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Review article

Extrusion Technology for Encapsulation: Principle, Method, and Application

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ABSTRACT

The use of functional compounds to improve the nutritional value of foods is becoming increasingly popular across all sectors of the food industry. Even though many of these ingredients are unstable under normal conditions or have an unpleasant aftertaste, their use is restricted. Thus, it is necessary to use techniques that preserve the stability of these functional components, allow their use in a variety of food matrices, and improve their absorption in the gastrointestinal tract. Encapsulation technology is a new technique that has the potential to improve the stability of these functional ingredients while also allowing for their use in a variety of food matrices. Numerous methods have been used to microencapsulate active agents, such as the coacervation, co-crystallization, spray drying, lyophilization, and extrusion. Among these, encapsulation using the extrusion has proven to be the simplest, enabling the formation of resistant microcapsules while preserving the bioactivity of the encapsulated material. Extrusion-based encapsulation is advantageous economically and environmentally because it involves the formation of encapsulated material through direct dispersion of active components into wall material without the use of an organic solvent, as well as the benefits of lower energy and water consumption. This paper discusses the basic concept, fundamental principles, and applications of extrusion encapsulation in the food industry. The review focuses primarily on the effects of extrusion encapsulation technologies on a variety of food ingredients, including oils, antioxidants, probiotics, and flavours.

Keywords: Encapsulation; Wall material; Capsule; Extrusion Technology; Co-extrusion; Food Industry

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1. Introduction

Microencapsulation is a relatively new technique that has been employed to protect and stabilize sensitive compounds. The bioavailability of functional foods can also be improved by using various food-grade encapsulated nanoparticles (Maurya and Aggarwal 2017). During this process, very small particles of solids, liquids or gaseous material are coated with or enclosed in a continuous film of polymer materials to release their contents under certain specified conditions. Additionally, regardless of the complexity of the food matrix, microencapsulation ensures that the nanomaterials formed have a good bioavailability, water dispersibility, with increased homogeneity in the target food (Shahidi and Han 1993; Madene et al. 2006; Maurya et al. 2020). Microencapsulation has a broad spectrum of applications in the pharmaceutical, cosmetic, agrochemical and food industries and is often used for encapsulation of bioactive ingredients such as flavors, essential oils, colorants, sweeteners, vitamins and micro-organisms (Fangmeier et al. 2019; Maurya et al. 2020a). This technique preserves the closed active ingredient against the surrounding environment and delivers it when its functional properties are desired after receiving a special stimulus (Abbas et al. 2012). Also, selective delivery of food ingredients through encapsulation improves food safety by increasing the inhibition of microbial growth and masking unwanted taste (e.g., bitterness, polyphenols) improves sensory quality. Encapsulation may increase the bioavailability and water solubility of bioactive components, as well as protect against harmful conditions in a particular area of the digestive tract (e.g., stomach) and release them to the target area (e.g., intestine) for improved human absorption (Maurya and Aggarwal 2017; Maurya et al. 2020b). For instance, the encapsulation of isoflavones (genistein) by amylose inclusion complexes has improved the *in*

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vitro bioavailability of rat models (Zhu 2017). Encapsulation can be used also to change the physical properties of the original substance to enable efficient handling, help sort the components in a mixture that can interfere with each other and finally, provide an appropriate concentration and consistent dispersal of the active agent (Nedovic et al. 2011). The first commercial use of microencapsulation technology began in the late 1930s and 1940s with the introduction of carbonless carbon paper by the National Cash Register. Gelatin was used to microencapsulate a colorless dye precursor by a process known as coacervation (Jackson and Lee 1991). The wall material used in the encapsulation process may also be known by different names like core, active, fill, internal or payload phase. The encapsulant is frequently referred as the shell, coating, membrane, carrier material, capsule, matrix or external phase (Fang and Bhandari 2010; Nedovic et al. 2011). The materials used for making the shell should be biodegradable and food-grade, providing a good barrier to both the inner phases and environmental. The encapsulation or wall materials generally consist of starches, starch derivatives, lipids, proteins, gums or combinations of them. Polysaccharides are most commonly used for food encapsulation among different materials. Proteins and lipids (triglyceride oils of varied chain length and unsaturation; and essential oils) are also suitable for encapsulation. Small packages or "microcapsules" formed after microencapsulation may vary in size and shape depending on the materials and methods used during preparation (Desai and Park 2005). The selection of material depends on the characteristics of the core, as well as some wall material, such as molecular weight, conformation, electrical charge, flexibility, thermal stability, hydrophobicity, solubility, viscosity, surface activity, gelation and digestibility (Speranza et al. 2017).

Microencapsulation methods are divided into three groups: **(a)** physical methods, including solvent evaporation, freeze-drying, spray drying and supercritical liquid precipitation; **(b)** physicochemical methods like coacervation, ionic gelation and liposomes; **(c)** chemical processes, for example, interfacial polymerization and complexation of molecular inclusion (Tyagi et al. 2011; Ozkan et al. 2019). Apart from these approaches, several innovative techniques such as PEGylation, nanoemulsions and electrospinning (da Silva Freitas and Abrahao-Neto 2010; Silva et al. 2012; Wen et al. 2017) have also been developed to enhance the efficiency of microencapsulation processes. The particles formed during these processes exhibited different features, including biocompatibility, reduced bioactivity, levels of safety and cost of production. Wall materials typically contain carbohydrates (alginate, pectin and chitosan and amylose), proteins and synthetic polymers (Chawda et al. 2017). Detailed information regarding the coating material and the methods used during the microencapsulation process are described in Table 1. When choosing an encapsulation procedure, some of the parameters set out by Whelehan and Marison (2011) should be put into consideration, including the size and location of the application of the molecule, the characteristics of the encapsulated components, the delivering mechanism and the functionality of the wall material. The selection of the most promising methodology is determined by the application of the microcapsule, the shape of the component, the desired particle size, the physicochemical properties of the core and wall, the necessary release mechanism, and cost of production (Suave et al. 2006; Silva et al. 2014).

Some encapsulation methods employ organic solvents or encapsulating agents that alter sensorial properties, making subsequent application in food matrices difficult. Encapsulation based on extrusion, on the other hand, has proven to be effective in this application (Wang et al. 2017; Fangmeier et al. 2019). During this process, very small particles of solids, liquids or gaseous material are coated with or enclosed in a continuous film of polymer materials to release their contents under certain specified conditions. Microencapsulating food ingredients via extrusion offer a number of advantages over other methods, including convenience of use, simplicity, cost effectiveness, and moderate formulation conditions that promote cell viability retention (Krasaekoopt 2003). Numerous studies have been conducted to investigate the ability of extrusion to encapsulate a variety of food constituents, including flavours, probiotics, bacteriocins, and antioxidants (Yilmaz et al. 2001; Chang et al. 2010; Rodrigues et al. 2012; Nualkaekul et al. 2013; Sun-Waterhouse et al. 2014; Fangmeier et al. 2019). Additionally, the total number of publications relating to extrusion-based encapsulation has increased dramatically between 1961 and 2015 (Fig 1). Based on the foregoing, the primary goal of this review is to highlight recent advances in protection of food constituents through extrusion technology, and research works that have used this technique are described. The encapsulated and wall materials under consideration, as well as the various parts of equipment and their associated parameters, assessments performed, and primary results, will be highlighted.

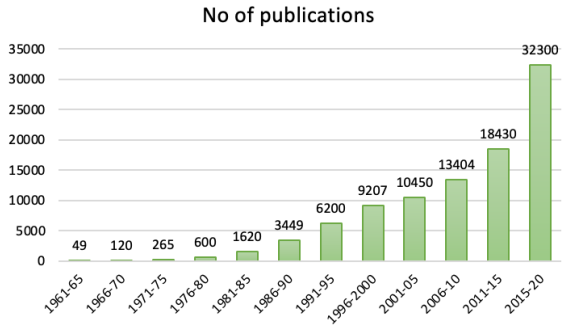

Fig. 1. Number of extrusion-related publications.

2. Extrusion encapsulation

Encapsulation based on extrusion is a relatively new technique in comparison with spray drying. Extrusion is a low-temperature process under which the core material is applied to a melted carbohydrate mass via a series of nozzles in a dehydrated liquid bath (Shahidi and Han 1993). The procedure involves the release of drops of an aqueous polymer solution (for instance 0.6-3% sodium alginate) that are active in the gelling bath (for alginate, 0.05-1.5 M calcium chloride solution is a gelling bath). The instrument used for dripping might be a syringe, pipette, nozzle, vibrator, jet cutter or atomizing disc. Jet cutters are usually the best equipment for industrial applications compared to different extrusion methods (Nedovic et al. 2011). Typically, the combination of pressure and temperature used during extrusion encapsulation is <100 psi and rarely 115 °C. When the coating materials that make up the encapsulation matrix interact with the liquid, they harden in order to keep the core material in place. The extruded filaments are removed from the gelling bath and dried with calcium triphosphate to minimize hygroscopicity and size.

Table 1. Coating methods and material employed during the microencapsulation process.

Encapsulation method	Wall materials	Encapsulated compounds	Capsules size (µm)	Maximum load (%)	Basic steps involved	Reference
Spray drying	Maltodextrin, gum, soluble soybean polysaccharide, β-cyclodextrin, mesquite gum, protein isolate, sodium caseinate	Probiotics, antioxidants, lipids, flavors	1-50	<40	Dispersion preparation Homogenization of the dispersion Atomization of the infeed dispersion Dehydration of the atomized material	Desai and Park 2005
Molecular inclusion	β-cyclodextrin	Flavors, mericana oils, vitamins	0.001-0.01	5-10	Complexes preparation through mixing/grinding/spray drying	Desai and Park 2005
Coacervation	Gum acacia, gelatin, sodium polyphosphate	Flavors, essential oils, drugs, antioxidants	20 -500	<60	Development of tri-immiscible chemical phases Deposition of the coating Solidification of the coating	Poshadri and Aparna 2010
Extrusion microencapsulation	Molten carbohydrate (simple sugar or modified starch, low DE maltodextrin)	Probiotics, flavors, proteins	50-2000	6-20	Formulation of molten coating solution Core dispersion in the molten polymer Cooling or movement of core-coat blend via liquid dehydration	Desai and Park 2005
Spray chilling	Low melting point fractionated vegetable oils or hydrogenated vegetable oils	Probiotics, enzymes, flavors, antioxidants, vitamins	20-200	10-20	Dispersion formation Dispersion homogenization Atomization of the infeed dispersion	Desai and Park 2005; Poshadri and Aparna 2010
Fluidized bed coating	Sugars, hydrocolloids, solvent-soluble polymers	Probiotics, solid particles, minerals, salts, nutraceuticals	>100	60-90	Formation of coating solution Core particle fluidization. Coating of core elements	Schell & Beermann (2014).
Lyophilization	Starch, inulin, chitosan, maltodextrins, dextran, corn syrup solids, cyclodextrins	Probiotics, lipids, vitamins, flavors, cells	1-100	<40	Mixing of core with coating solution Freeze-drying of the blend	Silva et al. 2014;
Spray-cooling	Starch, maltodextrins, modified starch, cyclodextrins, chitosan, corn syrup solids, dextran	Probiotics, enzymes, flavors, antioxidants, vitamins	20-200	10-20	Formation of the dispersion Dispersion homogenization Infeed dispersion atomization	Silva et al., 2014; Desai and Park 2005
Liposome entrapment	Wax, paraffin, beeswax, diacylglyerols, oils, fats	Fatty acids, minerals, bacteriocins, enzymes, proteins, flavors, vitamins	0.02-3	15	Microfluidization Ultrasonication Reverse-phase evaporation	Desai and Park 2005; Feizollahi et al. 2018

As a carrier, more than a single component may be useful, for example, sucrose, glucose, maltodextrin, corn syrup and glycerine (Desai and Park 2005). Sodium alginate in calcium chloride solution is the most commonly used extrusion system. The advantages of this approach are mild treatment, the wide spectrum of use and stabilization of gel particles during storage (Ozdal et al. 2020). The extrusion-based encapsulation is also economically and ecologically beneficial since it involves the formation of encapsulated material by direct dispersion of active components within the wall material without the use of organic solvents. It also offers the advantages of lower water and energy consumption compared with the spray drying process (Castro et al. 2016).

Schultz et al. (1956) invented these extrusion encapsulation processes. They obtained the encapsulated orange peel oil using a melted dextrose mass as the encapsulation material. They discovered that the microencapsulation process made it possible to maintain the flavor and stability of orange juice over a storage period of 6 months. Combining the original composition of Schultz et al. (1956) with extrusion, Swisher (1957) developed a new encapsulation method comparable to that currently used in most flavor industries. The main benefit of this method was that the encapsulated citrus oils had a fresh taste without oxidation and unpleasant flavors formed throughout the storage period. Furthermore, the storage study on encapsulated orange peel oil under accelerated conditions indicated that the product can be safely stored for a year without undergoing adverse changes. The main advantage of this approach is that the substances are surrounded by a wall (real encapsulation) and any residual oil or core content is removed from the surface in an alcohol bath (Jones et al. 1988). It guarantees high oxidation stability and therefore it is possible to store the product for 1-2 years without losing the quality features (Dezarn 1995; Abbas et al. 2012). The limitations of this technique are the production of large and porous particles, which allow the diffusion of encapsulated particles, limited options of coating materials and expensive and difficult technology (Gouin 2004).

3. Principle

Extrusion encapsulation is based on the concept of extruding the liquid mixture of phytochemicals in its shell matrix via an orifice and then gelling the formed particles by immersing them in a gelling bath as shown in Fig. 2 (Whelehan and Marison 2011).

This method has now been widely used in the manufacture of alginate-based microbeads (Zeeb et al. 2015; Gómez-Mascaraque et al. 2018), while certain biopolymers (e.g. the gum, κ-carrageenan and whey proteins) were also been used to a lesser extent for this purpose. In addition to the traditional extrusion process where the nozzle has a straight orifice, various advanced models are offered to reduce capsule size and increase technique efficiency (McClements 2017). Electric or electrostatic assisted extrusion is one of the most common extrusion techniques used, in which the size of the capsule can be controlled by exposing the polymer solution to a high-powered electrical field (Tapia-Hernández et al. 2015). This electrical field induces electrostatic repulsive forces in the charged polymeric fluid which causes the original particles to split down into smaller sizes. Also, this process was available for alginate-based preparation (Gómez-Mascaraque et al. 2018). Electrostatic extrusion is particularly effective in generating extremely small molecules (50 µm). Another technique for extrusion is co-extrusion/gelation, which is used to create spherical nanoparticles consisting of a hydrophobic core and hydrophobic/hydrophilic coating (Zuidam and Shimoni 2009; Nedovi et al. 2011). Some studies also report that co-extrusion can be effectively used to encapsulate volatile compounds (Gouin 2004; Lucía et al. 2017).

This technology consists of creating droplets by moving the shell materials and active agent solution over the extruder at a fixed speed

and drop size using a vibrating overlay. The resulting droplets fall into the bath containing the solution with the gelling agent which enables capsule production (Dolca et al. 2015). It has been documented that the co-extrusion process is used to develop core-shell materials, and it depends on the extrusion via the concentrated nozzle, which extrudes the payload dispersion through the inner nozzle and extrudes the wall material through the outer nozzle. Due to its capacity to manufacture multiple layers of coating (with multiple concentricities extrusion nozzles) in a simple and reliable operation, co-extrusion is used where a slow and controlled discharge and flavor masking of the payload is required. Particle formation occurs either by cooling and transforming the wall material into glass, gelatin or solvent evaporation (Ozdal et al. 2020). According to Serp et al. (2002), extrusion/gelation encapsulation occurs when fluid flows in a laminar state and disintegrates into droplets of the same size as a result of overlapping vibrations. In the presence of an ionic solution, these drop transformed into a gel that allows the active ingredient (core) to be encapsulated with a polymer (shell). Gelation arises when divalent (Ca^{2+}, Fe^{2+}, Ba^{2+}, and Sr^{2+}) or trivalent ions (Al^{3+}) are involved in the inter-chain ion bond between the polymer's G chain blocks, creating a tri-dimensional structure. These ions stabilize alginate chains by acting as cross-linkers, forming a gel structure where the cross-linked chains combine with more freely moving chains which hold more water. The gelation process is known to be a rearrangement of the gel network, followed by the elimination of water.

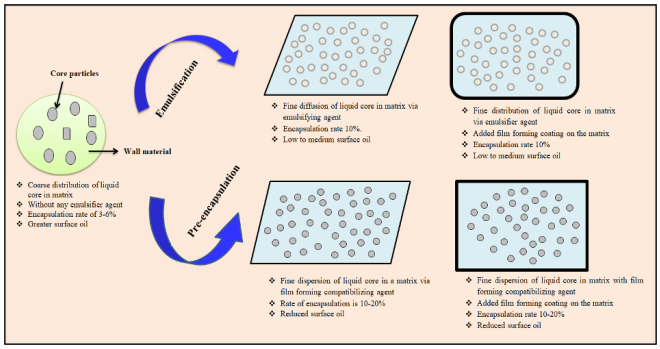

Fig. 2. Extrusion based encapsulation morphologies (Castro et al. 2016).

4. Wall material used and potential advantages

The carbohydrates like starch and its derivatives, due to their excellent binding and oxygen barrier effects, are the most widely used material for extrusion encapsulation. Binding properties are partly attributed to hydrophobic and hydrogen bonding together while oxygen barriers of carbohydrates could be result of their low water solubility (Whitcombe et al. 2005). Maltodextrin is another most widely used extrusion encapsulation material for food ingredients (Pierucci et al. 2006), due to its good film-forming capacity and oxidation resistance. It is made by partial starch hydrolysis with enzymes/acids and is well-known for its efficiency, water solubility, low cost, and ideal viscosity profile (Madene et al. 2006). Maltodextrins are categorized based on the dextrose equivalent (DE) and ranged from 3 to 19 depending on the level of hydrolysis. The degree at which starch changes to reduced sugar (greater DE means higher hydrolysis) is related to the amount of sweetness within maltodextrin (Abbas et al. 2012). As compared to other encapsulation methods (coacervation, co-crystallization, spray drying, lyophilisation), this extrusion process has many advantages: (a) There is no need for heat during the operation, which would otherwise cause degradation of the active core and shell content; b) This method allows for greater control over the volume of active core encapsulated as well as the thickness of the membrane; c) On a commercial level, this method is continuous, fast, and cost-effective, and it secures the core material; d) The size and morphology of the particles can be regulated; e) These microcapsules can be used in different sectors such as cosmetics, agriculture and textiles; f) It gives better control on the processing parameters including screw speed, temperature, moisture content, size, feed speed of core component and the wall material; g) Thermal-sensitive components can be encapsulated into the molten wall matrix by means of thermostatically regulated inlets at the end of the barrel to minimize the residence time, hence minimizing the degradation (Dolçà et al. 2015). That is why extrusion encapsulation technology is becoming a common form of encapsulation in the food industry. Despite of above advantage, the structural defects and surface viscosity of the extruded material remain obstacles that can be tackled by a clear understanding of the core and wall content, physicothermal improvements and better regulation of processing variables. Furthermore, the relatively low inclusion efficiency of bioactive liquids and the deterioration of bioactive components due to high shear forces and temperature are the major limiting factors (Abbas et al. 2012).

5. Application of extrusion-based microencapsulation in food industries

Extrusion encapsulation technology has shown an excellent way to prevent oxidation of sensitive food bioactive and has allowed scientists to explore new food formulations with improved solutions. Extrusion is primarily used to encapsulate oils, flavors, probiotics, antioxidants and colorants. In this process, food-grade starch and its derivatives are used as wall matrix, making it inexpensive, easily accessible and offering better protection to bioactive against oxidation. Improved stability makes this approach very attractive for food producers. There are several research articles on the use of extrusion technology with good encapsulation performance (Van Lengerich et al. 2007; Dolca et al. 2015; Phoem et al. 2015; Ying et al. 2016; Chang et al. 2019). To optimize the benefits of this technique, consideration must be given to the type of encapsulation, consistency of the encapsulated material in the final product, stability under operating conditions, discharge mechanism, the ideal level of phytochemicals in micro-beads, size and density of capsules, manufacturing costs and stability during storage (Abbas et al. 2012).

5.1. Encapsulation of oils using extrusion

The increased use of oil rich in monounsaturated, polyunsaturated and essential unsaturated fatty acids in food preparations has been shown to reduce the risk of developing diabetes, heart disease and inflammatory disorders. Moreover, highly unsaturated oil is a good source of fat-soluble bioactive substances, like carotenoids, tocopherols, phytosterols and polyphenols with a strong antioxidant activity (Chew et al. 2016). However, owing to the high content of unsaturated fatty acids, these oils are chemically reactive and can be oxidized easily after contact with light, humidity, oxygen and high temperatures (Velasco et al. 2012). Extrusion is a useful approach for protecting the oil from unfavorable environmental conditions and oxidization as well as for

enhancing the use of the encapsulated product (Dolca et al. 2015; Ying et al. 2016). Extrusion can be used to make high-density omega-3 oil micro-beads. In this process, melted carriers are mixed in omega-3 oils, which allow the emulsion to pass through a nozzle or die at higher pressure. The basic method for this technology was patented by Saleeb and Arora (1999). Valentinotti et al. (2006) also conducted microencapsulation of omega-3 oil via melt injection processes that begin at around 130 °C by mixing oil in a matrix with antioxidants, sugars, starch, emulsifiers and water. The paste is then extruded through the die and placed in a bath lined with cold organic solvent (liquid nitrogen or isopropanol), solidifying the sugar layer and converting it to a glassy material which is then treated with limonene (terpene) to extract the surface oil. Microencapsulated oil was found to be very stable when stored at water activity (a_w) 0.3 using this method. This procedure also avoids the widespread use of gelatin or other proteins in the encapsulation of omega-3 oils. However, the particle size increases, which limits the use of this method when processing food ingredients. Fish oil is also microencapsulated in a mouldable mixture or dough with one or more screws using a continuous process extruder (Van Lengerich et al. 2004; Van Lengerich et al. 2007).

Dolca et al. (2015) used alginate (4% w/w) as wall material and calcium chloride (0.5 M) as a crosslinking agent to encapsulate the rosemary oil using co-extrusion techniques. The microencapsulation of rosemary oil resulted in better antimicrobial properties and pesticide retention ability. Instrumental analysis via differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) showed improved oil stability for the microcapsule dried at 60 °C. Microencapsulated pellets of choline bitartrate (CBT) using hydrogenated soya bean oil (HSO) as an encapsulating agent were developed using a modified extrusion spheronization method (Gangurde et al. 2015). During the study, the effect of different process parameters including spheronization speed (500-1400 rpm), the temperature of jacketed spheronizer (60-65 to 70-75 ºC) and time of spheronization (5-20 min) on the selected responses (flow behavior, hardness-friability index, morphological features, encapsulation efficiency, drug content and *in vitro* drug release) was studied to optimize the pellet formulation. The results showed that coating with 90% and 60% CBT was effective concerning on all necessary parameters of the evaluation. Optimized formulation did not reveal any change in color, odor, moisture and drug content after 6 months storage under accelerated environment (75% RH at 40°C). In another study, Chew and Nyam (2016) investigated the impact of co-extrusion variables like variations in flow levels for core and shell feed, vibration frequencies, shell compositions and drying processes on the functionality of microcapsules of kenaf seed oil (MKSO). The MKSO was made with a Buchi Encapsulator B-390 and the shell formulation was carried out using high methoxyl pectin (HMP)-alginate and sodium alginate (1.5%, w/w). Results indicated a better encapsulation efficiency of MKSO at 500 Hz vibrational frequency and a core-shell flow rate of 0.2-7.0 ml/min (for HMP-enhanced alginate) and 0.2-7.2 ml/min (for 1.5% alginate).

Ying et al. (2016) embedded microencapsulated fish oil powder in a gelatin-based dough mixture (47%, 60% or 70% w/w total solids (TS)) and extruded through the die after being preconditioned at different temperatures (20, 35 or 50 °C). The extruded noodles produced were dried and pressed into tablets (35% by weight of oil) to analyze the impact of the dough TS and the preconditioning temperature on the amount of oil leaking from the tablets. They found that during compression, the TS and the preconditioning temperature of the dough affected the surface oil of the extruded granulate and the oil leakage. The lowest oil leakage was observed for tablets that were made from dough extrude with 47% TS and preconditioned at 35 °C. Further, fish oil microencapsulated gelatin matrix (50% by weight oil) in the also tended to improve the oxidation stability of eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA). This result was mainly due to the protective effect of the gelatin matrix against oxidation that occurred after extrusion and subsequent tableting. Similarly, Lucía et al. (2017) developed almond oil capsules with co-extrusion using sodium triphosphate pentabasic (STP) as the crosslinking agent and chitosan as the shell material. The impact of various process variables such as cross-linker concentration (4-8 g/L), nozzle size (0.75/0.90 to 0.45/0.90 mm) and voltage potential (250-350 V) was studied to optimize the microencapsulation process. Outcomes indicated the size of the nozzles and crosslinking concentration as the key variables that can affect the encapsulation process. The optimal concentration of the crosslinking agent was 8.0 g/L at 1% (w/v) chitosan concentration. The microcapsules with better cross-linking and morphological properties occurred at nozzle size (inside/outside) ratio of 0.45/0.90 mm. Degradability studies revealed rapid weight loss from chitosan capsules during 24 h of storage, which makes their uses suitable for the products requiring high degradation.

5.2. Encapsulation of antioxidants and bacteriocin compounds

Food antioxidants have been categorized according to their chemical structure and function into various groups, including the lipid-soluble components (carotenoids, vitamins E, tocopherols and terpenoids) and water-soluble bioactive agents (citrates, betalains, norbixin, flavonoids, phenols and anthocyanins) (Carocho et al. 2017). As a food preservative, these antioxidants have become increasingly popular because they prevent the oxidation of food; minimize nutrient loss along with ensuring the freshness via better retention of flavors, odors, color, taste and texture. As a consequence, the role of dietary antioxidants has been associated with various health advantages in the fight against cancer, neurological and cardiac diseases, as well as hypotensive, anti-allergic, anti-inflammatory, antibacterial, antiviral and skin wound healing benefits (Alvarez-Suarez et al. 2016). The related antioxidant activities of bioactive substances can be affected by degradation resulted from moisture, oxygen, light, temperature and the unsaturated bonds in the molecular structure. Microencapsulation with a compatible carrier is also an effective strategy to encourage bioactive conservation and environmental safety as well as for advancement in hiding bitter taste, off-flavor and astringency of polyphenols (Ballesteros et al. 2017). Extrusion technique does not use high temperatures; therefore, bioactive compounds are not thermally degraded (Chew and Nyam 2016). It involves the passage of bioactive compounds mixed with the wall polymer through an extrusion needle or nozzle to form a falling drop into a calcium solution, allowing ionic gelation to occur and enclosing the core compounds. Extrusion technology can be easily adapted to obtain microcapsules with sophisticated equipment like Buchi® Encapsulator or using a simple syringe with or without an extrusion needle in a calcium bath. The concentrations of alginate and calcium and their ratio are the critical parameters that need to be controlled during this process (Macías-Cortés et al. 2020).

Many research papers are documenting the effectiveness of extrusion technology for the microencapsulation of phenolic

compounds (Table 2). Swisher (1957) performed an accelerated shelf-life study on orange peel oil containing antioxidants and recorded a shelf-life of approximately 1 year. Anbinder et al. (2011) encapsulated the phenolic extract of Yerba Mate (*Ilex paraguariensis*) in 2% (w/w) sodium alginate and 0.5% (w/v) chitosan to form the beads. To understand the interaction between wall materials and polyphenols, the effect of developed beads (1900 µm) in the gastrointestinal system was investigated. The highest levels of polyphenol for both encapsulation systems were found to be released in simulated gastric fluid (93.8% for alginate polyphenol and 66.6% for alginate-chitosan polyphenol systems) while being more discrete in simulated intestinal fluids (10.0 and 19.6%). However, the release of polyphenols in the simulated gastric fluid by the system of alginate-chitosan-polyphenols is more than 60% lower than the system of alginate-chitosan. This finding is based on the defense against the chitosan barrier and its close association with extracts from Yerba Mate.

Sun-Waterhouse et al. (2011) evaluated the impact of coating materials including caffeic acid (300 ppm) and sodium alginate (1.5% w/w) on the storage stability of olive oil. Results indicated that the integration of caffeic acid improved the total phenols and storage stability of encapsulated olive oil during 30 days of storage under two different temperatures (20 or 37 °C). Both encapsulation and incorporation of caffeic acid resulted in slower oxidation changes with better protection of unsaturated fatty acids (UFAs) like omega-3 FA (C18:3), omega-6 FA (C18:2) and omega-9 FA (C18:1). It has been suggested that the process of encapsulating oil with alginate microspheres could be a viable approach to improving olive oil stability and nutritional value of the final oil product in terms of increased total phenol and unsaturated fatty acids.

Rijo et al. (2014) successfully encapsulated the microwave-assisted extract of *Plectranthus ecklonii* with a high yield (22.9±0.3 mg/mL), antioxidant efficiency (25.47±1.1%) and inhibition activity of acetylcholinesterase (AChE) (59.14±4.97%) into alginate beads (98.64-100.0%) via extrusion technology. They observed that microencapsulated plant extract showed higher rosmarinic and caffeic acid content along with better antioxidant activity in terms of AChE inhibition assay. It was concluded that encapsulation can promote extraction by encouraging the safe and sustainable release of active components. Pasukamonset et al. (2016) published very promising results on the extrusion-based encapsulation of phenol extracts from *Clitoria ternatea* (CT) petal flower using calcium chloride ($CaCl_2$) and alginate coating. The study revealed that the optimized conditions for CT-loaded alginate beads were the following: 10% CT, 3% $CaCl_2$ and 1.5% alginate (w/v).

The maximum encapsulation efficiency (84.8±0.4%) and antioxidant activity (11.8±0.1 mg gallic acid equivalent/g) were achieved under these conditions. Also, prepared microcapsules showed a smooth surface including particle size of 985±1 µm. The study undoubtedly offers a new type of food-grade encapsulation for improving the stability and biological activity of plant phytochemicals. Microencapsulation of polyphenols from residual grape pomace was performed by dissolving 1% (w/v) alginate and 3% (w/v) chitosan in a pH-adjusted calcium solution (Moschona and Liakopoulou-Kyriakides 2018). The resulted microcapsules had a particle size of 1004-1070 µm and encapsulation efficiency in the range of 55-79%. These microcapsules had an initial phenol content of 13 to 22 mg/g, with molecules including quercetin, transferula acid, kaempferol, caffeic and ellagic acid derivatives. The antioxidant capacity assessed via DPPH and ABTS assay was reported between 65-94 and 67-97%, respectively. Chang et al. (2019) used the carbohydrate-based coating matrices (maltodextrin-gum arabic/maltodextrin/maltodextrin-trehalose) for the encapsulation of ascorbic using hot-melt extrusion technology. Studies have shown that the existence of the wall matrix affects the physical properties of the extruders. They achieved a lower glass transition temperature (T_g) after coating with trehalose (low molecular weight carbohydrates), while the use of gum arabic (high molecular weight carbohydrates) mainly reduced the expansion ratio. The breakdown rate of the microcapsule powder was slightly lower compared to un-encapsulated ascorbic acid, which indicates potentially good release profiles. The microstructure analysis shows that ascorbic acid and carbohydrates were miscible and generated a solid solution during the melting phase.

Kim et al. (2019) investigated the influence of zeolite encapsulation and titanium dioxide (TiO_2) coating on the thermal stability of caffeine with polyethylene terephthalate (PET) during melt extrusion. The coating methods used to form caffeine nanocomposites include: *in situ* coating where the precursor TiO_2 was applied to the zeolite/caffeine solution; and *ex-situ* encapsulation where prepared zeolite/caffeine capsules has been added to a TiO_2 coating precursor. Finally, the TiO_2-coated nanocomposites are extruded with PET at 285 °C to produce the caffeine microcapsule. The *in situ* TiO_2 coatings led to higher residual caffeine concentrations than *ex-situ* TiO_2 process, even after the melt extrusion at 295 °C. They have found that the addition of a higher level of caffeine in the zeolite/caffeine solution resulted in an improved rate of caffeine absorption. However, the variation in the reaction time (5 min to 4 h) did not change the rate of caffeine absorption. Caffeine was shown to have higher temperature stability (400 °C) in the TiO_2-coated zeolite/caffeine capsules compared to uncoated, free capsules.

Extrusion encapsulation has been shown to protect bacteriocin from contact with food ingredients, potentially improving effectiveness and stability. Pei et al. (2014) reported that the encapsulation of bifin C6165 with calcium-alginate gel extrusion prevents bacteriocin from food contamination and theoretically improves potency and stability. In addition, encapsulated bificin C6165 can be used as a promising tool for controlling *Acidoterrestris* (DSM3922 and CFD1) spore and vegetative cells in the food juice industry.

Kennedy (2007) observed the influence on the release action of calcium polysaccharide gel (CaPG) coated pellets. Three different drugs with identical chemical structure but different water solubility were used, viz. caffeine (CAF), theobromine (TBR) and theophylline (TPL). An extrusion spheronization process was used to prepare drug-filled spherical pellets and the CaPG was added to pellets filled with different drugs by an interface complexing coating. Depending on the solubility of the active drug and the polysaccharide form, the encapsulation power of the coated pellets ranged from 57.6 to 84.3%. For all the drugs tested, the release behavior in CaPG-coated pellets showed varying release kinetics. This may have been due to the difference in the dissolution of drugs within the core prior to dissemination and diffusion through the CaPG coat. Due to the low solubility, the release of TBR from the coated pellets was considerably slower than the TPL or CAF. Furthermore, the release of all drugs was approximately four to six times slower for coating compared to free pellets. It has been suggested that CaPG coating facilitated the delay of drug release from the pellets, providing a long-lasting mechanism for caffeine release.

Table 2. Application of extrusion-based encapsulation in different functional ingredients. Source: Speranza et al. 2017; Fangmeier et al. 2019; Ozdal et al. 2020.

Source	Encapsulated substance	Encapsulation mode/agent	Main outcomes	References
Emulsion/dispersed phase	Sunflower oil	Emulsion/starch matrix extrusion	The diameter of the scattered oil droplets reduced with hydrophobic-hydrophilic ratio. Parameters like improved screw speed, high melt temperature and low throughput often result in decreased oil droplet sizes.	Yilmaz et al. (2001)
Glassy extrudates	Ascorbic acid	Melt extrusion/maltodextrin, maltodextrin-gum arabic and maltodextrin-trehalose	Small molecular weight carbohydrate (trehalose) decreased glass transition temperature thus, making the extrusion processing more efficient. The expansion ratio decreased significantly with high molecular gum arabic. The lowest dissolution value was reported maltodextrin-gum arabic and maltodextrins-trehalose encapsulated powders.	Chang et al. (2010)
Dough matrix (grain flours and vegetable shortening)	Ferrous fumarate	Agglomeration through extrusion	Encapsulated iron particles showed good iron content and excellent in vitro digestibility. Extruded materials displayed larger density, lower porosity and a finer texture on the surface. Extruded iron capsules with appropriate particle size (300-700 µm) were suitable for salt fortification.	Li et al. (2011)
Olive oil	Caffeic acid (CA)	Extrusion/alginate	Encapsulation of CA improved the total phenolic content of the product. Slower oxidation changes in the encapsulated samples during storage (20 or 37 °C/30 days). Protective effect of encapsulation on unsaturated fatty acids such as omega-9 FA (C18:1), omega-6 FA (C18:2) and omega-3 FA (C18:3).	Sun-Waterhouse et al. (2011)
Avocado oil	Phloridzin (at 300 ppm)	Extrusion/alginate and hydroxyl-propyl methylcellulose (HPMC) (3:1)	The combined use of phloridzin or BHT was synergistically effective for enhancing the oxidative stability and preventing hydrolytic rancidity in avocado oil (for 90 days at 37 °C).	Sun-Waterhouse et al. (2012)
Orange, peach juice	*Lactobacillus* paracasei L26	Extrusion/alginate with or without double coating (chitosan or dextransulphate)	The double-coated alginate microcapsule (viable load as 9 Log CFU/ml) was ideal for probiotic protection after 50 days at 5 °C Encapsulated juices showed decreased pH, glucose, fructose and citric acid, whereas an increase in formic acid during storage.	Rodrigues et al. (2012)
Cranberry, pomegranate juice	*Lactobacillus plantarum*, *Bifidobacterium longum*	Extrusion/alginate or pectin with gelatin, chitosan and glucomannan	Gelatin coated pectin micro-beads provided the best protection for probiotics after 6 weeks of storage (viable load of 4.5 Log CFU/ml). The hardness of the beads declined owing to the sequestration of calcium ions.	Nualkaekul et al. (2013)
Cowberry juice .	Sacromycesis cerevisiae boulardii	Extrusion/alginate-inulin, xanthan gum	Encapsulations enhance cell viability (7.59 Log10 CFU/ml) during storage (4 weeks/4 °C). Encapsulation facilitates the absorption of polyphenols and anthocyanin.	Fratianni et al. (2014)
Apple juice	Bificin C6165	Extrusion/calcium-alginate	Bificin encapsulations (40 µg/ml) had an inhibitory effect on vegetative cells of A. acidoterrestris. Thermal resistance of A. acidoterrestris spores reduced after addition of bacteriocin. The non-significant effect of bificin against the endospores of A. acidoterrestris in apple juice.	Pei et al. (2014)
Canola oil	Quercetin, vitamin E or butylated hydroxy terpenes (BHT)	Co-extrusion/alginate only (A), alginate-novation 2300 starch with 1.4% amylose (A-N) and alginate-Gelose 80 starch with 89.8% amylose (A-G)	Encapsulation with antioxidants enhanced the phenolic content of canola oil. Quercetin was more effective and equivalent to BHT in preventing oil oxidation than vitamin E. Overall, the three shell formulations were ideal for the safety of unsaturated oil, with the best and worst barriers being A-N and A-G shells.	Sun-Waterhouse et al. (2014)
Pineapple juice	Bifidobacterium longumlongum	Extrusion/mixture of oligosaccharides, fructo oligosaccharides and Eleutherine Americana extract	Extrusion improved the cell viability of probiotic. Good sensory characteristics of juice containing probiotic capsules.	Phoem et al. (2015)
Raspberry, ground ivy, hawthorn, nettle, yarrow, olive leaf extracts	Chlorogenic acid and caffeic acid	Electrostatic extrusion/alginate-chitosan (with ascorbic acid)	Good encapsulation performance was obtained for all capsules (80-89%). Refrigerated storage decreases antioxidant capacity; however, the bioactivity of the capsules is still good for food uses.	Belscak-Cvitanovic et al. (2016)
Apple juice	*Lactobacillus* rhamnosus GG	Extrusion/chitosan alginate with or without inulin	The higher survival rate of encapsulated probiotic (4.5-fold) after 90 days of storage at two different temperatures (4 and 25 °C). Inulin improved bacterial survival under gastrointestinal transition during storage. Probiotic encapsulation did not have any negative impact on the organoleptic properties of apple juice.	Gandomi et al. (2016)
Microcapsule	Nysine	Extrusion-vibration/sodium alginate (16 g/L)	The alginate coating protected nysine from protease activity. Improved encapsulation efficiency (71 to 76%). The antimicrobial activity of the microcapsules was greatly conserved at 4 °C and pH 4.5 and 6.0.	Maresca et al. (2016)
Clitoria ternatea (CT) petal flower	Phenolic extract	Extrusion/alginate with calcium chloride	Use of optimized conditions (10% CT, 1.5% alginate and 3% calcium chloride (w/v) results in maximum antioxidant activity (11.76 mg GAE/g). Improved encapsulation efficiency and pancreatic alpha-amylase inhibitory activity.	Pasukamonset et al. (2016)
Hibiscus sabdariffa L. cups	Bioactive component	Dripping-extrusion/2.0% pectin	Improved encapsulation efficiency for polyphenols (95.6%) and anthocyanin (93.9%). Dripping-extrusion encapsulation at 100 Hz/400V generated the lowest anthocyanin degradation during refrigerated storage.	de Moura et al. (2018)

5.3. Encapsulation of probiotics

Probiotics are the living organisms that offer the host numerous health benefits if they are provided in sufficient quantities (Jiménez-Pranteda et al. 2015). Probiotics are important for the control of gastrointestinal disorders, including infectious diarrhea, antibiotic-associated diarrhea, allergy, lactose sensitivity, inflammatory diseases and modulation of the systemic immune response (Majamaa and Isolauri 1997; Malchow 1997). The critical probiotic scale is 106 to 107 CFU/g or ml (Bhat et al. 2015); although there are so many variables that can affect their viability and health effects (pH, oxygen, storage temperature and hydrogen peroxide). There are multiple strategies to overcome this obstacle: a sufficient range of acid and bile resistant strains, stress adaptation, usage of oxygen-impermeable cans, micronutrient absorption (amino acids and peptides) and microencapsulation (Martın et al. 2015). Probiotics are usually inserted into capsules by extrusion resulting in gelled droplets called beads, which have a network of porous core (Gbassi and Vandamme 2012). Extrusion benefits include flexibility, lower

price and mild operating conditions that guarantee higher cell viability (Rathore et al. 2013). Krasaekoopt and Kitsawad (2010) encapsulate Lactobacillus casei 01 by extrusion technology using alginate and chitosan as the coating material. The sensory attributes of prepared probiotic beads in fruit juices (grape juice/orange juice) were evaluated using consumer-based descriptive investigation. The application of probiotic beads has been shown to influence the sensory characteristics of the juices by increasing the swallowing difficulty and the residual particles in the products. Most of the sensory panelist approved grape juice and orange juice with probiotic beads (84.3 and 82.3%, respectively), with taste and general preferences of 6.8 and 6.9 for grape juice; and 6.6 and 6.7 for orange juice. Microbead coatings may be used in delayed cell-release for targeted intestinal probiotic delivery. Doherty et al. (2012) examined the influence of encapsulation of Lactobacillus rhamnosus GG with whey proteins on the probiotic viability during storage of cranberry and pomegranate juice for 28 days at 4 and 25 oC. Coated micro-beads result in significant improvement of probiotic viability (Log 8.6 CFU/ml) along with the good binding of hydrophobic molecules during 28 days of storage period. Coated micro-beads also showed a high gastric survival rate (9.5 Log 10 CFU/mL) with 30 min delayed intestinal release compared to uncoated micro-beads.

Mirzaei et al. (2012) select extrusion for encapsulating Lactobacillus acidophilus with calcium alginate and resistant coating material and observed a better survival rate of probiotic in Iranian, white-brined cheese during 182 days of storage. Further, the number of viable cells was also assessed during 6 months of storage under refrigerated conditions. Coated micro-beads reduced probiotic viability (11 Log CFU/g) over the end of 182 days of storage period. Gaanappriya et al. (2013) documented improved survival of Lactobacillus acidophilus in sapodilla juice after extrusion encapsulation using the alginate wall matrix. Nualkaekul et al. (2013) investigated the influence of encapsulation (alginate or pectin with chitosan, gelatin or glucomannan coating) on the survival of Lactobacillus plantarum (NCIMB 8826) and Bifidobacterium longum (NCIMB 8809) in cranberry juice. The findings showed that pectin beads in gelatine had the maximum protection and the viable number of probiotics after six weeks was 4.5 Log CFU/ml. Fratianni et al. (2014) microencapsulated Saccharomyces cerevisiae using different shell materials (alginate-inulin-xanthan gum) to evaluate their growth and survival rate under gastrointestinal conditions in berry juice for 4 weeks at refrigerated conditions. Results indicated a significant improvement in cell viability of microencapsulated prebiotic (7.59 Log 10 CFU/ml)) during storage than free yeast (6.98 Log 10 CFU/ml, respectively) along with better protection against simulated gastrointestinal transit storage. Coated microcapsules demonstrated the absorption of a certain amount of polyphenols and anthocyanin salon with ensuring better protection against exposure to simulated gastrointestinal conditions during the entire storage duration. Chaikham (2015) used alginate and plant extracts (pennywort, cashew and yanang) as the shell material to encapsulate probiotic bacteria (Bifidobacterium lactis Bb-12, Lactobacillus casei 01 and Lactobacillus acidophilus LA5) via extrusion technology. The survival rate of encapsulated probiotic in different fruit juices (maoberry, mulberry, melon and longan juices) and stirred yogurt was evaluated under refrigerated conditions after 1-month storage. Results showed that both cashew flower and green tea extracts significantly increased the safety of probiotic beads in fruit juices and yogurt compared to uncoated bacteria. The encapsulated L. casei 01 and B. lactis Bb12 showed a greater survival rate during storage compared to encapsulate L. acidophilus LA5.

Shinde et al. (2014) used probiotic bacteria and polyphenols to prepare microcapsules because of their high demand in foodstuffs. Probiotics are living organisms that can provide a host with many health benefits if provided in adequate quantities. Probiotics (lactic acid bacteria) have been useful in the fermentation of various foods, including cheese, yogurt, butter, buttermilk, kefir and koumiss. Polyphenols are phytochemicals known for their high antioxidant capacity, anti-carcinogenic, anti-inflammatory and protective effects against various chronic diseases. The results illustrated that co-extrusion technology was successful in protecting probiotics after the end of 50 days of storage due to a very low reduction in cell viability. Phoem et al. (2015) used extrusion for encapsulation of *Bifidobacterium longum* using different wall material (oligosaccharides extract, commercial fructooligosaccharides mixture and *Eleutherine americana* extract) and evaluated cell viability under simulated gastrointestinal conditions in pineapple juice. Results indicated that the number of viable cells of encapsulated *B. longum* was improved in the juice during the gastrointestinal assay. Tootoonchi et al. (2015) used extrusion for microencapsulation of probiotics (*Lactobacillus acidophilus* LA5, *Lactobacillus. casei* 431) and reported a better survival rate of bacteria in orange juice (8 Log CFU/ml) after the end of 4 weeks storage under refrigerated conditions. The sensory acceptability of probiotics enriched juices was improved significantly after alginate and chitosan-based extrusion microencapsulation process. Extrusion-based encapsulation of probiotic bacteria indicated a successful technique for improving the bacteria viability, both during the preparation of apple juice and in the gastrointestinal tract without harmful organoleptic effects.

Gandomi et al. (2016) examined the influence of chitosan and alginate encapsulation (with or without inulin) on the efficacy of probiotic *Lactobacillus rhamnosus* GG in apple juice over 3 months storage at 25 and 4 °C. In addition, the impact of the inulin addition on the survival rate of probiotic was also examined under simulated gastrointestinal conditions. The results showed that chitosan-alginate coating material resulted in a 4.5 times higher survival rate of encapsulated probiotics in apple juice compared with the free bacteria. The microencapsulation also showed a better viability rate of bacteria (27.7%) in the gastrointestinal transition model. The addition of inulin reported having a positive impact on the viability of encapsulated bacteria during gastrointestinal transition test and product storage. Apple juices with encapsulated bacteria displayed satisfactory sensory scores after 3 months of storage. In another study, Silva et al. (2016) published promising data regarding the encapsulation of probiotic (*Lactobacillus paracasei* BGP-1) dispersed in coconut fat or sunflower oil through co-extrusion. During the process, they employed alginate or alginate-shellac mixture as coating materials and lyophilization or fluidized bed as post-treatment methods for drying the capsules. The authors were able to develop capsules with a diameter of 0.71 to 0.86 mm using the co-extrusion technique, which allows the use of solid foods (dark chocolate, cereal bars and mixed nuts). They also noted that, after 60 days of storage at 25 °C, the viability in fluidized capsule loads was up to 6 Log CFU/g, which corresponds to 90% of the initial population of probiotics. In addition, the microcapsule formed with alginate-shellac and coconut fat was the most successful in enhancing the survival rate of the probiotic in simulated gastrointestinal fluids, mainly due to the porosity of the microcapsules where 7.5 Log CFU/g probiotics (95%) remained at

the end of the analysis. Mathews (2017) studied the cell viability of *Lactobacillus acidophilus* and *Lactobacillus casei* encapsulated using different shell materials (calcium alginate-gelatin and prebiotic such as inulin and lactulose) by extrusion processing. Microencapsulation of probiotics resulted in the successfully delivery of viable bacterial cells to the colon against the adverse simulated human gastrointestinal condition. Petraitytė and Šipailienė (2019) used different divalent cross-linkers (calcium chloride, calcium carbonate, calcium lactate, calcium edetate and strontium chloride) to encapsulated *Lactobacillus plantarum* F1, *Lactobacillus reuteri* 182 and *actobacillus helveticus* 305 via an extrusion process. The effect of probiotic encapsulation on the encapsulation efficiency and viability has been investigated. They have reported the highest mortality and encapsulation efficiency (>90%) after extrusion-based encapsulation using $CaCl_2$ and the strontium chloride/calcium lactate based cross-linking agents.

5.4. Encapsulation of flavors

The flavor and aromatic components play a vital role in customer satisfaction and in influencing the further intake of food (Madene et al. 2006). However, prolonged residual activity and thermal stability of flavor and fragrance are the key factors preventing the advancement of the flavor industry (Jun-Xia et al. 2011). The encapsulation process is perhaps most commonly used to improve flavor stability. Material characteristics, formulation and operating parameters are the variables that may decide the properties and end-use of microencapsulated products (Paulo and Santos 2017). The final properties of micro-particles can influence the release rate of the activated compound (Li et al. 2008). Many factors, including technical problems (production and storage characteristics), economy and consumer satisfaction, have to be considered for the microencapsulation of flavors in the food industry (Aguiar et al. 2016). Difficult extrusion conditions, however, can lead to the loss of many volatile flavors due to thermal deterioration, oxidation, interaction with food ingredients and a rapid pressure drop flashing from the die. Extrusion based flavor encapsulation has been used in glassy carbohydrate matrices for volatile and unstable compounds (Gunning et al. 1999; Benczedi and Bouquerand 2001). The major benefits of the extrusion process are that flavor is stable against oxidation. In the glassy state, carbohydrate matrices have excellent barrier properties and extrusion is an easy technique for encapsulating flavor in those matrices (Gouin 2004). Structural defects such as cracks, thin walls or pores that arise during or after treatment often improve the process variables and the flavor dispersion of the extruded carbohydrates (Madene et al. 2006).

The extrusion is second in volume for the manufacture of dry encapsulated flavoring products. This method was based on the fundamental concepts of Schultz et al. (1956) who simply applied citrus oils to a molten mass of carbohydrate (such as hard candy), stirred it in a crude emulsion and then allowed it to solidify as mass. The obtained mass was crushed to the ideal particle size and sold as an enclosed flavor. In 1957, Swisher received the first patent to encapsulate flavor by extrusion, adapting the basic extrusion method of Schultz et al. (1956) to create a process similar to that used in the flavoring industry today. The key aspect of this patent was the preservation of fresh flavor in encapsulated citrus oils, which would otherwise easily oxidize during storage, leading to undesirable flavors. Swisher used corn syrup (42 DE) as an encapsulation matrix. A strong antioxidant was applied to the citrus oils during treatment at high temperatures. In Swisher's patent, butylated hydroxyanisole and 4-methyl-2,6-ditertiarybutylphenol were listed by name and the applied level was suggested to be 0.05% of the flavor oil. Emulsifiers have also been added to facilitate the formation of emulsions and to promote stability. Although his patent proposed various synthetic and natural emulsifiers, patent examples used a mixture of mono-glycerides and monoglyceride sodium sulfoacetate at a 1% level (based on the weight of total emulsion). This corn syrup/antioxidant/emulsifier mixture consisted between 3 and 8.5% moisture and temperatures of around 120 °C had been required to keep the solution low enough in viscosity to allow the flavoring oils to be added. Approximately 10% of the flavoring oils were added and the mixture was vigorously agitated under nitrogen to produce an oxygen-free emulsion. This emulsion was forced into a hot, immiscible liquid (vegetable or mineral oil) through a die, which was then rapidly cooled or simply extruded into pellets that have been allowed to solidify and then crushed to appropriate particle size. To extract surface oil, the ground material was washed with solvent (isopropanol) and later dried under vacuum. The result of this method was a free-flowing granular material with a flavoring of 8-10% (Reineccius 1989).

Beck (1972) substituted the high-DE corn syrup solids by a mixture of maltodextrin and sucrose (Fig. 3). A carbohydrate melt consisting of approximately 55% sucrose and 41% maltodextrin (10-13 DE) was used during the study. The remaining components involved moisture and additives. They continued the use of an anticaking agent and suggested pyrogenic silica instead of tricalcium phosphate. The amount of flavor obtained usually ranged from 8 to 10% with 12% recognized as a feasible limit. Miller and Mutka (1985, 1986) received two patents on extrusion-based flavor encapsulation. The first patent (1985) relates to the process for encapsulating orange juice solids. Miller and Mutka's second patent was established specifically for process optimization. The aim was to increase the loading capacity and encapsulate the flavors (Miller and Mutka 1986).

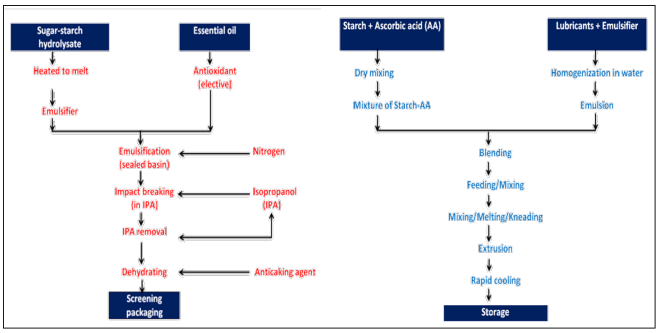

Fig. 3. Encapsulation of bioactive compounds by extrusion technology (Shahidi and Han 1993; Desai and Park 2005; Abbas et al. 2012).

The study of the effect of the cooking temperature on flavor loading and encapsulation performance showed that the optimal cooking temperature of high loading products (>22%) is about 123 °C. Temperatures above or below have limited encapsulation capability. Cooking temperatures of 123 °C corresponded to around 5% moisture (Reineccius and Coulter 1969). Since cooking temperatures are essentially determined by the moisture content, Miller and Mutka (1986) hypothesized that too little moisture diminished the emulsifying capacity, whereas too much moisture impeded encapsulation. In the review of the Miller and Mutka (1986) study, it can be seen that maximizing boiling point, emulsifier concentration and pressure in the cooking vessel improved encapsulation efficiency at high flavor loads. Although their patent

claims load up to 35%, they only mentioned one example with loads of up to 27.6%. Most examples demonstrated 15 to 20% feasibility at flavor loads. However, this is still far above the conventional flavor loads of 8 to 10% achieved in commercial applications. Crocker and Pritchett (1978) used the extrusion process to encapsulate the citrus oils with glycerine and corn syrup solids. These authors note several factors that improve the quality of capsule, including glucose syrup dextrose equivalent (DE), emulsifier, flavor oil content and emulsification pressure.

An experiment with a complex β-cyclodextrin-β-ionone model was documented with some enhancements in the retention of β-ionone relative to un-encapsulated, but significant losses were still observed during extrusion (>90%) (Crouzet et al. 1984). Significant attempts have been identified to enhance the preservation of a pre-added flavor, like the use of pre-gelatinized starch, precursors, flavor infusion and encapsulated flavors (Sadafian and Crouzet 1988; Kollengode and Hanna 1997). Enhancements in the recovery of extruded flavor in microencapsulated flavor (up to 30% recovery) than the non-encapsulated flavor (up to 6.7% recovery) were evidenced from extrusion of different mixtures (starch, starch caseinate and cookie mixture) with certain model flavor compounds, such as a β-cyclodextrin-limonene complex by a twin-screw extruder operated at 160 ºC (Sadafian and Crouzet 1988). The latest work has shown a better recovery rate of flavor compounds (eugenol, cinnamaldehyde, 3-octanone and nonanoic acid) cross-linked with β-cyclodextrin in maize starch extrusion (up to 100%) (Kollengode and Hanna 1997). They utilized such an enormous amount of flavor in the premix that these reports have no practical consequences for the process. It has been documented that the molecular encapsulation of β-cyclodextrin flavors can increase the retention of the pre-added flavor during extrusion. Yuliani et al. (2006) encapsulated β-cyclodextrin with d-limonene to increase its stability during pre-added flavor starch extrusion. They investigated the influence of the maximum extruder barrel temperature (133-167 °C), the screw speed (158-242 rpm) as well as the β-cyclodextrin-d-limonene capsules in the feed (0-5%) on the flavor retention and the physiochemical characteristics of the extrudate (expansion, texture, color difference, residence time distribution (RTD), water absorption and water solubility index (WSI). Results indicated that the capsule level made a significant contribution to expansion, WSI and hardness and increased capsule concentrations from 0 to 5% caused a significant reduction in hardness. Capsules improve the expansion and retention of flavor and reduce the WSI. In conjugation with temperature, the concentration of capsules at low temperatures increased the retention of flavor while increasing the concentration of capsules at high temperatures decreased the flavor retention.

Manojlovic et al. (2008) used electrostatic extrusion to encapsulate ethyl vanillin in alginate gel microspheres and investigated the thermal activity of encapsulating ethyl vanillin using thermo-gravimetric (TG) and differential calorimetry scanning (DSC) measurements. The thermal decomposition results showed two well-resolved mass losses. The first was in the temperature range of 50-150 °C, the highest around 112 °C, resulting in the breakdown of the polymer network. The second decrease in the temperature range from 220 to 325 °C with an average temperature of ~247 °C is related to the release of vanilla. The findings showed that most of the vanillin remained intact up to 230 °C, although excessive heating at high temperatures contributed to the complete loss of the flavor. Pradeep and Nayak (2019) examined the storage stability and morphological of C-Phycocyanin (C-PC) after extrusion encapsulation using sodium alginate matrix. The stability of encapsulated C-PC was tested at different temperatures (40-80 °C). The degradation of 2.5% alginate-encapsulated C-PC was observed to be slow at 60 °C and the relative concentration was 90.28% for 120 min. While the degradation of C-PC capsules was 88.19 and 86.89%, respectively, at temperatures between 70 °C and 80 °C. FTIR, the structural stability of the protein tested by SDS-PAGE suggested that during the encapsulation process the C- PC was not damaged. The high-temperature encapsulation process preserves the nativity and structural features of the protein. It can be assumed that the stability of C-PC can be improved with a controlled extrusion encapsulation process.

6. Conclusion

The extrusion technology is successfully used to encapsulate flavors, vitamin C, essential oils and bioactive components. This process involves generating droplets via vibration overlay by passing the active ingredient and shell material solution through an extruder under controlled speed and droplet size. These drops fell into a gelling bath solution that helped formulate the capsules. The technology enables the production of homogeneous microcapsules with controllable dimensions and with sufficient efficiency and repeatability under optimal conditions. However, it is important to specify the encapsulation equipment specifications in advance, as this directly affects the properties of the microcapsule and thus the efficient safety of the bioactive component. The main advantage of extrusion is undoubtedly its excellent taste security against oxidation, controlled release and stability during processing and storage of the bioactive compounds. The solvent-free cooling and limited use of water to encapsulate extruded products also make this approach environmentally friendly and meet food safety requirements. However, the limited amount of wall materials and slower manufacturing speeds of the microsphere are some of the disadvantages that make it difficult to expand the technology to an industrial scale. Literature research suggested that due to its versatility, low processing costs and ability to produce glassy extrudate with several formulations, extrusion has the potential to emerge as future technology. Also, the potential to encapsulate two or more sensitive ingredients in a matrix in a single step is beneficial for the development of a new product.

Conflict of interest

The authors declare that there is no conflict of interest.

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