

Part I

# **New technologies for functional food manufacture**

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# **1 Microencapsulation in functional food product development**

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## **1.1 Introduction**

Functional foods provide health benefits over and above normal nutrition. Functional foods are different from medical foods and dietary supplements, but they may overlap with those foods developed for special dietary uses and fortified foods. They are one of the fastest growing sectors of the food industry due to increasing demand from consumers for foods that promote health and well-being (Mollet & Lacroix 2007). The global functional food market, which has the potential to mitigate disease, promote health and reduce health care costs, is expected to rise to a value of US\$167 billion by 2010, equating to a 5% share of total food expenditure in the developed world (Draguhn 2007).

Functional foods must generally be made available to consumers in forms that are consumed within the usual daily dietary pattern of the target population group. Consumers expect functional foods to have good organoleptic qualities (e.g. good aroma, taste, texture and visual aspects) and to be of similar qualities to the traditional foods in the market (Klont 1999; Augustin 2001; Kwak & Jukes 2001; Klahorst 2006). The demand for bioactive ingredients will continue to grow as the global market for functional foods and preventative or protective foods with associated health claims continues to rise. Over the last decade, there has been significant research and development in the areas of bioactive discovery and development of new materials, processes, ingredients and products that can contribute to the development of functional foods for improving the health of the general population.

New functional food products launched in the global food and drinks market have followed the route of fortification or addition of desirable nutrients and bioactives including vitamins, minerals, antioxidants, omega-3 fatty acids, plant extracts, prebiotics and probiotics, and fibre enrichments. Many of these ingredients are prone to degradation and/or can interact with other components in the food matrix, leading to loss in quality of the functional food products. To overcome problems associated with fortification, the added bioactive ingredient should be isolated from environments that promote degradation or undesirable interactions. This may be accomplished by the use of microencapsulation where the sensitive bioactive is packaged within a secondary material for delivery into food products. This chapter covers the microencapsulation of food components for use in functional food product formulations and how these components can be utilised to develop commercially successful functional foods.

**Table 1.1** Food ingredients that have been microencapsulated

Types of ingredients
Flavouring agents (including sweeteners, seasonings and spices)
Acids, bases and buffers (e.g. citric acid, lactic acid and sodium bicarbonate)
Lipids (e.g. fish oils, milk fat and vegetable oils)
Enzymes (e.g. proteases) and microorganisms (e.g. probiotic bacteria)
Amino acids and peptides
Vitamins and minerals
Antioxidants
Polyphenols
Phytonutrients
Soluble fibres

## 1.2 Microencapsulation

Microencapsulation is a process by which a core, i.e. bioactive or functional ingredient, is packaged within a secondary material to form a microcapsule. The secondary material, known as the encapsulant, matrix or shell, forms a protective coating or matrix around the core, isolating it from its surrounding environment until its release is triggered by changes in its environment. This avoids undesirable interactions of the bioactive with other food components or chemical reactions that can lead to degradation of the bioactive, with the possible undesirable consequences on taste and odour as well as negative health effects.

It is essential to design a microencapsulated ingredient with its end use in mind. This requires knowledge of (1) the core, (2) the encapsulant materials, (3) interactions between the core, matrix and the environment, (4) the stability of the microencapsulated ingredient in storage and when incorporated into the food matrix and (5) the mechanisms that control the release of the core. Table 1.1 gives examples of cores that have been microencapsulated for use in functional food applications. The molecular structure of the core is usually known. However, information is sometimes lacking on how the core interacts with other food components, its fate upon consumption, its target site for action and in the case of a bioactive core, sometimes its function in the body after ingestion may also be unclear (de Vos *et al.* 2006).

### 1.2.1 Encapsulant materials

Depending on the properties of the core to be encapsulated and the purpose of microencapsulation, encapsulant materials are generally selected from a range of proteins, carbohydrates, lipids and waxes (Table 1.2), which may be used alone or in combination. The materials chosen as encapsulants are typically film forming, pliable, odourless, tasteless and non-hygroscopic. Solubility in aqueous media or solvent and/or ability to exhibit a phase transition, such as melting or gelling, are sometimes desirable, depending on the processing requirements for production of the microencapsulated ingredient and for when it is incorporated into the food product. Other additives, such as emulsifiers, plasticisers or defoaming agents, are sometimes included in the formulation to tune the final product's characteristics. The encapsulant material may also be modified by physical or chemical means in order to achieve the desired functionality of the microencapsulation matrix. The choice of encapsulant material is therefore dependent on a number of factors, including its physical and chemical

**Table 1.2** Materials that have been used as encapsulants for food application

Encapsulant materials		
Carbohydrates	Proteins	Lipids and waxes
Native starches	Sodium caseinate	Vegetable fats and oils
Modified starches	Whey proteins	Hydrogenated fats
Resistant starches	Isolated wheat proteins	Palm stearin
Maltodextrins	Soy proteins	Carnauba wax
Dried glucose syrups	Gelatins	Bees wax
Gum acacia	Zein	Shellac
Alginates	Albumin	Polyethylene glycol
Pectins		
Carrageenan		
Chitosan		
Cellulosic materials		
Sugars and derivatives		

properties, its compatibility with the target food application and its influence on the sensory and aesthetic properties of the final food product (Brazel 1999; Gibbs *et al.* 1999).

The ability of carbohydrates to form gels and glassy matrices has been exploited for microencapsulation of bioactives (Reineccius 1991; Kebyon 1995). Starch and starch derivatives have been extensively used for the delivery of sensitive ingredients through food (Shimoni 2008). Chemical modification has made a number of starches more suitable as encapsulants for oils by increasing their lipophilicity and improving their emulsifying properties. Starch that was hydrophobically modified by octenyl succinate anhydride had improved emulsification properties compared to the native starch (Bhosale & Singhal 2006; Nilsson & Bergenståhl 2007). Acid modification of tapioca starch has been shown to improve its encapsulation properties for  $\beta$ -carotene, compared to native starch or maltodextrin (Loksuwan 2007). Physical modification of starches by heat, shear and pressure has also been explored to alter its properties (Augustin *et al.* 2008), and the modified starch has been used in combination with proteins for microencapsulation of oils (Chung *et al.* 2008).

Carbohydrates used for microencapsulation of  $\beta$ -carotene, from sea buckthorn juice, by ionotropic gelation using furcellaran beads, achieved encapsulation efficiency of 97% (Laos *et al.* 2007). Interest in using cyclodextrins and cyclodextrin complexes for molecular encapsulation of lipophilic bioactive cores is ongoing, especially in applications where other traditional materials do not perform well, or where the final application can bear the cost of this expensive material. The majority of commercial applications for cyclodextrins have been for flavour encapsulation and packaging films (Szente & Szejtli 2004).

Proteins are used as encapsulants because of their excellent solubility in water, good gel-forming, film-forming and emulsifying properties (Kim & Moore 1995; Hogan *et al.* 2001). Protein-based microcapsules can be easily rehydrated or solubilised in water, which often results in immediate release of the core. Proteins are often combined with carbohydrates for microencapsulation of oils and oil-soluble components. In the manufacture of encapsulated oil powders, encapsulation efficiency was higher when the encapsulation matrix was a mixture of milk proteins and carbohydrates, compared to when protein was used alone (Young *et al.* 1993). Soy protein-based microcapsules of fish oil have been cross-linked using transglutaminase to improve the stability of the encapsulated fish oil (Cho *et al.* 2003). Protein-based hydrogels are also useful as nutraceutical delivery systems (Chen *et al.* 2006).

The release properties of protein-based hydrogels and emulsions may be modulated by coating the gelled particles with carbohydrates. A model-sensitive core, paprika oleoresin, was encapsulated in microspheres of whey proteins and coated with calcium alginate to modify the core's release properties (Rosenberg & Lee 2004). Whey protein-based hydrogels with an alginate coating altered the swelling properties of the gelled particles. The stability of these particles was increased at neutral and acidic conditions both in the presence and absence of proteolytic enzymes (Gunasekaran *et al.* 2007).

Lipids are generally used as secondary coating materials applied to primary microcapsules or to powdered bioactive cores to improve their moisture barrier properties (Wu *et al.* 2000). Lipids can also be incorporated in an emulsion formulation to form a matrix or film around the bioactive core (Crittenden *et al.* 2006).

The increasing demand for food-grade materials that will perform under the different stresses encountered during food processing has spurred the development of new encapsulant materials. Understanding the glass transition temperature of various polymers (e.g. proteins and carbohydrates) and their mixtures is also becoming important as this can influence the stability of the encapsulated core. The low water mobility and slow oxygen diffusion rates in glassy matrices can improve stability of bioactives (Porzio 2003). It is possible to exploit thermally induced interactions between proteins and polysaccharides and then to use the modified materials for encapsulation. Hydrogels formed by heat treatment of  $\beta$ -lactoglobulin – chitosan have been investigated, and it has been suggested that under controlled conditions these complexes may be useful for microencapsulation of functional food components (Hong & McClements 2007). Maillard reaction products formed by interactions between milk proteins and sugars or polysaccharides have been used as encapsulating matrices to protect sensitive oils and bioactive ingredients (Sanguansri & Augustin 2001).

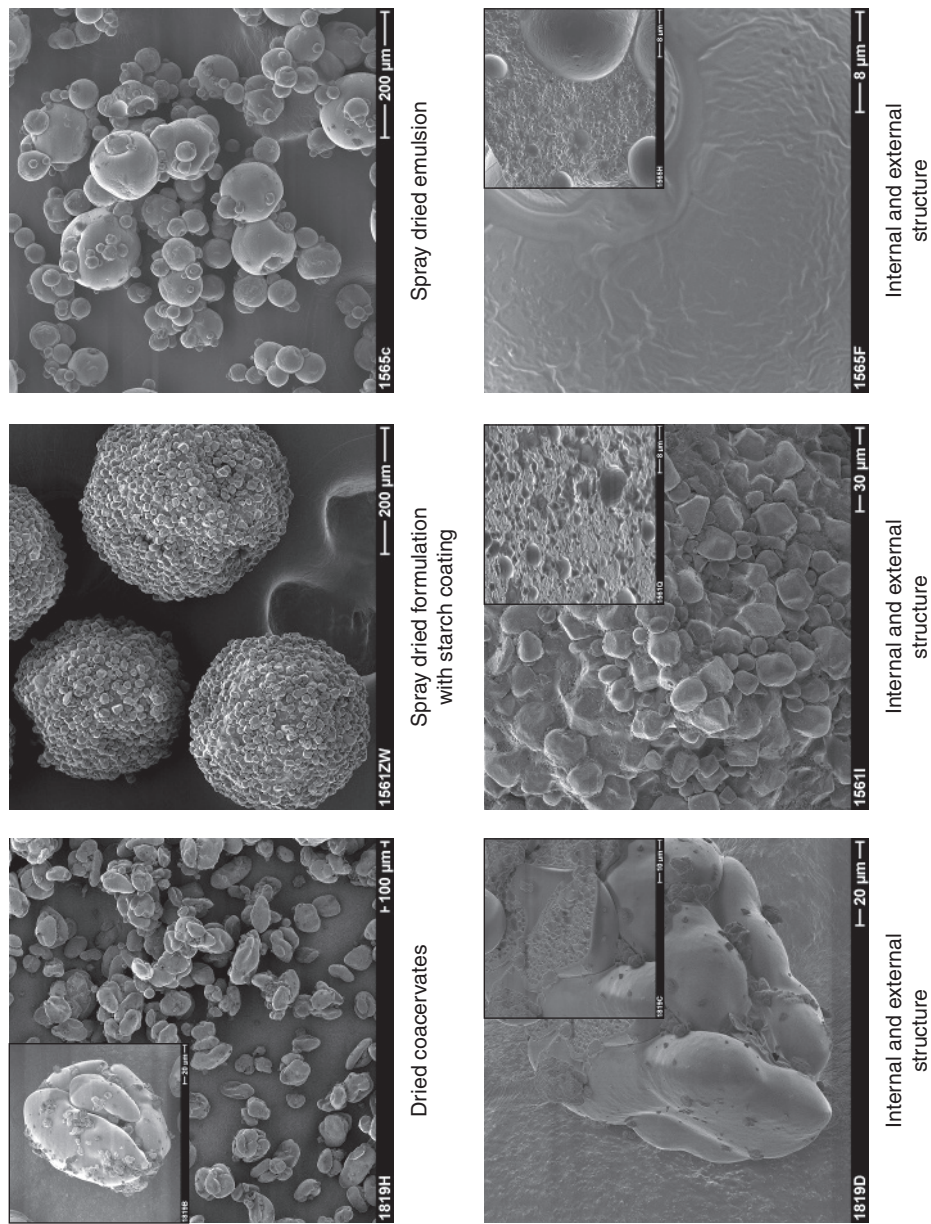
### 1.2.2 Microencapsulation processes

Microencapsulation processes traditionally used to produce a range of microencapsulated food ingredients are listed in Table 1.3. A number of reviews give further details on microencapsulation technology in the food industry (Augustin *et al.* 2001; Gouin 2004). The choice of method used for microencapsulation depends on the properties of the core, the encapsulant materials and the requirements of the target food application. Figure 1.1 shows the structure of microencapsulated oil produced using three different microencapsulation processes.

Methods used for microencapsulation in the food industry have generally been adapted from technologies originally developed for the pharmaceutical industry. Mechanical processes use commercially available equipment to create and stabilise the microcapsules,

**Table 1.3** Encapsulation processes used in the food industry

<b>Mechanical processes</b>	<b>Chemical processes</b>
Emulsification	Ionotropic gelation
Spray-drying	Simple coacervation
Fluidised-bed coating	Complex coacervation
Centrifugal extrusion	Solvent evaporation
Spinning disk	Liposomes
Pressure extrusion	Cyclodextrin complexation
Hot-melt extrusion	



**Fig. 1.1** Structure of microencapsulated oil produced using different microencapsulation processes.



whereas chemical processes capitalise on the possible interactions that can be promoted by varying the process conditions used to create the microcapsules.

Spray-drying is the most commonly used mechanical method for microencapsulation of bioactive food ingredients. Gharsallaoui *et al.* (2007) reviewed the use of spray-drying for the microencapsulation of food ingredients. It is efficient and cost-effective and uses unit processes and equipment readily available in most food processing plants. Spray-dried ingredients have reasonably good powder characteristics and good stability. Fluidised-bed coating is another mechanical process used for encapsulation of dry bioactive cores and ingredients. It consists of spraying an aqueous or solvent-based liquid coat onto the particles followed by drying. Dry particle coating of bioactive cores is an adaptation of the fluidised-bed coating technique that has been investigated by Ivanova *et al.* (2005) for microencapsulation of water-sensitive ingredients. The dry particle coating method avoids the use of aqueous or solvent-based coatings.

Of the different chemical microencapsulation processes available, only gelation and coacervation are widely used in the food industry. All current chemical methods are batch processes, although there is significant effort going into the development of continuous processes. Biopolymer–biopolymer supramolecular structures as complexes and coacervates may be formed under conditions where the two biopolymers carry opposite charges. These structures may have potential for controlled release and delivery of bioactives in foods (Turgeon *et al.* 2007; Livney 2008). The formation of native whey protein isolate-low methoxy pectin complexes by electrostatic interaction has potential for entrapment of water-soluble ingredients in acidic foods, as demonstrated by the entrapment of thiamine by Bedie *et al.* (2008).

Liquid emulsions may also be used as delivery systems in foods (Appelqvist *et al.* 2007; McClements *et al.* 2007). Oil-in-water emulsions are suitable for the delivery of lipids and lipid-soluble bioactives. Kinetically stable oil-in-water emulsions are made by homogenising a mixture of either an oil or an oil containing a lipid-soluble bioactive, with an aqueous solution containing the encapsulating material. Spontaneously formed, thermodynamically stable microemulsions may also be loaded with nutraceuticals and used as delivery systems. Garti and Amar (2008) have discussed the importance of understanding the nature of the microstructures and phase transitions in micro- and nanoemulsions for the effective delivery of nutraceuticals. Leal-Calderon *et al.* (2007) highlighted the need to understand the formulation and the design and characterisation of structured emulsions in order to control the release of bioactives in foods when ingested. Guzey and McClements (2006) explored ways of improving the release characteristics of conventional primary emulsions for controlled or triggered release delivery systems of bioactives, by developing multilayered emulsion formulations. Preparation of water-in-oil-in-water (w/o/w) emulsions by membrane filtration was explored by Shima *et al.* (2004) to encapsulate a model hydrophilic bioactive, with a view to protecting functional food ingredients for controlled release application.

### 1.2.3 Drivers for microencapsulation

The primary reasons for microencapsulation of food ingredient are to (1) protect the core from degradation during processing and storage, (2) facilitate or improve handling during the production processes of the final food application and (3) control release characteristics of the core, including its delivery to the desired site after ingestion.

Many bioactives (e.g. omega-3 oils, carotenes and polyphenols) need to be protected against degradation. For example, omega-3 oils are very susceptible to oxidation, leading to



the development of off-flavours and off-odours. Microencapsulation protects the sensitive oils from exposure to oxygen, light and metal ions during processing and storage (Sanguansri & Augustin 2006). Protecting the core from degradation and from interactions with other food components can extend the shelf stability of the ingredient itself, as well as that of the final food product to which it is added.

Microencapsulation can facilitate or improve handling of ingredients during production processes used in the final applications. The conversion of a liquid ingredient into a powder offers significant convenience, as it is much easier to store, weigh and add a powdered ingredient, compared to its liquid version. Microencapsulation can aid in the addition and more uniform blending of bioactive ingredients into a food formulation. Bioactive ingredients in their pure or very concentrated forms are usually added in very low amounts (sometimes at ppm levels). Addition of a few milligrams or grams of ingredients into hundreds of kilos or tonnes of products can lead to uneven distribution within the food matrix, especially when ingredients are dry-blended. Microencapsulated forms with much lower payload can be used in these applications to facilitate a more homogeneous blending of these highly potent bioactive ingredients into food because the lower payloads provide a larger amount of the microencapsulated ingredient to be added to the mix into which it has to be blended.

Bioactive ingredients may require microencapsulation to improve or modify their functionality and release characteristics. Understanding the core, the final application, and the mechanism required to release the core is essential for effective design of the microcapsule's release characteristics. Different release characteristics can be achieved depending on the requirement, e.g. controlled, sustained or delayed release. Bioactive ingredients are often known to possess undesirable tastes and/or odours that require masking before they can be used successfully in food formulations. A significant challenge associated with nutraceutical ingredients is the need to mask bitterness and aftertaste (Anon 2006). With new developments in understanding the science of taste, the introduction of new bitterness blockers and sweetness potentiators in food formulations (McGregor 2004) can be combined within a microencapsulation system to allow controlled, delayed or sustained release of bioactives.

During the addition of bioactive ingredients into food, it is essential that both the bioactivity and the bioavailability are maintained to ensure that the bioactives achieve the desired function in the body. When direct addition of the bioactive could compromise its bioavailability, it needs to be protected by microencapsulation. The protection of the core from the acidic pH of the stomach during transit through the gastrointestinal (GI) tract may potentially enable more efficient delivery of the bioactive to the target site in the body and may also reduce the dosage required to achieve the health benefits.

Advances in the development of microencapsulation technology for food applications have been driven by the need for (1) the core to be encapsulated, (2) new and alternative materials that are cost-effective encapsulants and (3) materials which will withstand the processes widely used in the food industry. More recent developments in microencapsulation technologies for food applications have focused on applying the technology to more cost-effective food-grade encapsulant materials and processes available in the food industry. The need for controlled release and delivery of bioactive food ingredients to target sites in the body continues to drive other new developments. Converting stable microcapsule formulations (emulsions, dispersions, suspensions, coacervates) into powders is still the preferred option for production of microencapsulated bioactive ingredients, as it offers more convenience and flexibility. An understanding of how these formulations will behave during the drying process and on reconstitution is critical to the success of powdered preparations.

With the primary reason or purpose of microencapsulation being clearly identified, other important factors need to be seriously considered to ensure proper selection of encapsulant materials and processes that are cost-effective, practical and scalable. Important considerations during the development of microencapsulated products include (1) core properties – e.g. chemical structure, solubility and stability, (2) product format – e.g. liquid or powder format depending on final application, (3) physical properties of the microencapsulated ingredient – e.g. particle size, bulk density and colour, (4) payload – i.e. amount of bioactive loading in the microcapsule, (5) release trigger mechanism – e.g. dissolution, pressure, heat and shear, (6) storage conditions and shelf-life requirements – e.g. refrigerated or ambient storage and (7) legal and regulatory requirements for addition into food in the country of its application. From a commercial perspective, there is the additional factor of material and production costs and whether the final food product can bear the additional cost of using a microencapsulated ingredient.

### 1.3 Microencapsulated food ingredients

There are several technical challenges in developing functional ingredients for incorporation into foods. They must satisfy the sensory demands of the consumers and ensure that the bioactive can be delivered to specific sites in the GI tract to exert the desired health benefit. Microencapsulation has been applied to a number of food ingredients to develop them into tailor-made bioactive ingredients (Augustin & Sanguansri 2008).

The increasing number of microencapsulated food ingredient launches has been the result of more creative translation and adaptation of microencapsulation techniques originally developed in the pharmaceutical industries. New encapsulant materials and more cost-effective formulations and processes have enabled the food industry to develop these new ingredients with added value and functionality. In more recent years, the addition of microencapsulated ingredients into a wider range of food products ensures that it does not significantly affect the cost of the final food product. This is a significant issue as food has very low profit margins compared to pharmaceuticals.

#### 1.3.1 Vitamins and minerals

Fortification with vitamins and minerals is often challenging due to their susceptibility to degrade during processing and storage and to react with other components in the food system. Vitamins and minerals are generally sensitive to temperature, moisture, light and pH, and their potency is often compromised by their reaction with other ingredients or premature release.

Vitamins and minerals are added to a range of food products for the following reasons: (1) to replace those that are lost during processing and storage; (2) to meet special nutritional needs, e.g. for infants and elderly; and (3) to prevent disease in specific consumer or at-risk groups. Traditionally, higher levels than that are required in the end product have been added to overcome losses during processing and storage. These high overages may be avoided by using microencapsulated forms.

For water-soluble vitamins (e.g. vitamins B and C) and minerals (e.g. iron and calcium), spray-drying, spray chilling, fluidised-bed coating and spinning disk coating have been used to manufacture dry powder microcapsules. Where liquid microcapsule formats are

preferred, microencapsulation in liposomal delivery systems can be used. There is also the possibility of entrapping water-soluble vitamins in double emulsions. Fechner *et al.* (2007) demonstrated that vitamin B12 in the inner phase of an oil/water/oil emulsion stabilised by caseinate–dextran conjugates, instead of pure protein, reduced the release of the vitamin under acidic conditions. For lipid-soluble vitamins (e.g. vitamins A, D, E and K) and provitamin A ( $\beta$ -carotene), stable emulsion formulations or spray-dried emulsions are commonly used as delivery systems. Emulsion-based systems are often used for delivery of lipid-soluble bioactives (McClements *et al.* 2007). However, where there are specific interactions between hydrophobic bioactives and a protein, an aqueous-based system may be exploited. Semo *et al.* (2007) demonstrated that casein micelles were useful for delivery of vitamin D2.

Microencapsulation has benefits when used for delivery of iron and calcium in foods. Direct addition of iron into foods may reduce its bioavailability through interaction with tannins, phytates and polyphenols. Free iron is also known to catalyse the oxidation of fats, vitamins and amino acids. These interactions can affect the sensory characteristics of the final food formulation, as well as decrease the nutritional value of the food due to iron-induced catalysis of deteriorative reactions. Many of these limitations of direct addition of iron may be overcome by microencapsulation. Other microencapsulation technologies used for encapsulation of iron include liposomal delivery systems and application of lipid coats by fluidised-bed coating (Xia & Xu 2005). Molecular inclusion of iron using cyclodextrins may also be used in its delivery (Leite *et al.* 2003).

The interaction of calcium with proteins can cause unwanted coagulation or precipitation of the protein, especially in calcium-fortified protein beverages. Calcium is naturally present in dairy products, but there is interest in fortifying other protein products with calcium, such as soy protein beverages. Calcium fortification of protein-based beverages may be achieved with the addition of calcium-chelating agents; however, this may result in an undesirable taste when high levels of calcium fortification are desired. Microencapsulation of calcium can prevent its negative interaction with other food components (e.g. soy proteins) in the food environment. A liposomal delivery system has also been examined for this application (Hirotsuka *et al.* 1984).

### 1.3.2 Functional fatty acids

Functional fatty acids, particularly docosahexaenoic acid, eicosapentaenoic acid,  $\alpha$ -linolenic acid and conjugated linoleic acid, have attracted significant attention due to their potential health benefits (Ohr 2005). Emulsion-based technologies and spray-drying are currently the most common approaches employed for microencapsulation and delivery of functional fatty acids into food (Sanguansri & Augustin 2001; McClements *et al.* 2007).

Omega-3 fatty acids are highly susceptible to oxidation and have an inherent fishy taste and odour. Therefore, most food applications of omega-3 fatty acids require microencapsulation for protection from oxidation and to mask the fishy taste and odour. Significant research has been carried out on microencapsulation of omega-3 fatty acids. An increasing number of food companies are developing new functional food products containing omega-3 fatty acids. This increase in the number of food products launched containing omega-3 fatty acids has also been driven by the qualified health claims that were allowed by Food and Drug Administration (FDA) in 2004. Technologies that have been successfully used to encapsulate omega-3 oils include emulsification and spray-drying (Sanguansri & Augustin 2001), coacervation (Wu *et al.* 2005), cyclodextrin complexation and liposomal preparations (Tanouchi *et al.* 2007).

### 1.3.3 Probiotics

Probiotics are live microorganisms that must remain alive during processing, storage and gastric transit to fulfil their desired function in the body (Mattila-Sandholm *et al.* 2002). Much clinical data have been accumulated to support the role of probiotics in human health by benefiting the immune system, strengthening the mucosal barrier and suppressing intestinal infection (Saarela *et al.* 2002). This has driven interest in adding probiotics to a wider range of food products, other than traditional fermented dairy products such as yoghurt. As probiotics are sensitive to heat and moisture, keeping them alive during food processing and storage is not easy. Even in fermented dairy product applications, the survival of probiotics during storage still remains a challenge for the industry.

Processes that have been used to encapsulate probiotics include spray coating, spray-drying, extrusion, emulsification and gel particle technologies. Of these technologies, the technique most widely investigated by researchers involves the use of polysaccharides to form gelled particles (Krasaekoopt *et al.* 2003; Anal & Singh 2007). However, the use of gelled particles for microencapsulation of probiotics has not been widely adopted by commercial companies, as it is a batch process. The use of alginate–chitosan microcapsules has also been explored to improve the mechanical strength of the capsules to survive *in vitro* digestion (Urbanska *et al.* 2007). The application of a lipid coating by a fluid-bed technique has also been used for probiotic encapsulation (Lee & Richardson 2004). Probiotics encapsulated in lipid-based materials are used, in a limited range of food products, with varying degrees of success. The application of high-melting-point lipids and waxes allows protection of probiotics from high-moisture environments and thermal protection below the melting point of the coat. Starch-based encapsulation was also explored by Lahtinen *et al.* (2007), but their results showed no effect on improving the viability of *Bifidobacterium longum* strains.

Spray-drying has always been an attractive process for production of powdered food ingredients because it is a continuous, high-volume and cost-effective process. A number of researchers have explored spray-drying for production of probiotic microcapsules with varying degrees of success (Desmond *et al.* 2002; Ananta *et al.* 2005; Anal & Singh 2007; Su *et al.* 2007). The most important step still remains the selection and formulation of an encapsulant that can protect the probiotics during drying. A novel microencapsulation technology using protein–carbohydrate conjugate in the matrix provided significant protection to probiotic bacteria during spray-drying, during exposure to acidic pH and during non-refrigerated storage at low to intermediate water activity (Crittenden *et al.* 2006). The use of appropriate materials and process conditions applied during microencapsulation has the potential to enable the addition of probiotics to a much wider range of food products with intermediate water activity which do not require refrigeration.

### 1.3.4 Phytochemicals

Phytochemicals are biologically active plant chemicals, with increasing evidence that they can reduce the risk of chronic diseases (Hasler 1998). Ingredients claimed to be rich in phytochemicals are extracted from plant sources. Once isolated from their natural environment, these bioactive ingredients generally require microencapsulation to stabilise the active component and mask undesirable tastes, colours and odours. The phytochemicals of interest to the food industry include phytosterols, tocopherols, carotenoids, coenzyme Q10, curcumin, garlic extracts and polyphenols (e.g. resveratrol).

Resveratrol is a naturally occurring non-flavonoid polyphenolic compound present in plants such as grapes, berries and peanuts (Halls & Yu 2008), as well as in cocoa and chocolate

(Counet *et al.* 2006). Resveratrol is photosensitive and benefits from microencapsulation to maintain its stability when added to food products. Shi *et al.* (2008) have shown that encapsulation of resveratrol in yeast cells can offer protection and enhance its stability as an ingredient. The use of chitosan–alginate coacervates as an encapsulant has also exhibited potential for preparation of encapsulated powder ingredients from aqueous (water-soluble) antioxidant plant extracts (Deladino *et al.* 2008).

The use of natural fruit fibres as encapsulating agents for the microencapsulation and spray-drying of sticky bioactive extracts (*Hibiscus sabdariffa*) has been explored by Chiou and Langrish (2007). Extracts containing curcumin have been encapsulated using commercially available lecithin to form liposomes by homogenisation or microfluidisation (Takahashi *et al.* 2007). The delivery of curcumin through oil-in-water nanoemulsions has been shown to enhance its anti-inflammatory activity in animal tests (Wang *et al.* 2008). Szente *et al.* (1998) demonstrated that the stability of curcumin and carotenes is enhanced by molecular encapsulation using cyclodextrins.

### 1.3.5 Proteins, amino acids, peptides and enzymes

Proteins have traditionally been encapsulated for pharmaceutical applications (Putney 1998). The demand for more protein in food and beverages is on the rise (Sloan 2004). Whey, casein and soy proteins are commonly used in high-protein food formulations either in their native or hydrolysed forms.

Protein-derived peptides and amino acids are also being isolated from their source to enable addition at the correct dosage required for physiological health functions. The direct addition of these components into food and beverage formulations can result in an undesirable bitter taste and astringency. Encapsulation of casein hydrolysates in lipospheres has been found to reduce the bitterness (Barbosa *et al.* 2004).

Encapsulation may also be used to preserve the activity of enzymes. Components in garlic have also been shown to offer beneficial health effects (Gorinstein *et al.* 2007), and microencapsulation of garlic powder results in protection of alliinase activity, which improves the ratio of alliin to allicin conversion under *in vitro* conditions (Li *et al.* 2007).

### 1.3.6 Fibre

The trend of adding dietary fibres to food and beverage formulations that traditionally do not contain these fibres is increasing due to the increasing evidence of health benefits of high-fibre diets. Examples of dietary fibres for which the FDA has allowed health claims are  $\beta$ -glucan from oats and psyllium fibre.  $\beta$ -Glucan, a cholesterol-lowering soluble fibre, shown to reduce the risk of heart disease was allowed an FDA health claim in 1997. Later, in 1998, the FDA extended the health claim for soluble fibre to psyllium fibre. Other dietary fibres added to food and beverage formulations include indigestible gums, polysaccharides, oligosaccharides and lignins (Prosky 1999).

High levels of fibre need to be added in the final food formulation in order to make a health claim. The problems associated with the addition of high levels of dietary fibres to food and beverages are the unpalatability of the high-fibre ingredients and the significant effects they have on the viscosity of the final product. This has resulted in the development of expensive, refined fibre ingredients, e.g. polydextrose (Sunley 1998). Microencapsulation can minimise palatability problems as well as minimise water absorption during formulation and processing. Much cheaper sources (e.g. indigestible gums) can also be added at a much higher

levels if the fibre in food formulations is encapsulated with materials that can reduce hydration and water absorption during processing. Chito-oligosaccharide, as a functional ingredient, offers a range of health benefits; however, direct addition to milk can affect its flavour and colour. Microencapsulation of chito-oligosaccharide with polyglycerol monostearate, as explored by Choi *et al.* (2006), reduced its adverse effects on the physicochemical or sensory properties when added to milk.

## **1.4 Development of microencapsulated ingredients**

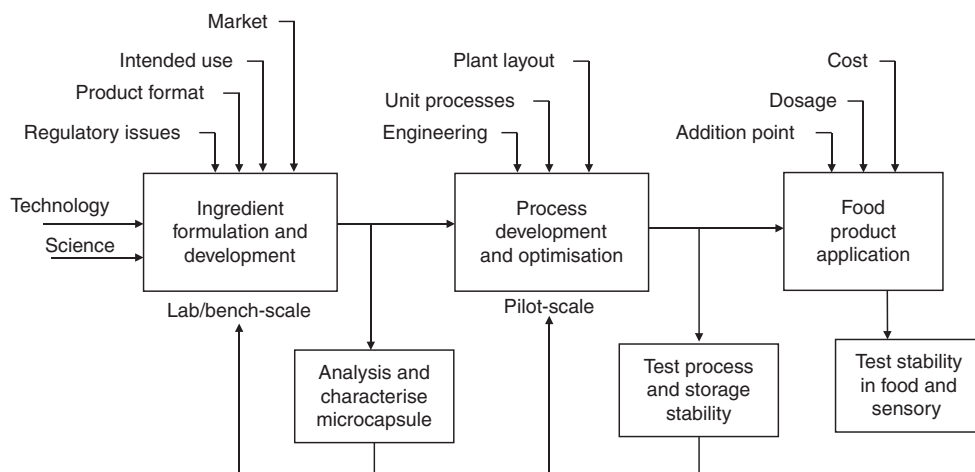
### **1.4.1 The approach**

During the development of functional foods using microencapsulated food ingredients, the selection of ingredients and processes was traditionally based on empirical approaches. Ubbink and Kruger (2006) have suggested that an alternative concept is to use a retro-design approach that relies more on a fundamental understanding of the required performance of the ingredient in a complex food environment. This approach encompasses an understanding of the effects of processing and the factors controlling the chemical and physical events that govern the stability and release properties of a microencapsulated product; however, the test of whether a microencapsulation system is suitably tailored for its end product application is its acceptance in the marketplace. The route from concept to acceptance of functional foods by consumers has many stages and requires input from scientists, technologists, nutritionists and an understanding of the regulatory processes (Jones & Jew 2007). Our own program of research in designing microencapsulated ingredients has utilised multidisciplinary expertise, involving chemistry, physics, food science and process engineering, with the regulatory and market requirements in mind to minimize or avoid issues during scale-up and commercialisation. This approach ensures that both the food and ingredient manufacturers' requirements are met while consumers' demands are also considered during the development. The final product application must be the focus of the microencapsulated product development in order that the core is protected from various stresses during incorporation into the final product. It is important to ensure that when microencapsulation is used to deliver active ingredients into foods, it provides a simple, efficient and cost-effective solution compared to direct addition of bioactives.

### **1.4.2 Product and process developments**

Understanding the fundamental science of the core, as well as a good knowledge of the materials and processes available, is a requirement for the process of developing a successful product. The stages in the development of a microencapsulated product from bench scale product concept to a commercial product acceptable to consumers in final food applications are shown in Figure 1.2. In designing a cost-effective and tailor-made microcapsule suitable for its intended use (i.e. the final food product application), the final product format (liquid or dry) and the market (size and value) need to be identified at the outset. These factors will significantly influence the choice of materials, formulation and process that can be employed. At this stage, the physical performance and characteristics, core stability and possible interactions with other ingredients during formulation and process should be tested. A few iterations of changes to the initial formulation may be required until reasonable product





**Fig. 1.2** Microencapsulated product development process.

properties are achieved at the laboratory scale. Once the formulation and desirable product properties are established, the next step is to develop a scalable process.

When considering processes for manufacture of microencapsulated food ingredients, the ability to use standard unit processes available in a conventional food processing operation is desirable. Their use will minimise future problems and assist in the commercial scale-up production of the microcapsules. During the scaling up of the process, the product specifications of the microencapsulated ingredient need to be clearly defined, as this will dictate the type of equipment and process conditions used during manufacture. For a powdered microencapsulated ingredient, these include the colour, particle size, bulk density, moisture content, payload, sensory aspects and other physical characteristics required in the final application. For a liquid (emulsion) microencapsulated ingredient, these include total solids concentration, viscosity, colour or clarity (if required), particle size, storage conditions and stability requirements. Sensory evaluation and storage stability trials of the final microencapsulated product need to be carried out during scale-up to assess consumer acceptability. Some minor formulation and process optimisation may be required at this final stage to achieve a product with the least production costs.

During scale-up, the final product performance during processing, the stability of the core and of the microcapsule under different processing conditions need to be fully established to define the conditions and the stage of addition during the manufacture of the final food application. The long-term stability of the microencapsulated ingredient itself also needs to be established to ensure that the ingredient stability equals or exceeds that of the final food product to which it is being added.

## 1.5 Delivery of microencapsulated ingredient into functional foods

### 1.5.1 Functional food product development

Diet has been a major focus of public health strategies aimed at maintaining optimum health throughout life stages. Nutrients and bioactive compounds (also called nutraceutical



ingredients) which have shown potential in preventing or ameliorating the effect of major diseases (e.g. some types of cancer, cardiovascular disease, neurodegenerative disease and eye disorders) have driven the interest in developing functional foods for special health and dietary uses. The FDA's authorisation of qualified health claims for a number of ingredients, when used at specific levels, has helped accelerate the market for functional foods and to raise consumer awareness of several nutraceutical ingredients, e.g. omega-3 fatty acids, dietary fibre, plant sterols and soy protein. Microencapsulation technologies, through the use of appropriate formulations and processing strategies, have the potential to deliver a single bioactive or a cocktail of bioactives (Champagne & Fustier 2007). Functional food product launches with specific target health categories have continued to increase in the last decade. Functional health claims have been primarily focused on gut health, heart health, immune function, bone health and weight management.

Functional food ingredients designed to enhance GI tract health include probiotics, dietary fibre and prebiotics, and bioactive plant metabolites (e.g. phytochemicals such as polyphenols). Some of these ingredients have a role in gut fermentation, and by influencing the microflora composition and fermentation metabolites, they consequently contribute to both local and systemic effects in the body (Puupponen-Pimia *et al.* 2002). Other bioactive ingredients, such as fish oil (omega-3), polyphenols (resveratrol) and short-chain fatty acids (butyric acid), have been investigated and shown to be beneficial for gut health and as chemoprotective and chemopreventive agents against colon cancer (Schneider *et al.* 2000; Dwivedi *et al.* 2003; Orchel *et al.* 2005; Stehr & Heller 2006; Athar *et al.* 2007). The benefits of these gut health-promoting ingredients may be more effectively utilised by the general population if they are added into food products without affecting their shelf-life and sensory properties.

Microencapsulation has been used to assist the delivery of these ingredients into food, to stabilise and control their release during GI transit and to enhance their desired function in the body. A microencapsulation technology has been developed to protect these bioactives during processing and storage, as well as to target the release of the bioactive to specific sites in the GI tract (Augustin *et al.* 2005).

Heart health has been a major emphasis for many new products around the globe. As consumers continue to look for more ways to lower cholesterol and lessen their risk of heart-related illnesses, food manufacturers have continued to develop functional food products for this category. Dairy, beverage and bakery products are the top three categories with the addition of plant sterols, omega-3 fatty acids, peptides and whole grains being just a few examples of ingredient focus in heart-healthy food product developments.

Of the mainstream functional food product categories available commercially, dairy products accounted for about 40% of total functional food sales, followed by cereal products, beverages, fats and oils, soya products, bakery, eggs, and others (Watson *et al.* 2006). In this respect, where the consumption of functional foods is promoted as a fundamental way to proactively prevent or delay the onset of the disease, the ability to target the release and delivery of the bioactives to a specific site in the body and the bioavailability of the nutrients or bioactive compounds when they are released at the target site are more important than the amount originally present in the food (Parada & Aguilera 2007).

Microencapsulation is a logical solution for delivery of bioactives into functional foods as it can protect the bioactive during GI transit, until it reaches the target site in the body, as well as enhance its bioavailability when it is released. It also offers other advantages such as reduced dosage and overages during formulation, resulting in reduced ingredient cost during production.

### 1.5.2 Major food categories

Successful functional food product development in mainstream food categories requires special consideration as there is usually little room for reformulation and process modification as a result of adding the new active ingredient. This means that the ingredients used in the production of the microencapsulated ingredient must already be on the product label, and the microencapsulated ingredient must survive the processes that the product has to go through without affecting its sensory properties.

#### 1.5.2.1 Dairy products

Functional dairy products account for 42.9% of the functional food market (Watson *et al.* 2006). Dairy products have been the most popular delivery vehicles for a number of functional and healthy ingredients, from vitamin and mineral fortification to addition of bioactives to promote health benefits. As milk and dairy products are a normal part of our daily diet, in all life stages, any new product launched can be expected to gain some market share. Much higher levels of vitamins and minerals have been added to dairy products in recent years. Omega-3 fatty acid fortification has also been popular despite the challenges in achieving acceptable flavour profiles in the final product. Addition of chito-oligosaccharide to milk has also been investigated by Choi *et al.* (2006).

#### 1.5.2.2 Cereal products

Healthy bars and cereal products account for 19.4% of the functional food market (Watson *et al.* 2006). This category is the second most popular delivery vehicle in a number of functional ingredients for a number of reasons, e.g. market size, convenient format, easier to add to formulations and presence of ingredients that can mask unpleasant flavours.

#### 1.5.2.3 Beverages

Functional beverages are the fastest growing product category for delivery of a range of functional ingredients. These currently account for 14.4% of the functional food and beverage market (Watson *et al.* 2006). The US market for fortified/functional beverages is expected to reach US\$29 billion for standard beverages and US\$815 million for dairy beverages by 2011 (Fuhrman 2007). Vitamin- and mineral-enriched drinks (e.g. with added calcium and vitamin C) are among the most popular, followed by weight-control beverages with added protein.

#### 1.5.2.4 Fats and oils products

The fats and oils market accounts for 11.8% of the functional food market (Watson *et al.* 2006). In 2005, the global omega-3 ingredient market was worth over US\$700 million (Haack 2007), and by 2010, the global market for omega-3 oils is expected to be worth US\$1.2 billion (Lavers 2007). The development of spreads with cholesterol-lowering phytosterols, healthy oils, healthy spreads, sauces and dips with added nutraceutical ingredients is also increasing.

#### 1.5.2.5 Bakery products

Bakery product launches containing functional ingredients account for about 1.7% of the functional food market (Watson *et al.* 2006); however, the use of microencapsulated

ingredients in bakery products has applications beyond the addition of bioactive ingredients. Microencapsulated ingredients used for bakery applications include leavening agents, sweeteners, antimicrobial agents, dough conditioners and flavours. These ingredients are widely used in commercial baking operations where high volumes of dough and batter pre-mixes are prepared for further distribution. The development of microencapsulated ingredients for bakery applications has additional challenges, such as protection during high-shear and high-temperature processing. The coating materials used for bakery applications include fats and waxes. Processes used for bakery ingredient applications include hot-melt coating (fluid-bed technology), spray chilling and high-pressure congealing. New launches in functional bakery products have seen the addition of extra vitamins (vitamins A, C and E) and minerals (calcium and iron), long-chain polyunsaturated fatty acids (omega-3 and omega-6) and soluble fibres.

### 1.5.3 Factors affecting use of microencapsulation in the market

Success in translating research to commercial products has significant challenges, especially in stabilising and masking any undesirable tastes and odours of bioactive ingredients being added, as well as maintaining the overall sensory quality of the final food product (Hargreaves 2006). Microencapsulation has been employed as a technology that can minimise, if not solve, these challenges, and it also offers the possibility of developing tailor-made ingredients for specific applications. Important issues to consider for successful delivery of microencapsulated ingredients into commercial food products are shown in Table 1.4.

The trend of developing and using microencapsulated ingredients has increased significantly in the last decade as more cost-effective materials and production processes suitable for food applications have developed. Microencapsulated ingredients are used in functional food product formulations to improve nutritional content, to replace nutrients lost during processing (fortification) and to add other bioactive ingredients with known healthy benefits, without changing the sensory characteristics of the final food product.

## 1.6 Conclusion

Microencapsulation technology holds promise for the successful delivery of bioactive ingredients into functional foods, and has the potential to enhance the functionality of bioactive ingredients, thus maximising the health benefits available to consumers from these foods. Microencapsulation can offer significant advantages for improved delivery and protection of bioactive ingredients in food, which would not have been possible by direct addition.

New developments in a range of microencapsulation technologies continue to address different functionality challenges that occur when formulating bioactive ingredients into functional foods (Sunley 1998; Pszczola 2005). Opportunities for use of microencapsulation in the food industry continue to grow as greater demands are being made on the integrity of the capsules to control the release and delivery of the core material at a specific time during digestion and to a specified site in the body (Champagne & Fustier 2007). This often requires tailor-made microencapsulated ingredients that are fit for this purpose to be individually developed to take into account the final food application and format for delivery of the bioactive ingredients.

**Table 1.4** Important issues to consider for successful delivery of microencapsulated bioactives into commercial food products

Important issues	Action or questions to ask
Regulatory standard	<ul style="list-style-type: none"> <li>• Check the regulatory standards in each country for addition of bioactives.</li> <li>• Can the bioactive be added to the chosen food?</li> <li>• Are the ingredients used as encapsulants allowed in the chosen food?</li> <li>• What levels are required if there is to be a health claim?</li> </ul>
Food product application format	<ul style="list-style-type: none"> <li>• What is the format of the final product chosen? <ul style="list-style-type: none"> <li>◦ For a powder, blending applications require good control of particle size, moisture and bulk density</li> <li>◦ For a liquid, rehydration and redispersion behaviour of powdered encapsulated bioactives is important</li> </ul> </li> </ul>
Protection and release characteristics	<ul style="list-style-type: none"> <li>• What processing stresses has the ingredient to survive during incorporation into the food?</li> <li>• Under what conditions or in response to what trigger is the bioactive released?</li> </ul>
Stage or point of addition	<ul style="list-style-type: none"> <li>• Is the food manufacturing plant set-up automated?</li> <li>• At what point during production will the ingredient be added?</li> <li>• Will the ingredient be added using an automated process or manually?</li> </ul>
Interaction with other ingredients	<ul style="list-style-type: none"> <li>• Is there a need to avoid interaction of the bioactive with other ingredients in the final food during processing and storage?</li> </ul>
Final product characteristics	<ul style="list-style-type: none"> <li>• What are the storage conditions and shelf-life of the final food product?</li> <li>• What flavour characteristics are present in the food chosen?</li> <li>• Does the food product have a delicate flavour or strong flavour that can mask some undesirable taste and aroma?</li> </ul>

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