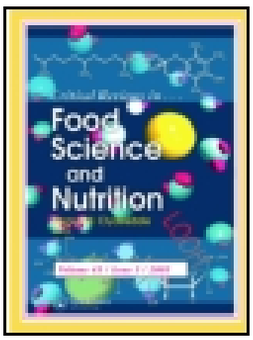

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REVIEW

Encapsulation of bioactive compounds by "extrusion" technologies: a review

Oluwaseun P. Bamidele and Mohammad Naushad Emmambux

Department of Consumer and Food Sciences, University of Pretoria, Hatfield, Pretoria, South Africa

ABSTRACT

This review described and explains the encapsulation of bioactive compounds by extrusion technologies. Bioactive compounds have many health benefits, but several drawbacks such as a high organoleptic impact due to the bitterness and acrid taste of some compounds, and limited shelf life reduce the utilization of bioactive compounds in food. Encapsulation of bioactive compounds to prevent their several drawbacks and to increase their utilization in food has been achieved by 'extrusion' technology. The extrusion technologies discussed in the review are hot-melt extrusion, melt injection extrusion process, co-extrusion, and electrostatic extrusion. Extrusion technology as a mode of encapsulation of bioactive compounds as increased the number of bioactive compounds that can be encapsulated. Also, extrusion technology helps to reduce the particle size of encapsulated bioactive compounds which increase their application in the food industry. The reduction in the particle size of the extrudate helps to increase the shelf life of encapsulated bioactive compounds and aid-controlled release in the targeted site in the body.

KEYWORDS

Atomization; complexes; health benefits; processing; screw speed

Introduction

Bioactive compounds are extranutritional constituents present in foods that can modulate metabolic processes and lead to the promotion of good health. Bioactive compounds exhibit health promoting effect like inhibition of enzymes, e.g., pancreatic lipases in obese patient (Correia et al. 2012); scavenging of free radical, and potential for prevention of formation of cancer cells (Mao et al. 2019).

There is major concern about the consumption of bioactive compounds when consumed as is. Bitter and acrid taste are generally the sensory characteristics of some of the bioactive compounds that are present in food materials, for example phytosterols and polyphenols in fruits and vegetables. Other limitations include; highly volatile, thermal instability, photodegradable and low bioavailability and bioaccessibility (Capelezzo et al. 2018). These limit the utilization of many bioactive compounds (Drewnowski and Gomez-Carneros 2000).

Degradation of bioactive compounds such as vitamin C that are sensitive to thermal and oxidative stress occurs during processing and possibly during their passage through the gastrointestinal tract (Dima, Dima, and Iordăchescu 2015). Bioactive compounds present high sensitivity to physicochemical factors. For example, they can become deactivated and inactive during food processing and storage, as well as during the digestion; thus, the necessity for protection.

Flexible encapsulation techniques have emerged, and efforts have been made to deliver these bioactive compounds at the target sites (Đorđević et al. 2015). Emulsion, suspension, particle, gel, hydrogel, microgel formation, liposome production, and coacervation have been designed for specific delivery systems consisted of food bioactive compounds (Anal, Shrestha, and Sadiq 2019). The smaller and the unique size of encapsulated bioactive compound in form of particle is advantageous in delivering bioactive compound to the active site when compared with other form of delivery systems. Particle below 100 nm, appear to get directly adsorb at the mucus (Anal, Shrestha, and Sadiq 2019). Biopolymers, consisting of polysaccharides and proteins, are used to create a transport system with pH-triggered launch specification (Zhang et al. 2016). Polysaccharides can intrinsically shape shell shape surrounding the core and lead to entrapment of respective bioactive compounds. While proteins on the whole have been applied to act as mighty emulsifiers, swelling and solubilization agents (Wang, Doi, and McClements 2019).

Bioactive compounds can be encapsulated to protect their activity during processing and storage, and increase their stability when transiting through the gastrointestinal tract (Dima, Dima, and Iordăchescu 2015). Encapsulation is a process in which bioactive compounds (active agents) are enclosed or coated by carrier (wall material or encapsulant) in order to form capsules or microcapsules at micrometre or nanometer scale. The bioactive compounds (active agent or ligands) are also referred to as core, fill, or internal phase, while the wall materials (coating or carrier material) are known as membrane, capsule, shell, matrix or external phase (Devi et al. 2017). Encapsulation process has been used in food and pharmaceutical industries to encapsulate bioactive compounds such as polyphenols, micronutrients, enzymes, and antioxidants by forming protective barriers against the

CONTACT Mohammad Naushad Emmambux naushad.emmambux@up.ac.za Department of Consumer and Food Sciences, University of Pretoria, Hatfield, Pretoria 0028, South Africa.

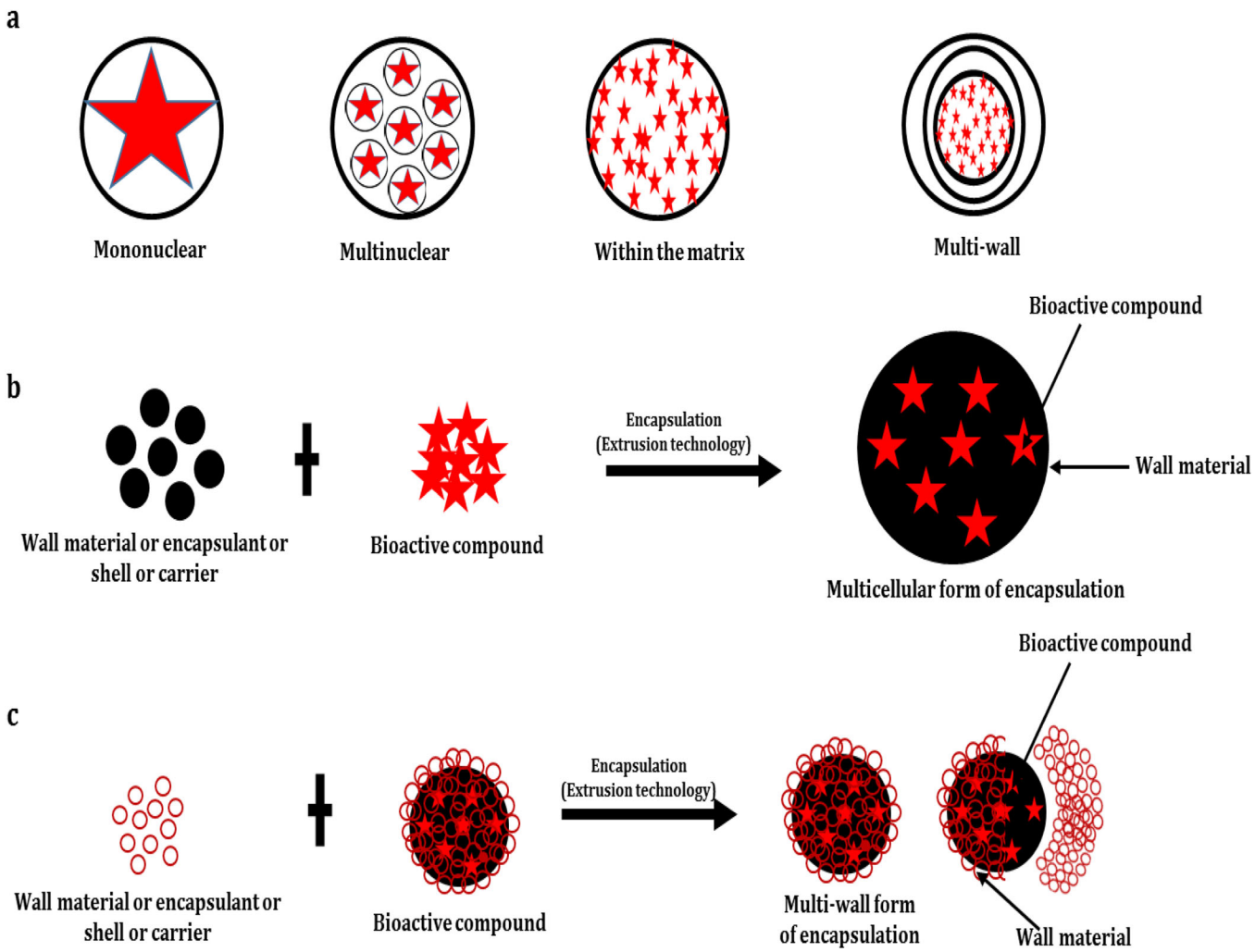

Figure 1. Illustration of (a) the modes of encapsulation of guest molecule in wall materials, (b) multicellular form of encapsulation and (c) multi-wall form of encapsulation.

light, oxygen, pH, moisture, heat, shear, or other extreme conditions (Devi et al. 2017).

There are so many processing technologies for encapsulation. These techniques include wet heat processing (pasting, kneading and co-precipitation), spray drying, spray cooling, extrusion coating, fluidized coating, liposome entrapment, coacervation, inclusion complexation, centrifugal extrusion and rotational suspension separation (Gibbs et al. 1999). Others are cocystallization, liposomes, nanoparticles, freeze drying and emulsion (Lengyel et al. 2019). One of the most recent method of encapsulating bioactive compounds is electrostatic extrusion (electrodynamic atomization) (Shishir et al. 2018).

Spray drying, the commercial method for encapsulation of bioactive compound has been reviewed extensively by various researchers (Drosou, Krokida, and Biliaderis 2017; Guerin et al. 2017; Arpagaus et al. 2018) compared to other method for example extrusion technology. In view of this, extrusion technologies will be considered in the review.

Encapsulation and mode of encapsulation

Encapsulation can be defined as a process to entrap one substance within another substance, thereby producing particles with diameters of a few nm to a few mm. The encapsulated substance is called the core material, the active agent, fill, internal phase, or payload phase. The substance encapsulating the encapsulant can be called the coating, membrane, shell, carrier material, wall material, external phase, or matrix (Zuidam and Shimoni 2010). In food industry, materials that are mostly used as encapsulating matrix are gum Arabic, dextrin, starches, maltodextrins, alginates, protein-based materials, cyclodextrins, and lipid components (Gibbs et al. 1999). Classification of particles can be done according to morphology and structure (regular and irregular). Both form of classification of particle determine the best method of encapsulation which suit each bioactive compound. Encapsulation can be classified based on morphology as mononuclear, multinuclear, within matrix and multi-wall form of encapsulation (Figure 1).

Mononuclear capsules are the most common type of microcapsule, consisting of a single core surrounded by a single shell. The core materials always occupy 10%–90% of the capsule volume which can be in any form (liquid or solid). This use of mononuclear form of encapsulation was to reduce cost and reduced energy, but most bioactive compounds (active agents) required more than single wall (encapsulants) because of interaction between the active agent and the wall materials (Jegat and Taverdet 2000).

Table 1. Overview of some of the different modes of encapsulation processes in food industries.

Mode of encapsulation	Type of food industry	Guest molecule	Encapsulant	References
Multinuclear	Food Beverages	Anthocyanin	Plury Quimica, Arabic Gum and γ-Cylodextrin	Burin et al. 2011
Multinuclear		Curcumin and Catechin	Water-in-oil-water emulsion	Aditya et al. 2015
Multinuclear		Lemon oil	Gum Arabic, maltodextrin and modified starch	Kausadikar, Gadhave, and Waghmare 2015
Mononuclear	Food Confectionary	Lycopene	Modified food starch (MFY-212, USA	Rocha et al. 2012
Within the matrix		Vegetable shortening	Oil-in-water emulsion	O'Brien et al. 2003
Multinuclear encapsulation	Meat and Poultry	Omega-3 fatty acid		Jimenez-martin et al. 2016
Within the matrix		Ascorbic acid	Polyvinyl alcohol (PVA)	Comunian et al. 2014
Within the matrix	Dairy Product	Flavourzyme	Calcium alginate	Anjani, Kailasapathy, and Phillips 2007
Multi-wall encapsulation		Probiotic bacteria in Ice-cream	Chocolate beads	Champagne et al. 2015

Also, controlled release of the active ingredient in some cases warrant more than single wall materials, so multinuclear form of encapsulation has been introduced. Multinuclear capsules have many active ingredients enclosed within a single shell in which the mode of formation may be a combination of various encapsulation methods (spray drying, spray chilling or extrusion cooking). In multinuclear capsules, the core materials are firstly coated with one wall material before encapsulating in another overall wall material (Jegat and Taverdet 2000). Conversely, in matrix-type (within the matrix) microcapsules, the core material is distributed homogeneously in the shell material. Matrix type microcapsules are most common in the pharmaceutical industry for drug delivery and presently used in food industries in encapsulating flavors. Multi-wall capsules consist of a single core surrounded by different wall materials to withstand different processing stages and achieve controlled release (Jegat and Taverdet 2000).

Table 1 shows some examples where the different modes of encapsulation are used in the food industry. It seems that multinuclear mode of encapsulation is most commonly use method of encapsulation in food industries. This is because in multinuclear mode of encapsulation, two or more bioactive compounds can be encapsulated in a single wall material, which reduce cost of production and reducing energy consumption during production. Multinuclear mode of encapsulation is used in food beverage industry, food confectionary and meat and poultry industry. Matrix type (within the matrix) mode of encapsulation is also common in food industry because it aids the controlled release of homogenously distributed bioactive compounds in the single wall material. Multi-wall mode of encapsulation is not common because it is requiring different type of wall materials which may interact during processing and require different encapsulation techniques.

Common encapsulants (wall materials) used in encapsulation of bioactive compounds

One of the major factors in a successful encapsulation process of bioactive compounds is selection of the encapsulant or wall materials (Đorđević et al. 2015). Table 2 shows different category and types of wall materials (encapsulant) used for encapsulation. Most wall materials used in encapsulation of bioactive compounds are biopolymers which can form matrix with bioactive compounds through molecular entanglement. Some of the wall materials like gums with low viscosity for example gum Arabic and this supports the use of such wall material at high concentration for higher encapsulation efficiency and number of bioactive compounds.

Suitable encapsulant must be safe, it should not be poisonous or cause deterioration to human health or hinder the release of the bioactive compound in the target site to be applied to food materials and must be biodegradable (Robin and Sankhla 2013). Many of the encapsulant used in encapsulating bioactive compounds (flavor compounds and antioxidants) aids their controlled release by diffusion, dissolution, osmosis and erosion (Singh et al. 2010). Encapsulant may be a good emulsifier, has low viscosity at high concentration, possess good dissolution and network-forming characteristics (Đorđević et al. 2015). They should have the ability to preserve bioactive compounds at different conditions of processing and storage (Shishir et al. 2018). In addition, encapsulating agents (encapsulant) should have the ability to overcoming acidic and enzymatic condition of the stomach and should have the ability to increase the adherence capability of bioactive compounds in target sites of the gastrointestinal tract (Shishir et al. 2018).

Food grade encapsulants are from carbohydrate, protein and lipids (Jain et al. 2016). Carbohydrate based encapsulants are polymers consisting of several bonds and are abundantly found in plants. The variation in chemical structures of carbohydrate encapsulants are responsible for their differences in solubility and water retention capacity (Fathi, Martín, and McClements 2014). Common carbohydrate used in encapsulation of bioactive compounds are starch, dextrin, maltodextrin, cyclodextrin, alginate, pectin, cellulose and gum (Shishir et al. 2018).

Protein base encapsulant have good functional properties (film formation, emulsification, gelation and water holding capacity) and they can be plant or animal based (Nesterenko et al. 2013). Proteins are recognized as encapsulant for hydrophobic base bioactive compound (Chen, Remondetto, and Subirade 2006). Animal proteins used as encapsulant are whey protein, collagen and gelatin. The use of animal proteins as encapsulant have some bias which may limit their uses. Such bias includes hypersensitive reaction (allergy) which is a large concern for its industrial application among consumers. In addition, markets for animal

Table 2. Category and types of encapsulants used in encapsulating bioactive compounds.

Category	Encapsulant types
Carbohydrates	Starch, modified starches, maltodextrins, cyclodextrins, cellulose, polysaccharides, chitosan
Proteins	Gluten, isolates (pea, soy), caseins, whey proteins, gelatin
Lipids	Fatty acids, alcohols, glycerides, waxes, phospholipids
Gums	Xanthan gum, gellan gum, seaweed (carrageenans and alginates), locust bean gum, guar gum, gum Arabic and karaya gum

Sources: Varzakas and Tzia (2014).

proteins are also confined by religious and other dietary requirement. The increase price of animal proteins, and customer issues over bovine spongiform encephalopathy (mad cow disease) which can be transmitted to human through ingestion of the diseased animal meat is of concern (Chang and Nickerson 2018). The process of extraction of gelatin which is obtained by acidic or alkaline hydrolysis of collagen is not considered as "green" chemistry because of these chemicals are not environment friendly.

For these reasons, plant proteins (derived from oilseeds, pulses, and cereals) are presently viewed as a "green" trend to be applied in food and pharmaceutical industries. They are recognized to be less allergenic compared to animal proteins, decrease cost, and readily available (Nesterenko et al. 2013). The application of plant proteins (soy protein, zein and gliadin) to replace animal proteins as wall materials in encapsulation of bioactive compound has become an increasingly interesting area for research (Tarhini, Greige-Gerges, and Elaissari 2017).

Lipids based encapsulant can be classified into polar and non-polar (Đorđević et al. 2015). Like protein base encapsulant, lipid based encapsulant have good functional properties (film formation, emulsification) to encapsulate bioactive compounds. Đorđević et al. (2015) reported that polar lipids particularly phospholipids are good surface-active compound and are suitable for stabilization, protection and controlled release of active compounds in active site. Most of the lipids encapsulant are hydrophobic which make encapsulation of hydrophobic bioactive compounds easy.

Extrusion as mode of encapsulation

Extrusion technology can be defined as a process which require forcing a material to flow under a variety of conditions (high and low temperature, high and low moisture and high and low speed) through an orifice with different diameter at a predetermined rate to achieve different types of products based on consumer requirements or producer specification (Alam et al. 2016). Extrusion is versatile with a wide range of products many which cannot be produced easily by any other process. Extrusion process involve high temperature (80–150 °C) applied for short time ($\leq$1 min) and this is one of the drawbacks of the technology since most of the bioactive compounds are thermolabile (George 2003).

Extrusion processing of food materials has emerged as an increasing number of vital manufacturing technique due to the fact it can function continuously with high output and lower fees of production. Extrusion technique is environmentally friendly because it does now not produce large manner effluents due to low-moisture process. Therefore, reducing water cure cost and stage of environmental pollution (George 2003). Extruder which is the machine that help with extrusion processes consist of one or two rotating screws tightly fitting within a barrel, at the end of which is the die (Ramachandra and Thejaswini 2015). The principle of operation for all extruder are similar and include raw materials feeding through a feeder into the barrel and screw(s) push it toward die and cutter. More specific extruders can be comprised of six parts, namely, preconditioning system, feeding system, screw(s), barrel, die and cutting mechanism. Furthermore, they can vary with respect to the screw, barrel, and die configuration, the selection of which depends on raw materials used and the desired final product (Ramachandra and Thejaswini 2015).

There are different extrusion technologies based on different type of extruder (machine use for extrusion) use, condition of extrusion and other parameters such as type of extrudate produce. Extrusion technology can be categorized into five types:

i. Hot-melt extrusion
ii. Melt injection
iii. Centrifugal/co-extrusion
iv. Electrostatic/electrospinning
v. Particle from Gas Saturated Solution (PGSS)

The first type of extrusion makes use of screw which may be single or double screws depending on type of final products that is desired. Also, it has a barrel which is connected to a thermostat which regulate the set temperature for extrusion process. The last three type of extrusions (Melt injection, Centrifugal and Electrostatic) are screwless form of extrusion which require the use of different types of forces and dies. Melt injection extrusion is a relatively low-temperature extrusion technology which utilize entrapping of bioactive compound in the molten wall materials as a mode of encapsulation. Centrifugal extrusion utilizes centrifugal force to push out the bioactive compound which filled inside the concentric orifices into the wall material that is in the outer circumference of rotating cylinder (Seth, Mishra, and Deka 2017). Electrostatic extrusion is a system with needle setup and applied with electrostatic charges. The bioactive compounds are forces through the extrusion needle by electrostatic force into a hardening solution and electrostatic charges pass through it (Low and Lim 2014), thereby encapsulating the bioactive compound. All the types of extrusions are discussed in this review.

Hot-melt extrusion

Hot-melt extrusion is a continuous screw-extrusion process. It is like melt injection extrusion; however, the major

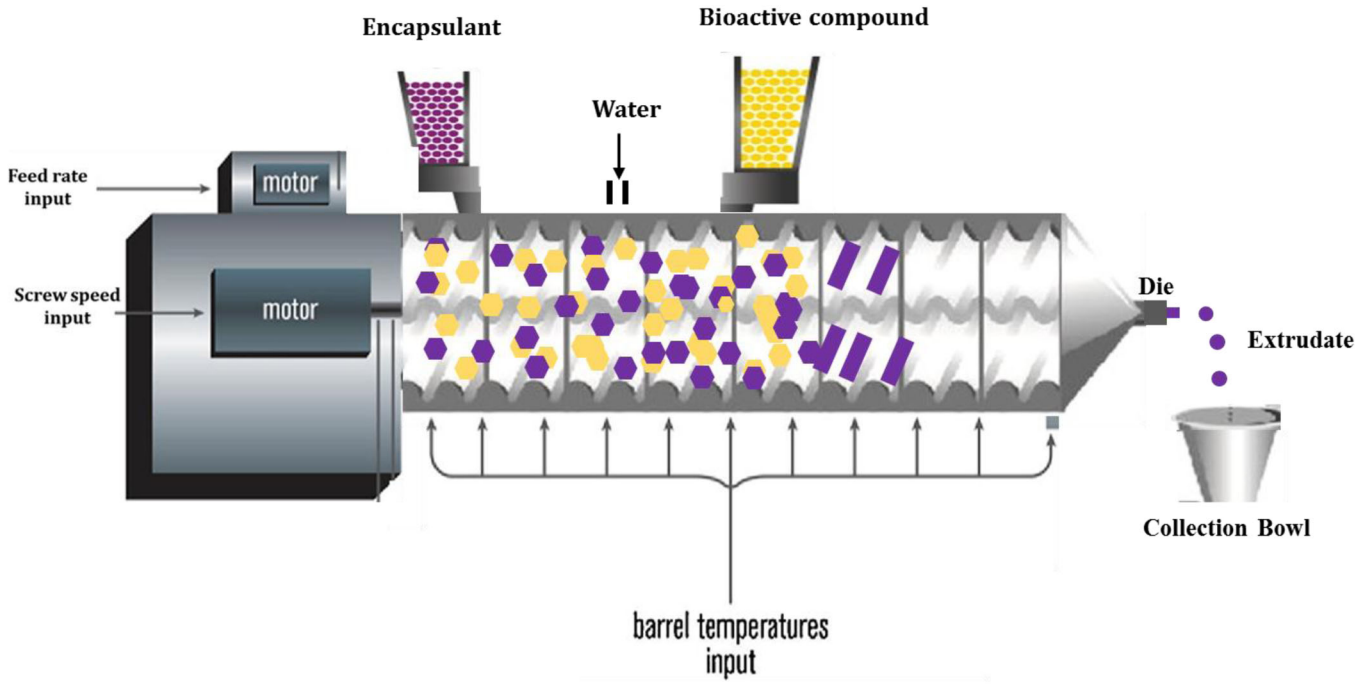

Figure 2. Schematic diagram of Hot-melt Extruder (Adapted from Patil, Tiwari, and Repka 2016).

difference is that melt injection is a vertical screwless process with surface washed particles while hot-melt extrusion is a horizontal screw process, with particles that are not surface washed (Bakry et al. 2016). Extruders used in hot-melt extrusion is in form of cylinder which contain thermomechanical mixers that consist of single or double screw equipped with self-wipe screws that favored encapsulation (Figure 2).

Depending on the extruder configuration, different barrel temperature, various inlets ports of liquids and solid feed, and screw profile can be set up depending on the active ingredient and the biopolymer matrix (Emin and Schuchmann 2013; Tackenberg et al. 2015). The process of hot- melt extrusion can be divided into 3 steps.

The introduction of the powder mixture of the encapsulant (e.g., starch, Protein or Cyclodextrins) into the extruder occur first. At this point, a plasticizer or additives may be added if necessary, before the introduction of active ingredients (Ubbink and Schoonman 2003). Encapsulation of bioactive compound in hot-melt extrusion occurs when the wall material is plasticized which enable easy interaction between the wall material and the bioactive compound. The bioactive compound gets encapsulated in the plasticized carrier matrix (e.g., starch). During the hot-melt extrusion, addition of plasticizer decreases the attraction between polymer chains of the encapsulant (wall material) thereby reduce the glass transition temperature (Bakry et al. 2016). Reduction in glass temperature makes the encapsulation occur at lower temperature, which has been reported favorable to protect heat sensitive bioactive compounds (Zuidam and Heinrich 2010).

The cooling of an extrudate can be done by air, or by a contact with a cold surface. Semi-crystalline polymers have a very sharp melting point and consequently a very sharp solidification temperature. Choosing the cooling rates is important when extruding semi-crystalline polymers to obtain a product with the required crystallinity. Rapid cooling would lead to the formation of small crystals and a relatively low overall crystallinity, whereas annealing would result in additional crystal growth and higher overall crystallinity. Thus, when a high crystallinity is preferred, the extrudate should be cooled slowly, with a rate determined by throughput rate and the temperature of the cooling medium (air, roll temperature of caterpillar) (Giles, Wagner, and Mount 2004).

Hot melt extrusion has been used for encapsulation of bioactive compounds and Table 3 shows some of bioactive compounds that have been encapsulated by the extrusion method Maltodextrin has been commonly used as wall materials in hot-melt extrusion when compared to corn starch, mannose and β-cyclodextrins, because it is cheap and readily available. Maltodextrin are starch hydrolysates that can withstand high temperature and have important matrix-forming properties as wall material (Ravichandran et al. 2014). Starch hydrolysates (e.g., maltodextrin) are normally applied to microencapsulation of oil soluble supplies such as flavor (Cinnamaldehyde, eugenol and Orange turpentine), vitamins (ascorbic acid and tocopherol) and oils (Lemon oil and Orange oil). They (maltodextrin) cannot be used alone due to lack of emulsifying capacity except in case of encapsulating water-soluble substances. They are mixed with other components imparting emulsifying capacity such as gums (e.g., gum Arabic) and water-soluble proteins (Forssel 2004).

Hot-melt extrusion is solvent free method with addition of limited amount of water. The process is continuous, fast with less unit operations. Excellent mixing of materials (encapsulants and the active ingredient), high throughput

Table 3. Summary of encapsulation of various bioactive compounds using hot-melt extrusion method.

Bioactive compound	Wall material (encapsulant)	Objective(s)	Extrusion conditions	Main findings	Reference
Ethyl butyrate (3.4); Lemon oil	Mannose/Maltodextrin	Utilization of volatile flavourant and essential oil in dry form	Barrel temperature – 60, 120, 130, 125 °C Screw speed – 60 rpm Die – 0.25 mm	Hot melt extrusion helps to encapsulate the volatile flavor in Mannose/ Maltodextrin	Saleeb and Pickup, 1989
Cinnamaldehyde, eugenol, nonanoic acid	Corn starch/ β-cyclodextrin	To evaluate the amount of flavor retained in extrudate during extrusion	Barrel temperature – 60, 120, 110, 100 °C Screw speed – 100 rpm Die – 7 mm	70% to 90% Cinnamaldehyde, eugenol, nonanic acid were encapsulated	Kollengode and Hanna 1997
Orange oil	Maltodextrin/corn sirup/ methylaldehyde	Encapsulation of orange oil in matrix to be stable in glassy state at ambient temperature	Barrel temperature – 50, 100, 150 °C Feed rate – 15 lb/h Water feed rate – 10 ml/min	Orange oil was encapsulated in matrix maltodextrins and was stable at glassy state	Porzio and Popplewell 1999
Orange turpentine and Tocopherol	Maltodextrin DE12 and Maltodextrin DE 17/sucrose	To improve the understanding of a counter rotatory twin screw extrusion process for encapsulating orange terpenes flavor	Barrel temperature – 105, 125, 145 °C Feed rate – 1.5, 2.25, 3.00 kg/ h Screw speed – 248, 497, 748 rpm Orange turpentine – 4%, 7%, 10% Barrel temperature – 105, 125, 145 °C Feed rate – 1.5, 2.25, 3.0 kg/h α-tocopherol – 4%, 7%, 10%	About 67% of the orange flavor was retained. The flavor loss correlated with insufficient mixing during extrusion	Tackenberg et al. 2015
Quercetin	Carnauba wax, Shella and Zein	To mask the bitter taste of quercetin	Barrel temperature – 80 to 90 °C Screw speed – 100 rpm	The amount of quercetin encapsulated varied from each encapsulant use. Zein encapsulated the highest amount of quercetin	Khor et al. 2017
Ascorbic acid	Glassy low-dextrose equivalent Maltodextrin matrix	To develop melt extrusion method for the encapsulation of ascorbic acid using maltodextrins as matrix material.	Barrel temperature – 80, 105, 115, 95 °C Screw speed – 200 rpm Feed rate – 2.0 kg/h Die – 1 mm	Ascorbic acid was encapsulated in the Maltodextrin matrix with yield rate of 96%	Chang et al. 2019
Carvedilol	Nicotinamide	To improve the solubility of Carvedilol by preparing cocrystals.	Barrel – 32, 85, 92, 90 °C Screw speed – 175 rpm Feed rate – 20 rpm Die – 1.7 mm	Carvedilol cocrystal was formed with distinct difference in morphological characteristic when compare to pure drug	Fernandez et al. 2019

and exposure to oxygen in extrusion channel is limited. The residence time is short and bioactive compound can be introduced at different points of the extrusion process with online monitoring. Bioactive compounds stabilizations are achieved with hot melt extrusion, which enhanced controlled release, taste masking and uniform particle size (500–1000 µm) (Zuidam and Shimoni 2010).

Temperature, screw speed, feed rate, moisture and die diameter determine the effectiveness of encapsulation of bioactive compounds by. The use of high temperature (e.g., 140 °C) which is too high for most of bioactive compound (omega-3 oils, vitamins and flavors), high screw speed (250 rpm) can cause loss of potency of the bioactive compounds, disintegration of crystalline structure of wall materials, thus causing interaction between the wall materials and bioactive compounds. Low feed rate allows high shear rate and residence time which increase starch melting and expansion. This allow more bioactive compound to form a complex with the amylose or get trap in the starch micropore. Increase in feed rate may have negative effect on the encapsulation efficiency by reducing the share rate and residence time which reduced starch melting and expansion.

Hot-melt extrusion is expensive compare to spray drying because it required various units. Its processing costs are estimated to be almost double in comparison to spray drying. Furthermore, the compound to be extruded must be able to tolerate high temperatures (Gately and Kennedy 2017). In addition, Hot-melt extrusion technology requires raw materials with high flow properties. The large size of extrudates (500–1000 µm), from Hot-melt extrusion, limits their use in food applications due to its negative effect on mouthfeel (Emin and Schuchmann 2013; Lakkis 2016) which may require another process for example milling before use. Orange turpentine was encapsulated in maltodextrin DE 17 at temperature range of 105 to 145 °C. Also, tocopherol was encapsulated in maltodextrin DE 12 at the same temperature range 105 to 145 °C (Tackenberg et al. 2015).

Melt injection extrusion process

Melt injection is a vertical, screwless extrusion process. In melt injection extrusion process, the bioactive compound is dispersed in heat (80–140 °C) melted carbohydrate (composed of sucrose, maltodextrin, glucose sirup, polyols, and/

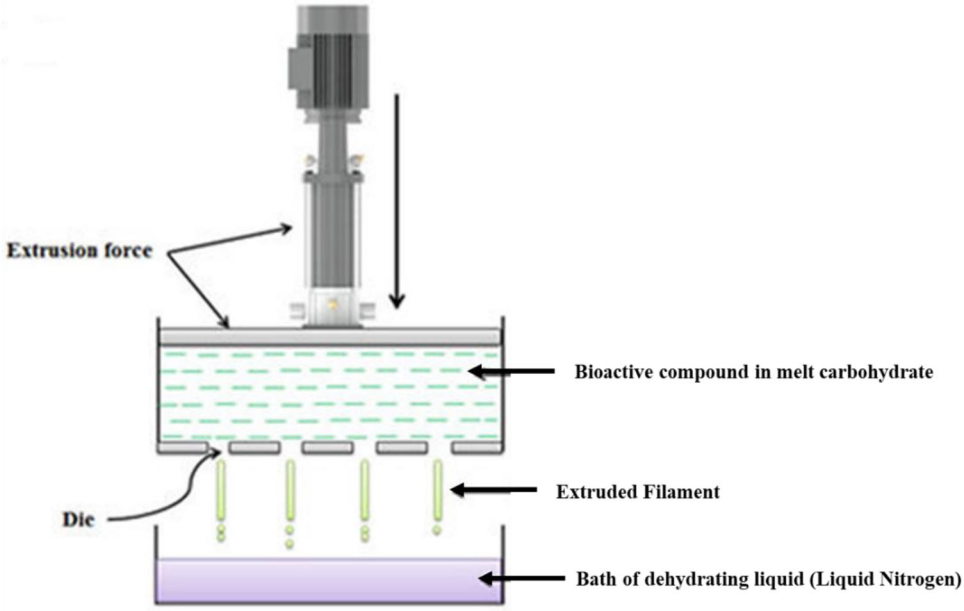

Figure 3. Schematic diagram of melt injection, a mode of encapsulation of bioactive compound (Adapted from Bakry et al. 2016).

or other mono- and disaccharides), and then pressed through one or more orifices (filter) into a bath of cold, dehydrating liquid (Liquid nitrogen or Isopropanol). The encapsulant or wall material solidifies with the liquid forming an encapsulating matrix, which encapsulate or trap the bioactive compound in the system (Figure 3). The wall material hardens on contact with the dehydrating solvent, thereby encapsulating the bioactive compound (Bakry et al. 2016). The size of the extruded strands is reduced to the appropriate dimensions inside the cold solvent during vigorous stirring, thereby breaking up the extrudates into small pieces. Any residues of active agent on the outside will be washed away by the dehydrating solvent. Encapsulates made by melt injection are water-soluble and have particle sizes from 200 to 2000 µm (Zuidam and Heinrich 2010).

Table 4 shows the reports of some researchers who have encapsulated bioactive compounds using melt injection extrusion technology. Flavor compounds (Lemon flavor and Cherry Peppermint flavor) are sensitive and volatile compounds with different physicochemical properties (Ubbink and Schoonman 2003), although flavor compound may suffer modification during encapsulation but the wall material used prevent the flavor compounds from the temperature and preserve the flavor compounds from being modified by heat. Spray-drying is commonly used as encapsulation technique for encapsulating flavor compounds, but 10% to 20% of the flavor compounds could not be encapsulated due to their nature (volatility). Melt injection extrusion techniques is suitable for encapsulating flavor compounds because it is flexible technique, with lower energy consumption and it allow better control of the state of the matrix (Porzio 2008). The use of certain biopolymer or mixture of biopolymers as being encouraged as wall materials in encapsulating flavor compounds because of simplicity of their release mechanism that is triggered by moisture or heat (Ubbink and Schoonman 2003). Most common wall material used in melt injection extrusion process are starch hydrolysate (Maltodextrin and β-Cyclodextrin), low molecular weight carbohydrate (sucrose, modified maize starches) as seen in Table 4. The viscoelasticity or "plastic" state of most wall materials help the dispersibility of the flavor compounds in the wall materials, thereby aids entrapment of the flavor compounds.

The shelf life of encapsulated oxidative compounds such as flavor and essential oil has been estimated to be five years using melt injection extrusion technology, while flavor or essential oil encapsulated through other means especially spray drying have a shelf life of one year (Gupta, Jadhav, and Singhal 2015).

Although the researchers did not mention any disadvantage of the melt injection extrusion method, it as being reported that melt extrusion produced large size of the granules (200–2000 µm) which limited its use in food application because of the small surface area and long diffusion path (Lakkis 2016). Thus, this limitation may prevent the use of melt extrusion for encapsulating bioactive compounds that require controlled release in human system. Melt injection extrusion process is batch process with limited amount of water. Short residence time for the bioactive compound with less unit operation. It is a small-scale equipment with better stability of encapsulated bioactive compound (Shishir et al. 2018).

One of the factors that limit the use of melt injection in food is that it requires solvent (Isopropanol) which is not encouraged in food production processes. It is a batch process, which means that continuous process for commercialization of encapsulating bioactive compound is a limitation by melt injection extrusion process (Emin and Schuchmann 2013).

Centrifugal extrusion (co-extrusion)

Centrifugal extrusion which is one of the types of co-extrusion is the third form of extrusion mode used in encapsulating bioactive compound such as Olive oil and Caffeic acid.

Table 4. Summary of encapsulation of various bioactive compounds using melt injection extrusion method.

Bioactive compound	Wall material (encapsulant)	Objective(s)	Extrusion conditions	Main findings	Reference
Lemon flavor	Modified waxy maize starch/modified maize starch	To encapsulate lemon flavor in different wall material (Octenyl succinic acid [OSAN]) in modified starch, and combination of dextrinized enzymatically hydrolyzed OSAN starch	Die opening – 0.78 mm Barrel temperature −113 °C Cold airflow – 13 °C	Two phases of encapsulation were achieved during encapsulation of lemon oil in the wall material	Zasypkin and Porzio 2004
D-limenione	Native corn starch/ β-cyclodextrin	To improve the retention of D-limenione during pre-added flavor starch extrusion. To determine the effect of processing condition on the flavor retention and extrudate properties	Barrel temperature – 133 to 167 °C Screw speed – 158 to 242 rpm Feed rate — 16 g/min Die opening — 2 mm	Barrel temperature and capsule level predominantly influences flavor retention and extrudate properties. Also, screw speed primarily affected the extruder performance	Yuliani et al. 2006
Cherry, Peppermint flavor	Sucrose/maltodextrins (52.8/47.2)	Investigate the effects of variations of temperature and water content on the release of flavor components that initially been encapsulated.	Barrel temperature — 30 to 100 °C Relative humidity – 11% to 97%	The largest amount of encapsulated cherry and peppermint flavor occurred when the matrix was above it glass transition temperature due to increase in water and temperature.	Gunning et al. 1999
Cherry Durarome (Benzaldehyde)	Sucrose/maltodextrins	To determine the physicochemical changes that occur to encapsulated cherry Durarome in an amorphous sucrose glassy matrix when exposed to humid environment	Barrel temperature — 50 °C Relative humidity — 6.48%	Humidification of encapsulated cherry Durarome increased the matrix moisture content and subsequently decreased the Tg which aid the release of the cherry Durarome (Benzaldehyde)	Bohn, Cadwallader, and Schmidt 2005
Ketoprofen	Klucel™ Hydroxypropylcellulose (HPC)	To improve the tabletting properties of Ketoprofen	Barrel temperature — 100 to 140 °C Screw speed — 50 rpm and 70 rpm Feed rate — 25 rpm Die — 1 mm	The tabletting properties of Ketoprofen was improved after encapsulation in KHPC.	Mohammed et al. 2012
Orange oil	Maltodextrin/trehalose/ lecthin/water 35.8/ 35.8/0.8/19.3	To encapsulate Orange oil for utilization in pharmaceutical	Barrel temperature — 110 °C Viscosity of the melt — 26.8 Pa s Die — 0.8 mm	Orange oil was encapsulated in the mixture ratio of the encapsulant.	Gregsin, Sillick, and Firmenich 2012

Co-extrusion is commonly used in production of core-shell particles with concentric feed tube through which the encapsulant (wall material) and bioactive compound are pumped separately through many nozzles (which may be stationary, rotating, vibrating or submerged in moving carrier and fluid) mounted on the outer surface of the extruder (Oxley 2012). The bioactive compound flows through the center tube, and encapsulant flows through the outer tube (Figure 4).

The mode of encapsulation adopted by centrifugal co-extrusion is based on the mechanism of interfacial polymerization. There are other mode of encapsulation of bioactive compound by co-extrusion technology which normally is not suitable for food applications due to toxic reaction between the wall materials and the bioactive compound (Garti and McClements 2012). Interfacial polymerization is a method that involves the utilization of a water-immiscible organic solvent to emulsify an aqueous mixture containing the enzyme and hydrophilic monomer. During interfacial polymerization, bioactive compound polymerizes with the encapsulant and form microcapsule. During co-extrusion, two reactive monomers (i.e., wall material and the bioactive compound) that are soluble in their respective miscible phases, come in contact at the interface with the help of centrifugal force acting on the surface of the extruder. The resulting polymerization reaction between the bioactive compound and wall material form a polymer microsphere (microcapsule) (Perignon et al. 2015). The resulting microcapsule (Figure 5) consists of liquid droplet of microcapsule enveloped with a polymeric membrane and can be collected on a moving bed (Zuidam and Shimoni 2010). Co-extrusion

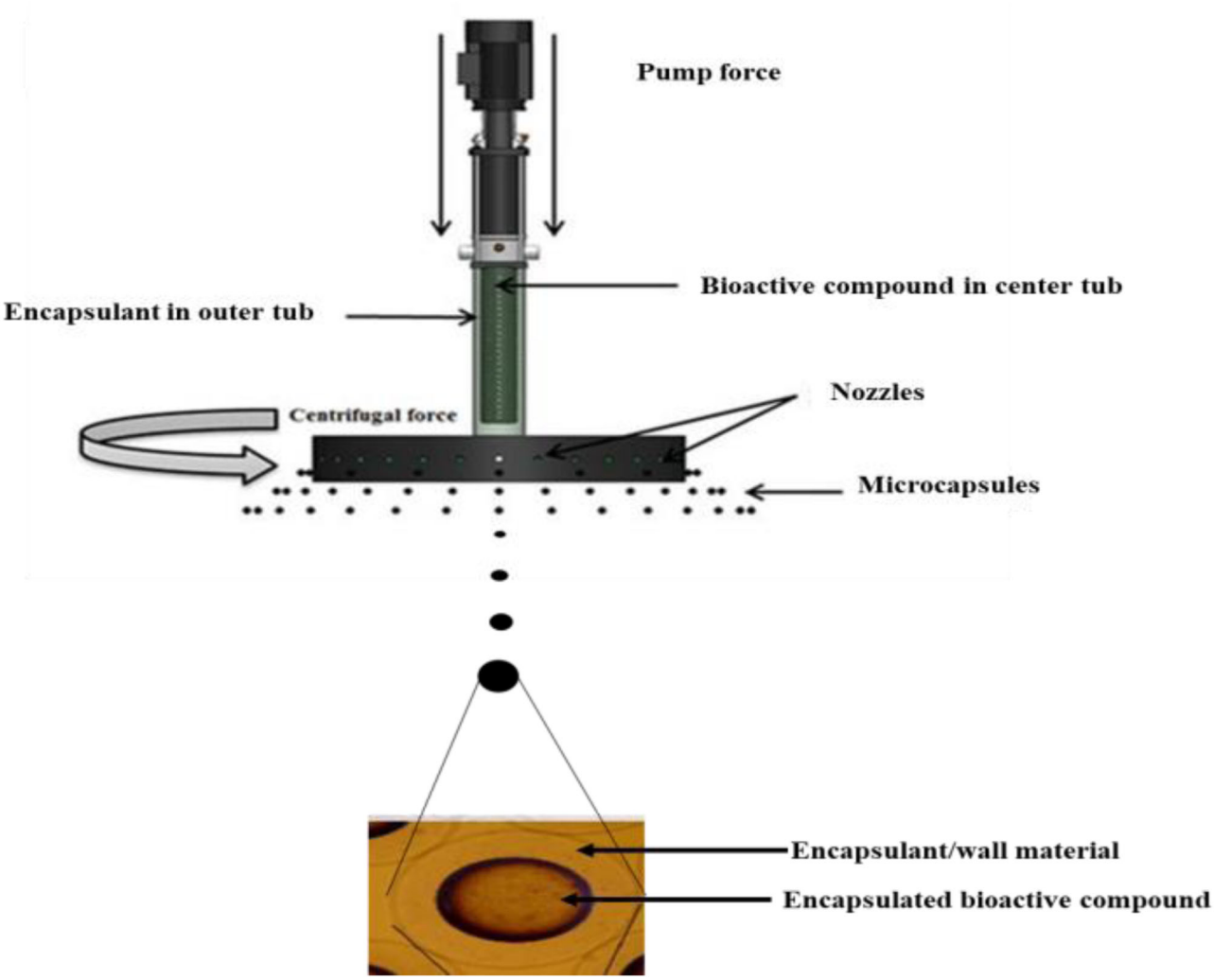

Figure 4. Schematic diagram of centrifugal extrusion (Co-extrusion) (Adapted from Bakry et al. 2016).

has been used in the encapsulation of olive oil, caffeic acid, essential oils, and protein (Sun-Waterhouse et al. 2011; Dolçà et al. 2015).

Table 5 shows summary of some of bioactive compounds that are encapsulated in different wall materials (encapsulant) by co-extrusion. Co-extrusion encapsulation technique are used for essential oil and probiotic bacterial because requires less heat for encapsulation compared to hot-melt extrusion. Alginates are used as wall material in encapsulating these materials (essential oils and probiotic bacterial). Alginates are anionic exopolysaccharides composed of variable proportions of 1,4-linked β-D-mannuronic acid (M) and its C-5 epimer α-L-guluronic acid (G). The alginate structure strongly impacts its material properties as wall materials in encapsulating bioactive compounds (Fata Moradali et al. 2015). Alginates are used because they absorb water quickly to form a viscous gum which aids encapsulation of bioactive compounds. Alginates gels are cold setting that when use as wall material in encapsulating probiotic bacterial increase viability of the bacterial and prevent it from harsh processing condition and that of stomach condition (Burgain et al. 2011). Most of the extrusion condition used for co-extrusion technology (Table 5), are of nozzle size ranged between 80 and 450 µm, frequency (vibrating) ranged of 500–3000 Hz and voltage ranged of 0.4 to 1.5 Kv.

The advantages of co-extrusion over other encapsulation methods e.g., spray drying is the stability of the encapsulated oil against oxidation and increase shelf life of encapsulated ligands by preventing oxygen contact during storage (Gouin 2004). Unlike spray drying process, in which the wall materials and the bioactive compound have short contact time (less 1 min) and mostly with mononuclear wall material, co-extrusion utilizes more than on wall material and the residence time between the wall material and bioactive compound is high (>1 min). It also helps to reduce the evaporation rate of essential oil during storage (Soliman et al. 2013). Co-extrusion is very expensive, and it produce large particle (150–8000 µm) that can limit its uses in various applications (Seth, Mishra, and Deka 2017). Large particle extrudate reduces the bioavailability, delivery properties and solubility of the functional (nutraceutical) food in target area due to small surface area and longer diffusion path length (Bakry et al. 2016).

One of the limitations of co-extrusion is the wall material (encapsulant) non-uniformity which means combination of one or two wall material with different components and

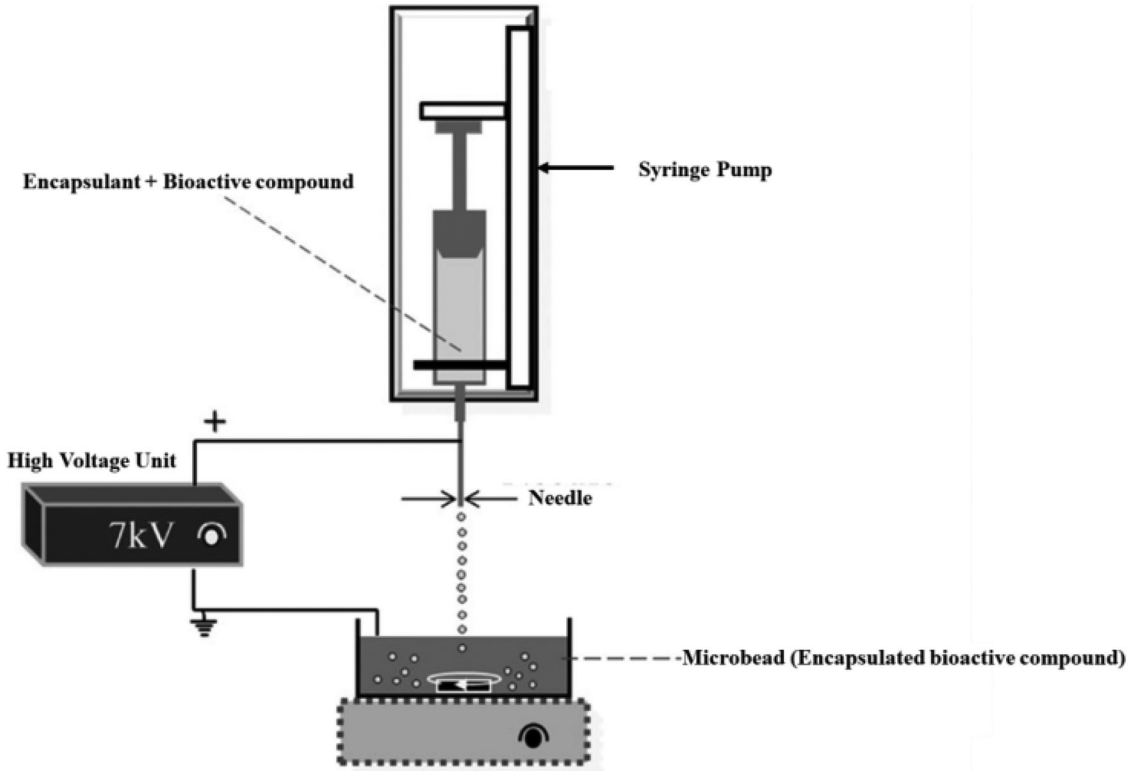

Figure 5. Schematic diagram of electrostatic extrusion (Balanč et al. 2016).

structure. Wall material non-uniformity can cause ineffective encapsulation and interfacial instability of bioactive compounds. Also, adhesion of different wall material if not properly chosen, can lead to separation during extrusion process. This lead to storage instability of bioactive compounds encapsulated by co-extrusion (Vynckier et al. 2014). Another limitation of coextrusion technology is that they need a second step to get the powder. The encapsulated bioactive compound must be dried in either in a mist chamber, powdered beds, flash volatilization chambers or liquid hardening baths (Garti and McClements 2012). Coextrusion technology are provided by OMIPA Extrusion Technology Company, Italy and CLEXTRAL Technology, France.

Electrostatic extrusion (electrospinning)

Electrostatic extrusion can also be called electro spraying or electrostatic atomization. Electrospinning and electrospraying are simple, one-step strategies for the manufacturing of micro and nanoencapsulates in dried form. This extrusion technique is suitable for encapsulation of warmth labile compounds as it is operated at ambient temperature and at atmospheric strain (Vega-Lugo and Lim 2009). Electrospinning and electrospraying makes use of biocompatible, biodegradable, meals grade, conducting polymeric factors as wall substances for encapsulation of bioactive compounds. Common protein specially based encapsulating substances used in electrospinning and electrospraying are whey protein isolate, whey protein concentrate (WPC), soy protein isolate, egg albumen, collagen, gelatin, zein and casein (Anu Bhushani and Anandharamakrishnan 2014).

The electrostatic charge repulsion customary in electrospraying prevents the agglomeration of sprayed droplets, resulting in tablets that are at least two orders of magnitude smaller than the traditional spraying process. These submicron particles can also be helpful to make bigger bioavailability of the bioactive compounds and improve sensory attributes of fortified meals products. Electrospinning and electrospraying can be considered a "bottom-up" method to collect complex fibrous and particulate materials beginning at the molecular level. To meet the end-use objectives, the meeting of these materials relies on the interaction between the polymer, solvent, and other the vital elements (e.g., bioactive, surfactant, spinning aid, precursors) in the spin dope solutions (Anu Bhushani and Anandharamakrishnan 2014).

This method is used in production of polymeric microspheres when polymers dropped into a hardening solution and electrostatic charges pass through it (Low and Lim 2014). The mechanism involves the application of electric field in between charged needle (positive) and collecting solution (Figure 5) to derange the liquid strand at the mouth of the needle, thus forming a charge stream of small drip (Balanč et al. 2016). Electrospinning of a polymer requires first solubilizing it in a well-suited solvent, alongside with additives, to shape a homogeneous spin dope solution. Alternatively, the polymer might also be melted using quite a number of heating techniques (e.g., heated enclosure, laser, heated extruder) and electrospun in a molten form (Zhou, Green, and Joo 2006). This process leads to the formation of very small particles (5 µm) which their presence does not affect quality and sensory characteristic of the food. However, the particle size of the polymer produce may range from 5 µm to 1000 µm (Liu et al. 2010). The particle

Table 5. Summary of encapsulation of various bioactive compounds using Centrifugal (Co-extrusion) extrusion method.

Bioactive compound	Wall material (Encapsulant)	Objective(s)	Extrusion conditions	Main findings	Reference
Kenaf oil seed (*Hibiscus cannabinus* L.)	Sodium alginate (1.5%) and alginate-pectin	To determine the encapsulation efficiency of Kenaf oil seed in Sodium alginate and alginate-pectin	Nozzle – 150–300 μm Vibration frequency – 500, 1000, 2000 Hz Voltage – 1.5 Kv	Sodium alginate encapsulated Kenaf oil seed with microcapsule size of 300 μm. Encapsulation of 74.25% was obtained for capsules with alginate-pectin.	Chew and Nyam 2016
Canola oil	Alginate	Investigating the feasibility of encapsulating antioxidant-fortified Canola oil through Co-extrusion	Nozzle – 200 μm Vibrating frequency – 1750 Hz Voltage – 1.5 kv Flow – 30 ml/min	It was reported that encapsulation conditions (core-shell flow rate and shell wall formation) influenced the retainment of the Canola oil	Wang, Waterhouse, and Sun-Waterhouse 2013
Canola oil	Alginate + Quercetin	Storage stability of Canola oil under pH 3 and temperature (20 and 38 °C)	Nozzle – 200 μm Vibrating frequency – 1750 Hz Voltage – 1.5 kv Flow – 30 ml/min	The bead appearance size and surface characteristic did not change at pH 3 for 2 h. Addition of quercetin increased the stability of the and suppressed Canola oil deterioration during storage.	Waterhouse, Wang, and Sun-Waterhouse 2014
L. plantarum ATCC8014, *L. paracasei* ML33, *L. pentosus* ML82	Cheese serum, serum permeated, sodium alginate and pectin	Investigating the morphological characterization of bioactive ingredient at storage; microcapsule resistance to different pH levels; potential acidification of microcapsules.	Nozzle – 80 μm Vibrating frequency – 1740 Hz Voltage – 950 mV Flow – 5 ml/min	Lactobacillus acidophilus (CSCC 2400 and CSCC 2409) were microencapsulated in calcium and sodium alginate. The increase in cell load during encapsulation increased the number of bacterial survivors at the end of 3 h incubation in simulated stomach conditions	Eckert et al. 2018
L. casei (DSM 20011). *L. reuteri* (DSM 20016) and *L. delbrueckii subsp. Bulgaricus*	2% alginate	To investigate the morphological characterization of microcapsules and microcapsule resistance to different pH levels	Nozzle – 450 μm Vibrating frequency – 500 to 3000 Hz Voltage – 250 mV Flow – 10–30 ml/min	*Lactobacillus acidophilus* 547 and *Lactobacillus casei* 01 were encapsulated and the both survive the simulated acidic condition of gastric juice (pH 1.55) while *Bifidobacterium bifidum* ATCC 1994 did not survive the acid condition of gastric juice.	Olivares, Silva, and Altamirano 2017

size of the polymer microsphere depends on various operating variables, the system effects and the properties of the polymer (Bugarski et al. 2005).

Electrostatic extrusion is useful in encapsulation of some bioactive compounds. Prüsse et al. (2008) encapsulated ethyl vanillin in alginate gel microbeads with electrostatic extrusion. About 10% (wt/wt) of ethyl vanillin was encapsulated in about 2% (wt/wt) alginate. *Thymus serpyllum L.* was encapsulated in calcium alginate bead using various methods including electrostatic extrusion. Stojanovic et al. (2012) reported that the bead size of calcium alginate was about 730 μm for electrostatic extrusion and encapsulation efficiency of *Thymus serpyllum L.* in calcium alginate varied between 50% to 80% depending of the method. Belščak-Cvitanović et al. (2011) encapsulated six polyphenolic antioxidants from medicinal plants using electrostatic extrusion. Encapsulation efficiency of 80%–89% was recorded for all the extract in alginate chitosan.

The oxidative and light stability of β-carotene was improved when encapsulated in electrospun zein fibers having minimal and most cross-sections of 540 nm and 3580 nm respectively (Fernandez, Torres-Giner, and Lagaron 2009). Also, curcumin encapsulated in zein nanofiber (310 nm) possessed increased free radical scavenging power and controlled released (Dhandayuthapani et al. 2012). The stability of (-) epigallocatechin gallate (EGCG) was more desirable via electrospinning in zein nanofibers (472 ± 46 nm) and via growing old the fiber for at least one day at 0% relative humidity below ambient temperature (Li, Lim, and Kakuda 2009). Furthermore, a composite combination of zein, polyethylene oxide (PEO) and chitosan was used to encapsulate a-tocopherol in mucoadhesive fibers (449 ± 126 nm) and the electrospun fibers were determined to decorate the muco adhesivity (Wongsasulak, Pathumban, and Yoovidhya 2014) thereby aiding in increased bioaccessibility and bioavailability of nutrients.

Table 6. Summary of encapsulation of various bioactive compounds using electrostatic extrusion method.

Bioactive compound	Wall material (encapsulant)	Objective(s)	Extrusion conditions	Main findings	Reference
ThymumSerpyllum L	Calcium alginate	Encapsulation of Thymumserpyllum L using electrostatic extrusion in order to produce dosage formulation that contain poly phenolic compound	Stainless steel needle – 22 gauge Voltage – 6.5 Kv Flow rate – 25.2 ml/h	Thymumserpyllum was encapsulated in Calcium alginate which improve the stability of the polyphenolic compound in food	Stojanovic et al. 2012
Aqueous Carqueja extract	Calcium alginate and Inulin	Encapsulation of aqueous carqueja extract in calcium alginate and insulin was carried out to determine the effect of insulin of type of microbead form	Stainless steel needle – 23 gauge Voltage – 6.3 Kv Flow rate – 39.3 ml/h	The aqueous carqueja extract was encapsulated in calcium alginate and insulin by forming a microbead. The insulin reduced stiffness of the hydrogel and protect the microbead from collapse	Balanč et al. 2016
Bifidobacterium lactic B107 and *Lactobacillus acidophilus*	Alginate, cellulose acetatephthalate (CAP) and Xanthan gum	To determine the stability of encapsulated *Bifidobacterium lactic* B107 and *Lactobacillus acidophilus* during storage and gastric acid	Stainless steel needle – 21 gauge Needle diameter – 50 µm Stirring rate – 250 rpm	Encapsulated Bifidobacterium lactic and Lactobacillus acidophilus were stable at 5 °C for 9 months of storage with improved resistance to gastric acid condition	Albertini et al. 2010
Bifidobacterium infantis ATCC 15677	Gellan gum and Xanthan gum	Influence of encapsulation of *Bifidobacterium infantis* ATCC 15677 in Gellan gum and Xanthan gum on stability during storage in simulated gastric juices at pH 2.5, 2.0 and 1.5	Stainless steel needle – 21 gauge Needle diameter – 1.50 µm Stirring rate – Gentle stiring	Encapsulated *Bifidobacterium infantis* ATCC 15677 survived simulated gastric juices gastric juices at pH 2.5, 2.0 and 1.5	Sun and Griffith 2000

Lević et al. (2015) investigated the encapsulation of a flavoring compound, the D-limonene essential oil. The results of this study showed that alginate particles carrying this compound are appropriate for the incorporation of D-limonene while maintaining its thermal stability under certain conditions. Balanč et al. (2016) reported encapsulation of aqueous Carqueja extract in calcium-alginate-insulin. They reported that the microbead produced by electrostatic technique had an average diameter from 50 µm to 830 µm depending on the portion of insulin added.

Electrostatic extrusion or electrospinning has been found useful in food industry because of small particle size of polymeric microspheres encapsulated bioactive compound it produced (Low and Lim 2014) through the mouth of the needle. Table 6 shows some bioactive compounds that are extracted from plant (*Thymum serpyllum L* and *Aqueous carqueja*) and probiotic bacterial (*Bifidobacterium lactic* B107, *Lactobacillus acidophilus* and *Bifidobacterium infants* ATCC 15677) that were encapsulated by electrostatic extrusion.

Electrostatic extrusion has been used to encapsulate probiotic cells and bacteriocins. The viability of *Bifidobacterium* traces (*Bifidobacterium* animal is subsp. *lactis* Bb12) was improved by means of encapsulation in polyvinyl alcohol (PVA) electrospun nanofibers (150 nm). The encapsulation did not have an effect on the viability of the cells and the viability of encapsulated bacteria. During storage, encapsulated bacteria was stable than that of non-encapsulated bacteria after forty days (40 days) of storage at room temperature (20 °C) and a hundred thirty days (130 days) at refrigerated temperature (4 °C) (López-Rubio et al. 2009).

Like co-extrusion, alginates are used as wall material in electrostatic extrusion, but in combination with other biopolymer (Xanthan gum, Gellan gum and Inulin), which aids easy encapsulation of the bioactive compounds. Gums (Gellan gum and Xanthan gum) are used as wall material in electrostatic extrusion because they have excellent emulsifying properties, film forming ability, good ability to produce small particles and low viscosity at high concentration (Atefi, Mohammadi, and Nayebzadeh 2016). Calcium alginate increase the cell activity of the encapsulated plant extract such as adhesion and proliferation (Gokarneshan 2019). Stainless needles are used in electrostatic extrusion to aids production of smaller particle size microbeads (Table 6) and the voltage use is higher (6.3 and 6.5 Kv) when compare to co-extrusion technology.

Electrostatic extrusion help in production of much smaller microbeads with conventional needles and the process can be easily controlled by varying the applied potential (needles size, applied voltage, encapsulant concentration and encapsulation efficiency). Electrostatic extrusion also helps in encapsulating high concentration of bioactive compounds.

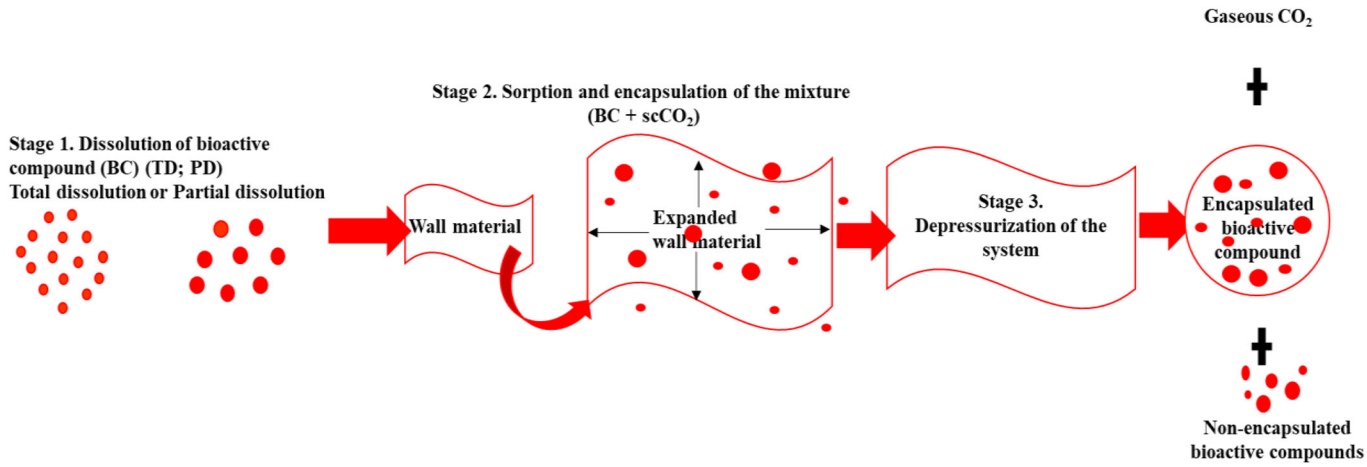

Figure 6. Schematic diagram showing three steps of encapsulation of bioactive compounds by PGSS method (Adapted from Rojas et al. 2020).

One of the limitations of electrostatic extrusion is that encapsulation of bioactive compound cannot be achieved in large scale. Also, the encapsulant (wall material) that can be used is limited (alginates and gums) (Knezevic et al. 2002).

The advantage of electrostatic extrusion over others (co-extrusion and melt injection) is that it does not require temperature or a second step to dry the encapsulated product. It is very versatile in terms of sizes, wall polymers and storage stability (Anu Bhushani and Anandharamakrishnan 2014). Electrostatic extrusion requires low energy thereby save cost and capable of delivering encapsulated bioactive compounds with unique characteristic. One of the main drawbacks is the scale-up or low throughput (i.e., it cannot be used industrially). This drawback restricts their commercial exploitation at a large scale (Anu Bhushani and Anandharamakrishnan 2014).

In other to address the drawbacks, researchers are suggesting modification of the structural aspects of the setup by using multi-needle arrangement for encapsulation of bioactive compounds. Also, the use of immobilized enzymes in bioreactor for continuous operation are being investigated (Anu Bhushani and Anandharamakrishnan 2014).

Particle from gas-saturated solution extrusion

The particle from gas-saturated solution (PGSS) extrusion process utilize the supercritical fluid like CO_2 to encapsulate bioactive compounds. A fluid is said to be supercritical, when its pressure and temperature exceed their respective critical value (Tc- critical temperature and Pc- critical pressure) (Parhi and Suresh 2013). The superficial carbon dioxide assisted encapsulation process has been used to develop active food packaging materials and encapsulate bioactive compounds (Rojas et al. 2020). In supercritical fluid encapsulation method, the solvent strength, excessive dispersibility and low interfacial surface tension of the fluid are of important that allow the encapsulation of a bioactive compound into the encapsulant matrix. In this process can occur with three steps:

i. dissolution,
ii. sorption of the mixture
iii. depressurization of the structure.

The first step of the supercritical fluid encapsulation technique includes the dissolution of the bioactive compounds to be encapsulated in the $scCO_2$. The conferment of the bioactive compound is performed during this step by using capability of its chemical absorption within the wall material, via an encapsulation mechanism called molecular dispersion (Rojas et al. 2020).

The next step (second stage) of the supercritical fluid encapsulation technique considers the sorption of the mixture (bioactive compound and $scCO_2$) in the encapsulant (wall material) (Fig. 6). The molecular dispersibility of a bioactive compound in the encapsulant shape is increased due to the encapsulant swelling and plasticization impact induced with aid of the high-pressure CO_2, whose extent relies upon by and large on the chemical nature of the encapsulant and CO_2 conditions (temperature and pressure) (Shen et al. 2008). In this stage, the encapsulation of the ligand (bioactive compound) in the encapsulant matrix is finished typically all through the depressurization of the structure (final step) by capability of its bodily entrapment in the encapsulant matrix. This mechanism of encapsulation, acknowledged as settling (deposition), is particular for fluids (supercritical), with potential that relies upon wall material swelling and on the solubility of the bioactive compound in the CO_2-phase (Rojas et al. 2020).

The final step of the supercritical fluid encapsulation technique is the depressurization of the structure. As referred to above, this step is related with an encapsulation mechanism unique for supercritical fluids. For bioactive compounds with low accord in the direction of an encapsulant, a quick depressurization rate could amplify the quantity of bioactive compounds encapsulated due to the speedy reduce of the solvent strength of CO_2 (Di Maio and Kiran 2018).

The supercritical fluid encapsulation technique has been meted out in three modes: Steady mode, Active mode and Semi-active mode. The most used mode is the steady mode. Figure 6 indicates a standard outline of the experimental setup for a supercritical batch encapsulation technique. Firstly, the amounts of bioactive compound and encapsulant are placed in

Table 7. Summary of encapsulation of various bioactive compounds using particle from gas saturation solution.

Bioactive compound	Wall material (encapsulant)	Objective(s)	Extrusion conditions	Main findings	Reference
Lavandin oil	Modified starch	To encapsulate lavandin oil in modify starch using supercritical fluid method	Pressure – 10, 11 and 12 MPa Temperature = 40 to 50 °C Depressurization rate – 0.07 to 0.15 MPa m Time – 2 h	Lavandin oil was encapsulated in the modify starch. The distribution of the oil depends on the density of CO_2 and the supercritical phase.	Varona, Martin and Cocero 2011
Flax oil	β-glucan aerogel	To evaluate the amount of flax oil encapsulated in β-glucan aerogel with increase in processing time	Pressure – 15 and 30 MPa Temperature = 40 to 60 °C Time – 28 h	Flax oil was encapsulated in β-glucan aerogel with increase in the amount encapsulated when the processing time increased.	Comin, Temelli, and Saldaña 2012
Cinnamaldehyde	Cassava starch	To experiment different processing conditions on encapsulation of cinnamaldehyde in cassava starch	Pressure – 15 and 25 MPa Temperature = 35 °C Time – 3 to 15 h Depressurization rate – 1 to 10 MPa m	All the processing conditions aids the encapsulation of cinnamaldehyde in cassava starch.	de Souza et al. 2014
Vitamin K3 and D3	Alginate aerogel sphere	To encapsulate Vitamin K3 and D3 in alginate aerogels.	Pressure – 15 and 25 MPa Temperature = 35 °C Time – 3 to 15 h Depressurization rate – 1 to 10 MPa m	About 12% of the vitamins (K3 and D3) were encapsulated in alginate aerogel sphere.	Pantić, Knez, and Novak 2016; Pantić, Kotnik, et al. 2016
Olive leaf extract and Caffeic acid	Polyethylene terephthalate (PEP)/ Polypropylene (PP) film	To determine the amount of the extract encapsulated in PEP/PP film. Also, to determine the antioxidant properties of the released extract	Pressure – 10, 20, 30 and 40 MPa Temperature = 35 to 55 °C Time – 22 h Depressurization rate – 1 to 10 MPa m	Small amount of the extracts were encapsulated and the antioxidant properties of the extracts were not affected by encapsulation	Cejudo Bastante et al. 2017
Phytosterol	Nanoporous Starch Aerogel (NSA)	To improve the water solubility of phytosterol by encapsulation in Nanoporous Starch Aerogel	Pressure – 45 MPa Temperature = 70, 90 to 120 °C Time – 3 h	Phytosterol was encapsulated in NSA. About 9.9% was encapsulated with size ranged between 59 to 87 nm.	Ubeyitogullari and Ciftci 2017
Cinnamaldehyde	Poly Lactic Acid (PLA) film.	To encapsulate cinnamaldehyde in PLA film using 2 pressure conditions (9 and 12 MPa) and 3 depressurization rates.	Pressure – 9 and 12 MPa Temperature = 40 °C Time – 3 h Depressurization rate – 0.1, 1 and 10 MPa m	Higher pressure and lower depressurization rate favor encapsulation of cinnamaldehyde in PLA, with improve thermal and structural properties.	Villegas et al. 2017

the identical encapsulation reactor, but bodily separated by means of a steel or paper filter in order to avoid direct contact between them. Then, pre-heated CO_2 is added into the thermostatic high-pressure cell (system) and the gadget is pressurized up to the working strain (Comin, Temelli, and Saldaña 2012). The dissolution of the ligand (bioactive compound) and therefore the sorption of the mixture (bioactive compound and $scCO_2$) are execute concurrently.

Active (mode) process, the dissolution of the active compound can be carried out both in the identical encapsulation reactor or in a preceding dissolution reactor. In the last option, the sorption of the mixture (bioactive compound and $scCO_2$) in the wall material begins solely when the saturated mixture (bioactive compound and $scCO_2$) can enter in the encapsulation phase with a consistent CO_2 glide (Comin, Temelli, and Saldaña 2012).

Belizón et al. (2018) reported the supercritical fluid extraction of polyphenols from mango leaves and their subsequent encapsulation in Polyethylene terephthalate (PET)/ Polypropylene (PP) films. In another work, Cejudo Bastante et al. (2017) reported the collection of an olive leaf extract and it's in addition encapsulation in PET/PP films. Where the bioactive compound to be encapsulated is a supercritical extract, it will be better to carry out the encapsulation process via an operation mode that blends the supercritical fluid extraction and encapsulation procedures to attain enormous reduction in cost of energy, time and raw materials. Table 7 shows summary of bioactive compounds encapsulated using supercritical fluid extrusion method.

The advantages of particle from gas saturated solution (PGSS) extrusion process are that the process is simple leading to low cost with wide range of applications. It can also

be used with suspension of active ingredient(s) in polymer(s) or other wall materials leading to composite particles. When in contrast to Rapid expansion of supercritical solution (RESS), particle from gas saturated solution (PGSS) utilize smaller solvent and gas which make it suitable to system inorganic powder to pharmaceutical compounds. One of the disadvantages of PGSS is that bioactive compound need not be soluble in the supercritical fluid ($scCO_2$) (Weidner, Petermann, and Knez 2003).e excessive know-how about binary systems (addition of pure bioactive compound with CO_2) has encouraged the find out about the supercritical fluid encapsulation method for the production of bioactive materials to be carried out often using a pure materials. It is important to know that a bioactive compound could be greater environment friendly containing a compound instead of a pure material. Therefore, carrying out research into multiphase mixture is important in order to assist future work in a variety of purposes of the supercritical fluid encapsulation process (Rojas et al. 2020).

Concluding remarks

Extrusion technology has provided many solutions to problems encountered during the encapsulation of bioactive compounds in food and pharmaceutical industries. Extrusion technology has helped in producing small particle size extrudate which can be used in food application. Also, with small particle size extrudate, the control release of encapsulated bioactive compounds may have target delivery for efficiency and activity. Extrusion technology as a mode of encapsulation of bioactive compounds has improved the shelf life of bioactive compounds.

Extrusion technology help in encapsulating probiotic bacteria in different encapsulants. Co-extrusion and electrostatic extrusion technology are used with alginate and calcium carbonates being among the common encapsulants. Probiotic bacteria encapsulated through extrusion technology remains active when release. Encapsulation of probiotic bacteria has improved their use in the manufacturing of nutraceutical food in food industries.

Carbohydrate (Starch, Maltodextrins, Gum Arabic, Alginate, and Cyclodextrins) as being found useful as wall material during extrusion technology. For flavor compounds, maltodextrins and cyclodextrins are commonly used because of the easy encapsulating bioactive compound during extrusion. Alginates (Sodium alginate) is commonly used for encapsulating probiotic bacteria during extrusion. Although, all extrusion technology is useful for encapsulating bioactive compounds, it seems that some (electrostatic and co-extrusion) cannot be used for commercial purposes because the utilize syringe which cannot handle large quantity and electricity which may increase the cost of production.

Hot-melt extrusion has been found useful in food industries to produce nutraceutical foods that contained encapsulated bioactive compounds. Hot extrusion is used to encapsulate heat-stable bioactive compounds in different wall materials. Electrostatic extrusion is commonly used in the laboratory and it is not a continuous process but batch process which help to encapsulate heat-labile bioactive compound with smaller particle size.

Encapsulation of bioactive compounds using extrusion technology is also an option when compared with spray drying as a mode of encapsulation. There is limited wall material that can be used for spray drying, while that of extrusion technology are many. The shelf life of the encapsulated bioactive compound using spray drying is limited when compared with encapsulated bioactive compounds using extrusion technology. It is important to know that extrusion technology has improved the application of different bioactive compounds and help in producing nutraceutical foods.

Due to drawbacks in some of the extrusion technology (electrostatic extrusion) that cannot be operated on commercial level, the use of multi-needle arrangement has been suggested for mass production of encapsulated bioactive compound. Also, extrusion technology as being suggested to help in encapsulating mixture of bioactive compounds in wall materials. These are the future research in extrusion technology.

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Disclosure statement

The authors declare that there is no conflict of interest concern this review work.

ORCID

Oluwaseun P. Bamidele http://orcid.org/0000-0003-2815-8558
Mohammad Naushad Emmambux http://orcid.org/0000-0002-9377-6056

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