly, G.Marquez-Ruiz, H.McGlynn, W.S.Gilmore, and J.J.Strain. 2000. Bioavailability of n-3 polyunsaturated fatty acids (PUFA) in foods enriched with microencapsulated fish oil. Annals of Nutrition and Metabolism 44:157-162.

Young,S.L., X.Salda, and M.Rosenberg. 1993. Microencapsulating properties of whey protein. 1. Microencapsulation of anhydrous milk fat. Journal of Dairy Science 76:2868-2877.

Zank,J., M.Kind, and E.U.Schlunder. 2001. Particle growth in a continuously operated fluidized bed granulator. Drying Technology 19:1755-1772.