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Review

Microencapsulation as a Noble Technique for the Application of Bioactive Compounds in the Food Industry: A Comprehensive Review

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Abstract: The use of natural food ingredients has been increased in recent years due to the negative health implications of synthetic ingredients. Natural bioactive compounds are important for the development of health-oriented functional food products with better quality attributes. The natural bioactive compounds possess different types of bioactivities, e.g., antioxidative, antimicrobial, antihypertensive, and antiobesity activities. The most common method for the development of functional food is the fortification of these bioactive compounds during food product manufacturing. However, many of these natural bioactive compounds are heat-labile and less stable. Therefore, the industry and researchers proposed the microencapsulation of natural bioactive compounds, which may improve the stability of these compounds during processing and storage conditions. It may also help in controlling and sustaining the release of natural compounds in the food product matrices, thus, providing bioactivity for a longer duration. In this regard, several advanced techniques have been explored in recent years for microencapsulation of bioactive compounds, e.g., essential oils, healthy oils, phenolic compounds, flavonoids, flavoring compounds, enzymes, and vitamins. The efficiency of microencapsulation depends on various factors which are related to natural compounds, encapsulating materials, and encapsulation process. This review provides an in-depth discussion on recent advances in microencapsulation processes as well as their application in food systems.

Keywords: encapsulation; natural molecules; functional properties; encapsulation techniques; food systems; functional foods; healthy foods

1. Introduction

Consumers are now well aware of the importance of natural food ingredients due to their health benefits over synthetic ingredients [1]. The natural ingredients are of animal, plant, or microbial origin and are comparatively safer for human consumption than

synthetic formulations. There are many bioactive ingredients containing antioxidant, antimicrobial, flavoring, coloring, nutritional, and therapeutic properties, and they are widely used for preservation in the food industry [2]. These compounds exert their effects on the food systems and provide better quality and stability to the food products. The main problem encountered during the processing and storage of these natural bioactive compounds is their degradation.

Microencapsulation is the process by which these natural bioactive compounds are encapsulated to protect them from degradation during various processing and storage conditions [3]. Under this process, the micro-sized particles are covered by wall material/encapsulant/shell material, which protects and isolates these particles from ambient conditions [4]. The proper selection of a solvent medium varies with the solubility of core material and encapsulants [5]. The properties of these coatings or matrices decide the release kinetics of bioactive or functional ingredients under specific conditions. In simple words, it is a process by which active ingredients are packaged within a secondary (wall) material [3].

Generally, microencapsulation comprises four steps, viz., formation of the core and encapsulants, incorporation, and solidification [4]. The particle size of the core material is reduced and solubility is increased by milling, grinding, or melting in liquid. For soluble core materials, a suitable emulsifier or surfactant is added to increase stability, whereas, for gaseous materials, porous solids or matrices are used [6]. However, solidification and exposure to high temperature may cause degradation, diffusion or leakage of heat-sensitive materials and lower retention of the core material. To minimize the loss of core materials, freeze drying, coacervation, spray chilling, or cross-linking can be useful techniques for encapsulation [3,5]. This can be performed by applying chemical treatments such as gelation, physical treatments such as heating or cooling, or a combination of chemical and physical treatments to obtain the desired level of stability of encapsulates [7].

There are many conventional and advanced microencapsulation techniques that are suitable for a variety of food ingredients [3]. However, the efficiency of microencapsulation, the stability of capsules, the stability of matrix properties, and the release kinetics of active ingredients are important attributes that depend on the process as well as on the materials of encapsulation. The molecular structure, interaction of natural ingredients with the coating material, molecular weight, polarity, and solubility are important factors that affect the encapsulation efficiency, the stability of the capsules, and their release kinetics. This review provides in-depth knowledge about recent advances in microencapsulation strategies.

2. Natural Bioactive Compounds for Food and Therapeutic Applications

Natural bioactive compounds are obtained from some natural sources. These natural bioactive compounds are of two types: (a) those which show functional effects in the food systems, such as antioxidants, antimicrobials, flavor enhancers, colorants, and stabilizers, and (b) those which show functional effects in consumers, such as antioxidant, anti-inflammatory, antidiabetic, and antihypertensive compounds. Some natural antimicrobials are very effective in controlling both, the pathogenic and spoilage microorganisms [8]. Essential oils [9,10] and plant extracts [11–17] have been used for improving the oxidative and microbial shelf-life of foods. Natural bioactive compounds prevent oxidative processes in food products [18]. This strategy is vital since the oxidative reactions are the most important degradative process in foods, including both lipid [18,19] and protein oxidation [20], which result in important losses of food quality (chemical composition and physicochemical characteristics) and also produce a significant reduction of nutritional properties of foods. Moreover, the encapsulation of natural colorants is also a promising strategy in order to limit the use of synthetic dies in the food and pharmaceutical industries [21]. Thus, natural functional therapeutic compounds are either used directly or incorporated in the food systems to provide necessary therapeutic

effects in consumers. Similar to the natural ingredients for food applications, these natural functional therapeutic compounds are preferred over synthetic [22].

The use of natural bioactive compounds as food ingredients or therapeutic agents has some limitations because their bioactivity is affected by processing and storage conditions [23] since these compounds are degraded with high temperature, pressure, light, extreme pH, etc. [24,25]. Physical and chemical transformations are other problems that affect their stability and bioactivity [3]. Therefore, a high concentration is required to obtain the similar antimicrobial effect in food products as in in vitro tests; however, a higher concentration may impart negative consequences in food quality (estrange flavors, colors, appearance, etc.). Hence, the direct incorporation of these natural compounds in the food product formulations results in their degradation and transformation at faster rates, which substantially destroys their bioactivity [3,25]. However, the encapsulated compounds have benefits on human health, since this process increases both the bioaccessibility and bioavailability of these compounds, which also improves their absorption efficiency [21].

On the other hand, many phytochemicals have been shown to have anticancer, anti-inflammatory, and antidiabetic potential, which are being used for the development of functional food products. Their stability, bioactivity, as well as their bioavailability, are important attributes that decide their therapeutic potential [26]. The liberation, absorption, distribution, metabolism, and elimination of these compounds are required in order to obtain their health benefits. Thus, the bioavailability of different therapeutic compounds depends on different factors. For instance, the phenolic compounds have lesser stability, poor solubility, and slow intestinal permeability; hence, they have poor bioavailability when they are used in free form. The pH range of various sections of the gastrointestinal tract also differs, hence, the free forms of bioactive compounds have different bioavailability, and this also affects their degradation rate at different sections of the intestinal tract. Anthocyanins are very sensitive to pH and temperature changes, the bioavailability of carotenoids largely depends on particle size and processing conditions, the antimicrobial and antioxidant potential of natural extracts are diminished by the gastrointestinal environment. Thus, the bioactive molecules' stability and their biological activity can be improved by encapsulating them into colloidal particles [3]. In this regard, several researchers studied the microencapsulation of active compounds obtained from multiple sources using different encapsulation techniques (Table 1).

Table 1. Studies on microencapsulation of different natural bioactive compounds.

Class	Bioactive Compounds	Source	Technique	Ref.
Essential oils	Rosemary oil	Commercial	Emulsion/freeze drying	[27]
			Spray drying	[28]
	β-Caryophyllene	Fruits of *Pterodonemarginatus*	Spray drying	[29]
	Kaffir lime oil	Commercial	Spray drying	[30]
	Peumus boldus oil	*Peumus boldus* Mol (boldo)	Complex coacervation	[31]
	Citronella essential oil	Citronella (*Cimbopogon winterianus*)	Complex coacervation	[32]
	Clove oil	Clove	Solid liposomal encapsulation	[33]
	Essential oil	Red and white thyme oil	Complex coacervation	[34]
	Healthy oils	Fish oil and olive oil	Spray drying	[18]
		Tigernut, chia, linseed	Spray drying	[35]
Natural colorants	Saffron	Stigma of *Crocus sativus*	Spray drying	[36]
		Commercial	Freeze drying	[37]
	Astaxanthin	Commercial	Spray drying	[38]
		Commercial	Complex coacervation	[39]
	Betalains	Cladodes of *Opuntiaficus-indica*	Spray drying	[40]

	Betanin	Beetroot	Liposomal encapsulation	[41]
	Lycopene	Watermelon, tomato	Liposomal encapsulation	[42]
	Astaxanthin-oleoresin	*Haematococcuspluvialis*	Spray drying	[43]
	β-carotene	Commercial	Spray drying/Freeze drying	[44,45]
	Betacyanin	Beetroot juice	Spray drying	[46]
	Carotenoids	Paprika (*Capsicum annum* L) oleoresin	Spray drying	[47–49]
	Soy lecithin	Soy	Liposomal encapsulation	[50]
	Anthocyanins	Jaboticaba (*Myrciaria cauliflora* (Mart.) O. *Berg*) pomace	Freeze drying	[51]
		Blueberry	Freeze drying	[52]
		Echium amoenum petal	Spray drying	[53]
		Sour cherries (*Prunus cerasus* L)	Freeze drying	[54]
		Grape skin	Emulsification/gelation and Spray drying/Freeze drying	[55]
		Black raspberry	Complex coacervation	[56,57]
		Black rice (*Oryza sativa* L.)	Double emulsion/complex coacervation	[58]
		Pomegranate powder	Spray drying	[59]
		Cornelian cherry fruits	Spray drying/Freeze drying	[60]
Flavors	Lactoferrin	Camel milk	Emulsion	[61]
Enzymes	Thiol	Hydrolysate of mushroom protein	Spray drying	[62]
	Mixed components	Roasted coffee oil	Miniemulsion/solvent evaporation	[63]
	Cellulases and Hemicellulases	Commercial	Spray drying	[64]
Vitamins	Folic acid	Commercial	Nano-spray drying/electrospraying	[65]
	Vitamin E		Spray drying	[66]
	Vitamin E and C		Spray drying	[67]
	Beta-carotene		Liposomal encapsulation	[68]
	Vitamin E and C		Liposomal encapsulation	[69–71]
	Vitamin B_{12}		Emulsion	[72]
Phenolics	Resveratrol	Commercial	Emulsion/freeze drying	[73]
	Rutin	Commercial	Supercritical fluid	[74]
			Liposomal encapsulation	[75]
	Hydroxycinnamic acid	Commercial	Ionic gelation	[76]
	Caffeic acid, carvacrol, thymol		Liposomal encapsulation	[77]
	Resvertol, methoxy pectin and epigallocatechin gallate	Orange juice	Liposomal encapsulation	[78]
	Procyanidins	Litchi pericarp	Liposomal encapsulation	[79]
	Polyphenol rich extract	Jabuticaba (*Myrciaria jabuticaba* (Vell.) Berg) peel	Freeze drying	[80]
		Laurel (Litsea glaucescens)	Spray drying	[81]
		Pomegranate flavedo	Freeze drying	[82]

Mate leaves (*Ilex paraguariensis*)	Spray drying	[83]
Red chicory and red cabbage	Spray drying	[84]
Oat bran	Complex coacervation	[85]
Morinda citrifolia leaf extract	Spray drying	[86]
Avocado peel extract	Complex coacervation	[87]
Cocoa extract	Ionic gelation	[88]
Yarrow extract	Supercritical fluid technology	[89]
Raspberry leaf, hawthorn, ground ivy, yarrow, nettle, and olive leaf extracts	Extrusion technology	[90]
Aqueous extract of *Piper sarmentosum*	Extrusion technology	[91]
Aqueous extracts of *Ilex paraguariensis* (yerba mate)	Extrusion technology	[92]
Ethanolic grape extract	Supercritical fluid technology	[93]

Another problem associated with the use of natural bioactive compounds either as food ingredients or as therapeutic agents is their distinct flavor and aroma. As aforementioned, any little deviation in the absorption rate affects the bioavailability of therapeutic compounds profoundly because the absorption rate of some phenolic phytochemicals such as anthocyanins, isoflavones, stilbenes, flavonols, and flavanones varies only from 1 to 9% [94]. Even a slight increase or decrease in their concentration may disturb the flavor and overall acceptability of the food products. This has posed a major obstacle for the development of food products with higher quality attributes and functional properties [3,25,95]. Thus, there is a need to develop novel delivery systems for these natural bioactive compounds into the food systems. These delivery systems must overcome all the limitations associated with the use of natural bioactive compounds as described above. One approach for the development of such a delivery system is the microencapsulation of natural bioactive compounds or active ingredients (Figure 1) in a capsule or matrix. This is basically done to protect the core bioactive material from degradation and maintain their stability, bioactivity, and bioavailability to a great extent [4,25].

Furthermore, a regulated and controlled release of the core material can be ensured to sustain the bioactivity for a longer duration [3]. This technique is immensely useful in reducing the evaporation as well as transfer rate of core material, thus masking the intense flavor and aroma, which may otherwise affect the acceptability of food products. This technique is also used for keeping food ingredients that are cross-reactive separate. However, the efficiency and success of microencapsulation techniques are affected by many critical factors associated with the process as well as with the properties of bioactive compounds along with the encapsulating materials.

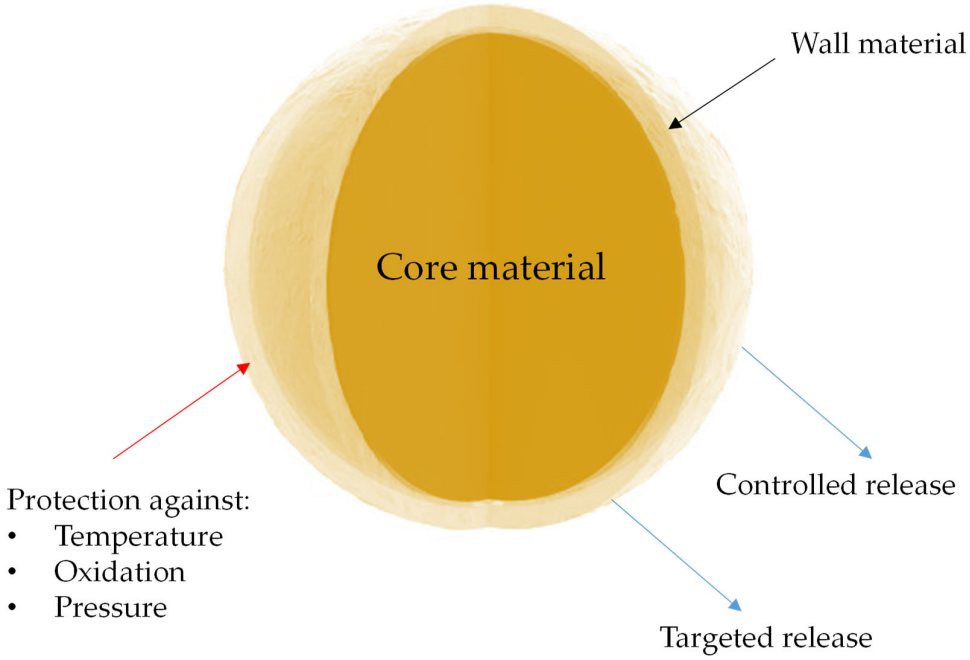

Figure 1. Basic concept of microencapsulation for bioactive compounds.

3. Microstructure of Encapsulate

During microencapsulation, microcapsules are produced containing active ingredients as core materials surrounded by encapsulated materials such as solids, liquids, or gases. The size of microcapsules varies from 1–1000 μm depending upon the microencapsulation techniques applied, as well as core materials, wall materials, processing parameters, etc., while when the size of these microcapsules falls lower than 1 μm, they are known as nanocapsules [3,96]. For food applications, generally speaking, microcapsules should be lower than 100 μm in order to avoid impacting the mouthfeel of the food product [25]. Additionally, microparticles can be classified into three types on the basis of structure such as microcapsules with a single core covered by wall material or cores dispersed in continuous matrix or multilayer or multishell complex microcapsules [97]. The size of encapsulates varies with the method used for preparation such as spray coating resulting in encapsulate size ranges from 5–500 μm, co-extrusion results with encapsulating size of more than 200 μm, micelle-based encapsulation with 5–20 nm size, liposome-based encapsulation with 10–1000 μm, coacervation with 1–500 μm size, emulsion-based encapsulation with less than 200 nm size, and ionic gelation encapsulation with a 100 nm–100 μm size of encapsulates [98]. The size of encapsulating has no direct effect on encapsulation efficiency, but it affects the controlled release of active ingredients in addition to physical and chemical properties of encapsulants, as smaller sizes result in larger contact areas for enzymes or releasing media and thus the release of larger amounts as compared to encapsulates with larger sizes [5]. The shell or wall material should preferably be insoluble, non-reactive to active ingredients, have excellent film-forming, and have the desired protective properties against various environmental conditions [3,99].

The encapsulation method, properties of core and encapsulant materials, and load quantity result in the different microstructures of encapsulates. The microstructure of capsules has an impact on the efficiency and retention of the core material and the encapsulation efficiency and regulates the release of active ingredients [25]. The microstructure of encapsulates varies in the external structure such as rough, porous, hollow, cracked, shrunken, etc., or the internal structures such as the arrangement and configuration of the core material [100]. Recently, several researchers noticed the higher

encapsulation efficiency, larger shell thickness, more compact shell, and slower release of ingredients in the case of encapsulates with a lower core load, whereas weak shells such as porous or cracked structures lowered protection and resulted in an early release of encapsulated ingredients. The higher load in microcapsules leads to thinner shells, resulting in lower protection and increased release, whereas fewer core materials in microcapsules usually results in a thicker wall and in the better protection and slower release of core materials [101–103].

4. Recent Progress in Microencapsulation Techniques for Natural Bioactive Compounds

A number of techniques are available for the microencapsulation (Figure 2) of natural bioactive compounds (core materials) and can be broadly divided into three types: chemical, physicochemical, and physical methods [3]. These techniques vary according to the type of core materials and their further application in the food system. The basic aim of these techniques is to obtain the maximum encapsulation efficiency, process reproducibility, and the desired release kinetics, as well as to eliminate the aggregation and adherence of capsules. The selection of microencapsulation methodology varies with the size, bioavailability, and biodegradability of microparticles, physical and chemical attributes of the core and coating materials, the application of the microencapsulated ingredients, and the release of active ingredients [67]. Khoshnoudi-Nia [104] reviewed the different methods applied for fish oil microencapsulation and noted spray drying, freeze drying, and hydrodynamic methods as the major methods applied by the industry, in addition to electrospun fibers and electro sprayed capsules and other methods such as liposomes, gelation, supercritical antisolvents, and spray freeze drying. Additionally, the selection of proper wall materials for the encapsulation process is vital for achieving the desired results [105].

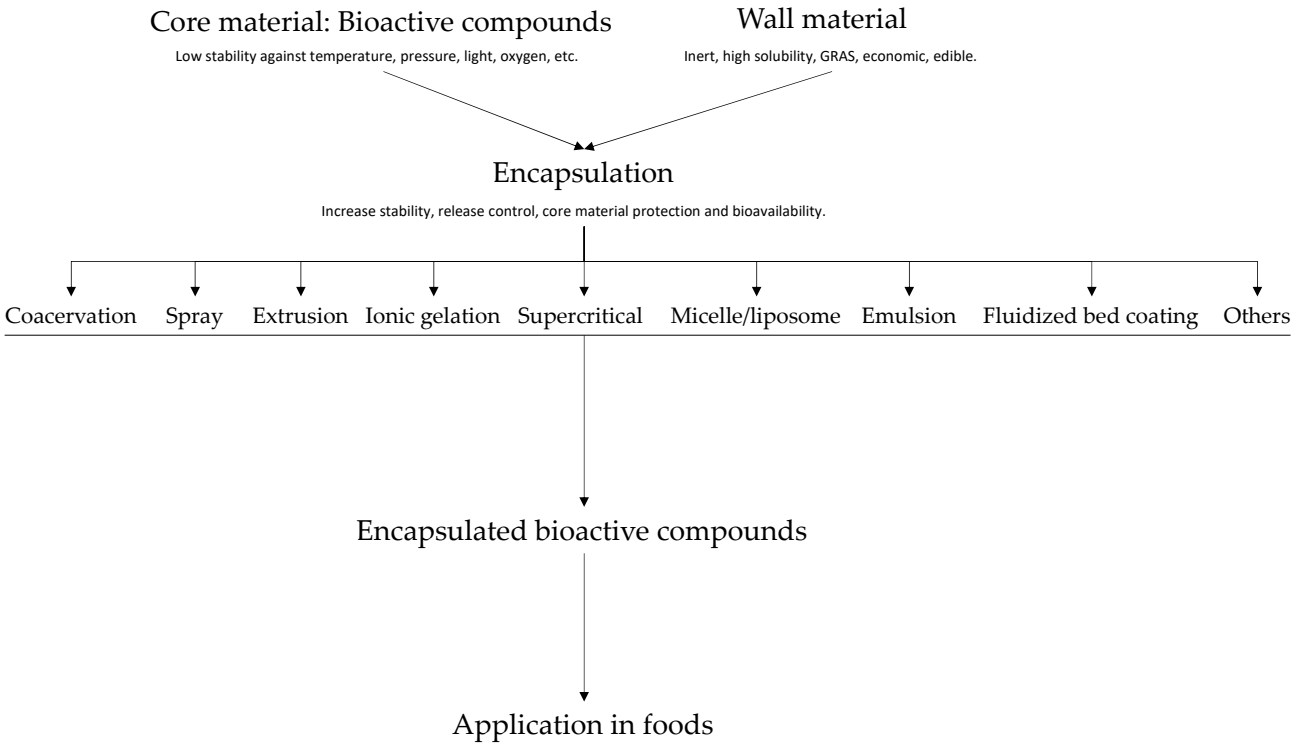

Figure 2. Microencapsulation techniques for bioactive compounds and food application.

4.1. Spray-Based Techniques

This is the most common and widely adopted technique for the encapsulation of bioactive compounds. It is carried out by spray drying, spray congealing, or spray freeze drying methods [4]. The spray drying method is a popular and well-established method in which the core particles are dispersed in a polymer solution, and spraying is performed in a hot chamber [25]. This is an easy to operate, flexible, fast, and economical technique to produce microcapsules [25,106]. This procedure consists of an initial dissolution and emulsification of dispersion of the core compound in the carrier material solutions, which is sprayed into a hot chamber [25]. The solvent evaporates, and the wall material coats around the core, leading to the formation of microcapsules of the polynuclear or matrix type. Therefore, this technique consists of the atomization of an emulsion or solution of core-carrier agents and their posterior recovery of the dehydrated microcapsules. Highly encapsulation-efficient and good-quality microcapsules are generally obtained with this procedure. However, this technique also has some limitations, such as the high temperatures that are required to evaporate the solvent, the use of water-soluble carriers, etc. [25].

To solve some of these problems, the spray-freeze drying technique was proposed. The spray-freeze drying method is suitable for heat-sensitive core materials. Under this method, solvent is immediately sublimed (lyophilized) from atomized droplets. Droplets are sprayed into a cold chamber filled with liquid nitrogen. These droplets are sublimed at ambient pressure in a fluidized bed, thus producing encapsulates with a matrix microstructure. Higher core loads lead to thinner shells, resulting in leakage of core material droplets on the surface of encapsulates, as core droplets adjacent to the shell are more susceptible to degradation [107,108]. The spray drying technology produces encapsulates with very high solubility and reconstitution attributes, low water activity, and easier transport and storage, but the non-availability of perfect wall materials and the agglomeration of microcapsules are still limiting the use of this technology [109]. Ozkan et al. [96] observed the loss of several heat-labile active ingredients during the operation of spray drying at a high temperature process. In addition, the yield during the spray drying process remains low due to the loss of dried particles in the drying vessel, poor control over the size of droplets, the constraints of common wall materials, which causes higher evaporation losses and less dry matter, low glass transition temperature, and the stickiness of materials with high sugar contents [110].

The choice of wall material for microencapsulation is very crucial to obtain higher encapsulation efficiency and higher stability of microcapsules [3,25]. Since almost all the spray drying processes in the food industry are carried out in the aqueous feed formulations; the wall material essentially should be soluble in water [4]. The most commonly used wall materials are low molecular weight carbohydrates, milk proteins, gelatin, and hydrocolloids such as gum acacia [3].

Microencapsulation also masked the unpleasant smell of these extracts, making them suitable for nutraceuticals and food applications. Alginates as a carrier and delivery system for natural bioactive compounds have been studied extensively. It can be cross-linked with various cations to modify the characteristics of matrices for multiple applications including food. Even some enzymes such as cellulases and hemicellulases have been encapsulated using alginate matrices [64].

Gums possess excellent film-forming and emulsion stabilization properties, hence they are used during the microencapsulation of a variety of bioactive compounds. Gum arabic produces stable emulsion with many oils at a wide pH range and hence is a better wall material for the encapsulation of lipids. It was used for the microencapsulation of rosemary essential oil [28] with powder recovery, surface oil, oil retention, and hygroscopicity varying from 17.25–33.96%, 0.03–0.15%, 7.15–47.57%, and 15.87–18.90%, respectively. Vitamin E and provitamin A were extracted from red pepper byproducts using the supercritical fluid extraction technique, and the extracts were microencapsulated by spray drying with gum arabic as a wall material to prevent their

degradation during the subsequent storage [111]. Cortés-Rojas et al. [112] used cellulose as the wall material for encapsulation of *Bidenspilosa* leaf extract at 13.37% concentration. The powder had a favorable particle size, residual moisture content, solubility, flow properties, and drying efficiency.

Since no single compound can provide all the essential characteristics to the microcapsules with desired quality attributes, a combination of different compounds as the wall material is also a better option to improve the encapsulation efficiency and stability of microcapsules. A combination of gum arabic and maltodextrin as the wall material has been studied [29] for the microencapsulation of essential oil from the fruits of *Pterodonemarginatus* using the spray drying technique. These authors noticed that a ratio of 1:3:3.6 of essential oil: gum arabic: maltodextrin had shown around 98.63% encapsulation and entrapment efficiency, with the best protection of β-caryophyllene, which is the major component of these essential oils. Adamiec et al. [30] studied the effects of spray drying temperatures and wall materials (konjac glucomannan and gum arabic) on the functional properties of kaffir lime oil microcapsules. The combination of konjac glucomannan and gum arabic enhanced the yield and retention of total oil in the microcapsules more than any of the wall materials used alone.

Spray chilling or spray congealing is a low-cost convenient microencapsulation technique that is also an easy process to scale up. It does not require organic solvents or the application of high temperature; hence, it is suitable for thermo-sensitive bioactive compounds such as enzymes, vitamins, and probiotics. In the spray coagulation technique, alginate is frequently used as a thickening and stabilizing agent, and its coagulation is facilitated by external gelation using calcium chloride or internal gelation using calcium carbonate as a calcium source. Martins et al. [113] carried out the microencapsulation of phenolic extracts from *Rubusulmifolius* flower buds in an alginate-based matrix. The yogurt containing the microcapsules of *Rubusulmifolius* flower buds extract showed a slightly higher antioxidant activity than other samples.

The matrix structure and presence of core droplets on the surface of encapsulates during spray-drying, leads to the rapid release of core materials [114]. Furthermore, this release becomes more rapid (which may not be desired in some cases) in water and in digestive enzymes by using water-soluble encapsulants such as modified starch, protein, maltodextrin, whey protein [103]. Tan et al. [101] encapsulated natural antimicrobial agents, e.g., eugenol by spray drying process by using whey protein isolates or lecithin in maltodextrin. The core active ingredient released rapidly and significantly inhibited *Escherichia coli* and *Listeria innocua* within 30 min. Alternatively, a controlled faster release at the beginning followed by a slower release at the later phase of active core ingredients from encapsulates formed by the spray drying process could be achieved by utilizing low-solubility encapsulants [101,115] ethyl cellulose and copolymer Eudragit RS 100 [116] cross-linked casein [117]. This targeted and controlled release could be due to the release of core droplets present at encapsulates at the beginning, followed by a slower release later due to the protection of the wall/shell. Agudelo et al. [118] observed a decreased concentration (42%) of heat-labile natural antioxidants such as phenolic and ascorbic acid encapsulates produced by using gum arabic and bamboo fiber as encapsulating materials and noted higher retention levels (92–94%) for the ascorbic acid.

4.2. Coacervation Technique

Coacervation refers to the production of complexes between oppositely charged biopolymers upon mixing core materials with charged biopolymer solutions. In this, an oppositely charged biopolymer is added, which forms a complex entrapping the core material and precipitates. These are collected and dried under suitable conditions, and complexes with a sphere or irregular shapes are obtained [119]. Coacervation is the process of separation of colloidal solutions into two phases, viz., a colloid-rich and colloid-poor phase. Under this process, the precipitation of wall materials is performed around the active core by altering pH or temperature or the addition of electrolyte or non-solvent

compounds [25]. Depending upon the phase separation methodology, the aqueous coacervation can be classified into either simple or complex. Under simple coacervation, the phase separation is facilitated by promoting interaction between macromolecules at the cost of macromolecule–solvent interactions with the help of electrolytes such as sodium sulfate or desolvate and by the addition of solvents such as ethanol or manipulating temperature [120]. The complex coacervation is facilitated by attraction forces of oppositely charged hydrocolloids such as pea proteins, gelatin, alginates, gum Arabic, pectins, etc., and forming a matrix wall around the active ingredient previously suspended or emulsified in the wall materials. This is mostly applied for encapsulating antimicrobial compounds [121]. Castro-Rosas et al. [120] noted the very high cost, complex process, and use of glutaraldehyde as a cross-linking material as the main challenges during the commercialization of coacervation technology in the industry. In contrast, this technology has the advantage of being a simple process, having high loading capacity, no need for sophisticated equipment, low stirring needed, working at low temperature leading to very less evaporative losses and thermal degradation, as well as an adaptability to a range of raw materials, etc. [96].

Coacervation is a phase separation encapsulation process that is most commonly used after spray drying [122]. This is the first reported process for the industrial production of microcapsules. The coacervation technique is widely employed for the production of gelatin and gelatin–acacia microcapsules. It provides high encapsulation efficiency with a triggered controlled release, which broadens its application in the food systems. In the simple coacervation technique, a single polymer is used; while in complex coacervation, two oppositely charged polymeric materials such as acacia and gelatin are used. Pectin, alginate, and milk proteins are commonly used wall materials for simple coacervation. The three basic steps for complex coacervation involve the formation of three immiscible phases, deposition, and rigidization of the coatings.

Wang et al. [123] developed complex coacervates of gelatin and sodium hexametaphosphate to microencapsulate tuna oil fortified with vitamin A, D_3, E, K_2, curcumin, and coenzymes Q_{10}. They reported this process as an effective method for the encapsulation of lipid-soluble bioactive compounds. Naik et al. [124] microencapsulated α-linolenic-acid-rich oil from *Lepidium sativum* seed using the coacervation technique. Gum ghatti, gum arabic, and soy protein isolate were used to prepare an oil-in-water emulsion, and the coacervates were then lyophilized to produce the microcapsules. The authors suggested that optimized α-linolenic-acid-rich oil capsules can be used for the development of novel food products. Ostertag et al. [125] used the emulsion phase inversion technique for encapsulation of orange and limonene flavor oils using low-energy nanoemulsions. This type of emulsion has the potential for the delivery of lipophilic bioactive compounds as nutraceutical agents. An oily dispersion of lycopene was encapsulated by a complex coacervation technique using gelatin and pectin [126]. The degradation of encapsulated lycopene was linear, with an average loss of 14% per week.

Huq et al. [127] developed nisin-microencapsulated edible beads to arrest the growth of *Listeria monocytogenes* and reported a 20 times higher availability of nisin in encapsulated form during the refrigeration storage of ready-to-eat (RTE) ham for 28 days without showing any adverse impact on pH and color. The zeta potential (ζ-potential) is a very important parameter to be considered during coacervation as it plays a critical role in balancing electrostatic force between oppositely charged biopolymers. The complete neutralization of oppositely charged biopolymers results in a more stable system and superior microstructure, resulting in the higher retention of bioactive ingredients [119].

However, it is important to highlight that this technique is still at the experimental stage and has not been widely applied in the food industry. Nevertheless, it is still a technique with great potential due to its great release control ability [25].

4.3. Ionic Gelation

It is a process of the formation of complexes while mixing a charged biopolymer with anionic salt. The process of gelation can be external or internal [7] and both do not have a major influence on encapsulation efficiency. The external gelation forms when core material and charged biopolymer drops are felled into an anionic salt solution with the help of extrusion, a syringe needle, or electrospinning [7,98,128]. The process can also be accomplished by first incorporating an ionic solution into the core materials and a charged biopolymer dispersed in a water-in-oil emulsion [7]. Under the outer gelation, the outer layer of the encapsulate comprising droplets of core materials is hardened by an anionic agent, which provides protection to the inner core materials by forming cross-links. In the case of internal gelation, the core materials, encapsulants, and anionic salt solutions are mixed together and continuously mixed with an emulsion to obtain the desired size of the encapsulates [7].

The stability and structure of microstructures in ionic gelation are determined by the appropriate concentration of anionic salt and biopolymer, core load, extent of cross-linking, and drying conditions. The improper conditions during encapsulation may compromise the stability and structure of microstructures, such as being cracked, porous, rough, shrunken, etc., due to poor cross-linkages, resulting in imperfect microstructures with poor encapsulation efficiency [128–130]. This technology works on the cross-links of polyelectrolytes on the addition of multivalent cations formed during extrusion, gelation, or emulsification [88]. Paques et al. [131] reported this method as the most commonly used technology to produce gel particles by taking aqueous polymer solution and dripping it through a syringe nozzle or into a gelling bath with calcium chloride. The alginates, konjac flour, chitosan, carboxymethyl cellulose, and pectin are some commonly used polymers used for the formation of cross-linked gelling systems, with alginate as the most commonly used, especially in the controlled release of active ingredients and enzyme immobilization, amongst all these due to its edible, biodegradable, and gelling attributes [96,132]. The betalain-loaded microcapsules produced by using external gelation technology utilizing sodium alginate and bovine serum albumin as shell materials has been noticed to exert antioxidant effects after 25 days of storage [40]. Internal gelation or emulsification has a higher entrapment of active ingredients as compared to external gelation or extrusion.

The main advantages of this technique are the low polydispersity and the high encapsulation efficiency [25].

4.4. Supercritical Fluid Based Techniques

It is an energy-efficient technology in which nontoxic supercritical fluid such as supercritical carbon dioxide gas ($ScCO_2$) is utilized for the encapsulation of bioactive compounds such as in the pharmaceutical industry [25,133]. Under this method, the solvent is economically utilized, but the need for high pressure and high-end equipment still hampers the popularization of this technology. Supercritical fluids are highly compressed gases that show characteristics of liquids (such as high density, improved solvating attributes) as well as of gases (such as low viscosity, higher mass transfer, and diffusion rate) above a particularly critical point [134]. There are several compounds that exhibit a supercritical state at a critical temperature (Tc) and critical pressure (Pc), such as carbon dioxide, nitrogen, water, propane, etc. However, carbon dioxide (Tc-31.1 °C, Pc-7.38 MPa) is the most widely used among all the available supercritical fluids. Supercritical fluids are used as a solvent for the rapid expansion of these solutions (RESS), as an anti-solvent (SAS) for precipitations or related processes, and as a solute for particles from gas saturated solutions (PGSS) and associated processes [135].

The supercritical fluid can act as a solvent, and the particle formation can be performed either by RESS or by supercritical solvent impregnation (SSI). On the other hand, supercritical fluid can act as a PGSS designed for making particles. Supercritical

fluid can also act as an anti-solvent, and the particle formation can be performed either by SAS or by the supercritical fluid extraction of emulsions (SFEE). As aforementioned, carbon dioxide (CO_2) is the most commonly used gas in supercritical encapsulation not only for its availability, since it is non-toxic, low priced, and easily separated from the final product by depressurization, but also because it has low critical temperature, thus being the most preferred material for the microencapsulation of natural bioactive compounds using the supercritical fluid technique. This technology is mostly used along with co-precipitation, comprises entrapping compounds, and is chemically binding followed by absorbing in polymer matrix or encapsulation [136]. Under this method of encapsulation, there is minimal degradation of the active ingredients, thus finding several applications in the industry such as micronization, extraction, drug impregnation, and the production of membranes and scaffolds [137–139]. Under this technology, encapsulation parameters such as particle size, shape, encapsulation efficiency, bioavailability, etc., could be controlled by applying proper process parameters such as temperature, flow rate and pressure, use of proper solution, etc. This method has certain inherent advantages over conventional methods such as the proper control of particle size and shape, high yield and bioavailability of heat-labile active ingredients, higher encapsulation efficiency, easier removal of solvents, and obtaining solvent-free droplets, but the selection of proper supercritical technology compatible with the active material and polymer matrix still remain limiting factors [139,140].

In general, some sensitive compounds such as essential oils and enzymes are microencapsulated by this technique. Aliphatic polyesters, dextran, inulin, cyclodextrins, polycaprolactones, polylactide aliphatic copolymers, and starch polymer blends are frequently used as a wall material in this technique. Almeida et al. [141] studied the encapsulation of oregano essential oil in a starch matrix. It was found that the higher diffusivity of the supercritical carbon dioxide in the starch matrix ensured the deep impregnation of essential oil, resulting in the higher antioxidant activity of oregano essential oil. The bioavailability of quercetin was enhanced through microencapsulation in a surfactant material using the supercritical fluid extraction of emulsions. Soy-bean lecithin and Pluronic L64® were used as surfactant and encapsulating materials [142]. Lutein-loaded liposomes with encapsulation efficiency of 56.7 ± 0.7%–97.0 ± 0.8% and a zeta potential of −54.5 ± 1.2 mV to −61.7 ± 0.6 mV were prepared using supercritical carbon dioxide [23]. Similarly, the encapsulation of lycopene with lecithin and α-tocopherol was accomplished using supercritical anti-solvent process [24]. During 28 days of refrigerated (4 °C), storage the degradation of lycopene was less than 10%.

4.5. Micelle and Liposome Based Techniques

Liposome refers to lipid-based small membrane vesicles formed by phospholipid hydration and is gaining popularity in the development of functional food, medical uses, nutraceuticals, and cosmetics for the delivery and sustainable release of bioactive compounds [143–145]. Phospholipids form a major part of liposomes, and the most commonly used phospholipids are natural, such as soy lecithin, egg lecithin, and marine lecithin, or semisynthetic or synthetic phospholipids such as DPPC (dipalmitoylphosphatidylcholine), etc., with cholesterol commonly added in the formulations for providing structural stability [146,147]. During the preparation of liposomes, mechanical treatments such as heating and stirring facilitate phospholipid bilayers to encircle the aqueous medium-forming vesicle in which hydrophilic groups are arranged towards aqueous phase and hydrophobic ends are arranged toward the inner phospholipid bilayer, leading to the entrapping of both hydrophobic and hydrophilic substances [148–150]. Some authors [148,151] noted the critical role of phase transition temperature (Tt) in the formation of liposomes as the temperature below Tt, and phospholipids present in gel state instead of crystalline phase, resulting in a problem in formation of liposomes due to very close arrangement of hydrophobic tails in such a state [145,152]. Liposome varies in size (from 0.025 μm to 5.0 μm) and layers, viz., a single

bilayer membrane (unilamellar vesicle; LUV) and more than one layer (multilamellar vesicle; MLU, diameter ranging from 200 nm to 10 μm) [3]. LUVs are further categorized into two types based on diameter, viz., small LUVs with a diameter of <200 nm and large LUVs with a diameter >200 nm [153,154]. Ajeeshkumar et al. [145] describe four important stages during the preparation of liposomes, viz., lipid drying in a suitable organic solvent, adding the lipid to aqueous media, purification, and analysis.

A liposome delivery system is adopted for encapsulating bioactive compounds that have poor water solubility and/or are chemically unstable. It provides higher encapsulation efficiency and bioavailability of active ingredients. This technology has limitations to scaling up due to the high cost involved, the poor chemical and physical attributes, the uneven particle size, being more prone to lipid oxidation, and the requirement of post-processing, etc. [96,154].

This technique has been used by many researchers to microencapsulate a variety of compounds without any deterioration in their bioactivity. Rasti et al. [155] reported an increase in the oxidative stability of microencapsulated polyunsaturated fatty acids in liposomes. Khanniri et al. [144] incorporated microencapsulated nisin in liposomes system in dairy products and observed significant improvement in stability by preventing enzymatic degradation, targeted availability, and the release of nisin. Imran et al. [147] encapsulated nisin in liposomes formed by using soy lecithin and observed equal concentrations of free and encapsulated nisin more effective in inhibiting the growth of *Listeria monocytogenes* as compared to using only encapsulated nisin. Silva et al. [156] used novel proliposomes to encapsulate curcumin for increasing its water solubility (80% curcumin even after 2 months storage) and chemical stability by utilizing a combination of phospholipids and micro-ionized sucrose. These have good potential to be used in the preparation of functional food products. These proliposomes were changed to liposomes by applying xanthum gum, guar gum, and hydration, and 72% curcumin was observed after 1 month of storage. The ethanolic coconut husk extract (ECHE) contains a large amount of several bioactive compounds, but the dark brown color limits their use. ECHE was encapsulated by using liposomes with soy phosphatidylcholine and cholesterol to improve the antimicrobial activity as well as minimize the adverse effects on product color [157].

Micelle and reverse micelle are thermodynamically stable colloids produced by single layer of one or more surfactants such as phosphatidylcholine, lecithin, and monoglycerides and are able to encapsulate only one of either hydrophilic or hydrophobic core materials, the latter owing to the high encapsulation efficiency [153,158]. Yang et al. [159] observed 96% encapsulating efficiency for curcumin (14% load) encapsulated in micelle developed by using monomethylpoly (ethylene glycol)-poly (ε-caprolactone)-poly (trimethylene carbonate) and the active ingredient, curcumin, released slowly (20% over 48 h and 36% over 168 h) into phosphate buffer solution (pH 7.4) at 37 °C as compared to the 92–96% release of curcumin in non-encapsulated form in the same time period [159]. Reverse micelle exhibited lower encapsulation efficiency for hydrophilic core materials such as tetracycline encapsulated in PLGA (poly-d, L-lactide-co-glycolide) in methylene chloride added to cetyltrimethylammonium bromide. This lower encapsulation efficiency in reverse micelle is attributed to small hollow cavities, the presence of several pores on the surface, as well as the lower molecular weight of the core material [160]. The hydrophilic core material with a molecular weight of more than 500 Da exhibited higher encapsulation efficiency (98%) during the reverse micelle method of encapsulation [161]. For preventing leakage of a low molecular weight core material in the case of the reverse micelle, the core material is incorporated in cyclodextrin, which, due to its higher molecular weight, reduces the chances of the slippage of the core. Lu et al. [162] summarized the approaches to be used for improving micelle stability, which would improve microstructure integrity, prevent the core material from slipping out, and improve encapsulation efficiency.

4.6. Extrusion-Based Techniques

In this technique, the droplets of an aqueous solution of polymer are dropped into a gelling bath solution. Sodium alginate (0.6–3%) is the most commonly used polymer with calcium chloride gelling bath solution. The dropping tool can be a pipette, syringe, vibrating nozzle, spraying nozzle, jet cutter, or vibrating disc. This method is widely used for the encapsulation of volatiles and unstable flavors. In general, conventional extrusion is applied for particle fabrication by forcefully flowing a mixture of core and encapsulants through the nozzle, whereas co-extrusion is applied for highly concentrated and viscous cores or encapsulant incorporation and particle fabrication, leading to the formation of matrix microstructures [163,164].

Under co-extrusion, the core and encapsulants flow from the same outlet at high frequency, resulting in small encapsulates possessing a mononuclear microstructure, while the flow rate of the encapsulants and core materials and the vibration frequency regulate shell thickness [7,128,165]. Over a range of pH levels, alginate exhibits a negative charge and is able to form hydrogel via cross-links with cations. Ca^{+2} is the most widely used cation due to the formation of a desirable final product, and adequate cross-linking formation occurs with higher concentrations of alginate and cations, i.e., calcium chloride solution and improved encapsulation efficiency [89]. The extruded encapsulates can be dried by freeze drying or fluidized bed drying for extending storage stability, while freeze drying leads to a porous structure and lowering encapsulation efficiency and poor core defense. Furthermore, alginate beads are more prone to the rapid release of active ingredients, such as during digestion or in an aqueous medium, due to their lower concentration (2–4% alginate hydrogel of high viscosity), leading to a low-density gel network. To overcome this problem of fast release, positively charged biopolymers such as chitosan are used along with alginate [28,166]. Furthermore, using dietary fiber sources such as microfibers isolated from psyllium and rice along with alginate prior to cross-linking with calcium chloride was noted to significantly improve the encapsulating efficiency and optimum release of active ingredients such as caffeine [166]. Sarıyer et al. [167] utilized combinations of calcium chloride and potassium chloride salts, κ-carrageenan, and alginate for increasing encapsulating efficiency and controlled release due to the protonation and chelating effect of these beads. The more complex cross-linking network is ensured due to the cross-linkage between calcium, alginate, and sulfate groups of κ-carrageenan, whereas potassium ion further increases the cross-linkage of sulfate with the anhydrous-O-3, 6-ring of galactose residue present in κ-carrageenan. These beads were reported to have controlled releases even during digestion, as the lower pH of gastric juice further increases the active site for further increasing cross-links due to the protonation of carboxylic and alginate-κ-carrageenan. However, the higher pH in the intestine resulted in replacing calcium cations with sodium cations, leading to swelling and higher release of the core material.

4.7. Emulsion Based Techniques

An emulsion consists of at least two immiscible liquids, with one of the liquids being dispersed (dispersed phase) as small droplets in other (continuous phase) [3]. This is a suitable method for the microencapsulation of lipophilic bioactive compounds. The emulsion can be categorized into two types, viz., a single emulsion, usually being an oil-in-water emulsion, used for encapsulating oil soluble core materials and double emulsion, utilized to coat several cores in a single droplet of emulsion [153,168]. Double emulsions refer to the dispersion of one liquid into other, resulting in double-layered liquid droplets such as water-in-oil-in-water (W/O/W) or oil-in-water-in-oil (O/W/O) emulsions [169]. It has the advantages of compatibility with the food matrix, economic production, higher loading capacity, and the retention and enhancement of bioavailability of bioactive compounds.

Different types of delivery systems that can be created based on emulsion technique are conventional emulsions (oil-in-water), multiple emulsions (water-in-oil-in-water), multilayer emulsions (multilayer oil-in-water), solid-lipid particles, and filled hydrogel particles. After the preparation of the emulsion, spray drying may or may not be carried out. Thus, the emulsion preparation is actually the first step for many encapsulation procedures. The encapsulation of various bioactive compounds such as phenolics, fatty acids, anthocyanins, and vitamins has been successfully carried out using the emulsion technique. The potential of emulsion electrospraying was evaluated for the encapsulation of epigallocatechin gallate (EGCG). Hydrophilic (H-EGCG) or lipophilized (L-EGCG) catechins were encapsulated either in the aqueous or the oily phase of the emulsions [170]. Similarly, the encapsulation of isoeugenol in spray-dried emulsions with β-lactoglobulin and n-OSA starch as emulsifiers was performed [171].

A number of lipophilic vitamins and bioactive compounds such as omega-3 fatty acids, carotenoids, phytosterols, flavonoids, and vitamins A, D, K, and E are very prone to degradation upon exposure to heat, oxygen, and light. Furthermore, these bioactive compounds are not easily water soluble and possess poor stability in the human digestive system leading to the limited absorption and bioavailability of these compounds. For increasing their bioavailability and stability, it is preferred to encapsulate these bioactive compounds and incorporate them into aqueous-based food. Talón et al. [103] noted a higher encapsulation efficiency (95%) in encapsulating these compounds by forming an emulsion with a combination of maltodextrin and whey protein as an encapsulant at pH 6.6, having a ζ-potential of −29 mV, which is within the prescribed limits of ζ-potential for stable emulsion, which is ± 30 mV [172–175]. Talón et al. [103] also noted a higher encapsulation efficiency in the case of highly stable emulsion, however, excessive ζ-potential or values close to zero could further breakdown emulsion droplets, leading to an excessive repulsion force or a lack of repulsive force, resulting in flocculation [172]. The use of materials with high gelation efficiency for emulsion formation facilitates a strong structure and thus a better protection of the core material and encapsulation efficiency [176–178]. Alternatively, a pickering emulsion prepared by incorporating solid particles such as cellulose, nanocrystals, etc., to stabilize emulsion in place of surfactants also improves the stability of emulsions, as indicated in terms of biodegradability and chemical stability. This methodology is especially recommended to encapsulate active ingredients exhibiting lower melting points, such as hydrogenated vegetable oil [179].

4.8. Fluidized Bed Coating

This technology is a modified version of the spray drying method and involves the use of coating onto a fluidized bed of solid particles [180]. It is mostly used in the pharmaceutical industry. In this technology, solid and liquid core materials are encapsulated by absorbing into porous solid material. It consists of three processes such as suspending the solid materials in the air, followed by the spraying of liquid material to encapsulate these solid particles, and at last hardening the shell by cooling or solvent vaporization. The core material is attached to encapsulants by hydrogen bonds or liquid bridges and electrostatic, hydrophobic, and hydrophilic interactions [6], and surface tension and viscosity are very important parameters of encapsulant solution. These three processes are repeated until the desired thickness of the wall is achieved. By using this technique, the microencapsulation of ascorbic acid has been performed by using polymethyl acrylate and ethyl cellulose. Knezevict et al. [181] fluidized and microencapsulated ascorbic acid by using hydrophobic coating materials as top-spray and polymethacrylate coating material.

Benelli et al. [6] used a fluidized bed spray coating for the encapsulation of *Rosmarinus officinalis* extract containing various bioactive polyphenols such as carnosol, rosmarinic, carnosic, and caffeic acids by using whey protein concentrate and gum arabic as encapsulant solutions. Th authors reported encapsulation efficiencies of more than 75% upon drying air temperature of 70 °C for carnosol, rosmarinic acid, and carnosic acid, and

for caffeic acid, it was possible by using less viscous whey protein concentrate solution. However, a highly viscous solution such as gum Arabic resulted in lower encapsulation efficiency (57%) due to the poor entrapping of caffeic acid. Sun et al. [182] reported a 95% encapsulation efficiency when encapsulating L-methanol using a gelatin emulsion as an encapsulant solution and noted a lower encapsulation efficiency upon using a gelatin solution with higher viscosity. Various authors recommended an optimum surface tension of encapsulants solution as 32–49 mN/m as it facilitates a perfect microstructure, uniform wall thickness, and higher encapsulation efficiency [5,182].

Air is the most commonly used fluidizing and drying medium, and air temperature has a direct impact on the quality and characteristics of microstructures; for example, high temperature causes pores, cracks, and uneven wall thickness and coating due to the drying of droplets prior to their contact with the surface of the core material [5,183]. The desirable quality of microstructures could be achieved by increasing the coating cycles, theproper flow of coating solution and its temperature, air velocity, atomizing pressure, the core to encapsulants ratio, and the use of coating material with desirable attributes such as lower viscosity, higher solubility, lower permeability, etc. [98,183,184]. Semyonov et al. [185] encapsulated *Lactobacillus paracasei* by using molten wax with ethylcellulose solution (7% *w*/*w*) and noted a higher survival rate in the case of wax-coated particles at an inlet temperature 45 °C and an outlet temperature of 25 °C. Authors further reported a lower survival rate upon coating with ethylcellulose due to crack formation in the wall. The main limitation of this method remains a complex and lengthy process, uncontrolled aggregation of particles due to wet-coated material and the presence of liquid particles among particles [180,186].

4.9. Other Techniques

All the above-mentioned processes are mostly used for the encapsulation of bioactive compounds; however, there are some other methods that are not so common for food applications. For example, ultrasonics were merely considered as an extraction method for bioactive compounds, but now, they are gaining popularity in the development of microcapsules. Another example is the fluidized bed technique in which a liquid coating material is sprayed onto the particles and rapid evaporation forms a coating around them. Different types of fluid bed coatings including top spray, bottom spray, and tangential spray are used in this technique. The modified fluidized bed technique has been used for the microencapsulation of garlic powder having high allicin content [187]. Microscopic studies revealed the good integrity of the core in these microcapsules.

The electrospinning and electro spraying process comprises the application of the direct or alternative current electric field of high voltage (1–30 kV) through a cell solution or dispersion. This is atomized by using a blunt-ended stainless-steel needle or capillary or spinning towards the collector, resulting in the production of dry particles of fibers [130,188]. These methods are cost-effective, easily adaptable, preserve microbial cells, and are simple processes [189].

5. Microencapsulated Bioactive Compounds: Application in Food Systems

The overall success of microencapsulation in terms of bioactivity or functional effects of the bioactive compounds can only be accessed by using them in real food systems. Sometimes, it is seen that the better encapsulation efficiency does not always result in better bioactivity due to undesirable release kinetics of active ingredients. The characteristics of microcapsules such as higher or optimum encapsulation efficiency, stability, morphology, and molecular interactions between core and wall materials are important, but the actual behavior of these microcapsules in the real food systems must be ensured to draw a conclusion. Thus, after optimizing the microencapsulation process, it is mandatory to ensure that the bioactive compound has its characteristics in real food systems.

Antioxidant potential, antimicrobial effect, flavor enhancement, and food stabilization are some important functions of many bioactive compounds which are used for the development of food products (Figure 3). The antioxidant activity of bioactive compounds either in free form or in encapsulated form can be judged by the free radical scavenging activity and DPPH (2,2-diphenyl-1-picrylhydrazyl), ABTS (3-ethylbenzothiazoline-6-sulphonic acid assay), TEAC (Trolox equivalent antioxidant activity), and FRAP (ferric reducing ability of plasma) assays [190–192]. Total phenolic content also indicates the antioxidant potential of extracts containing natural bioactive compounds. Microbial inhibition tests are conducted to analyze the antimicrobial effect and a critical sensory evaluation is followed for assessing the sensory profile of food products that are being developed using these microcapsules containing bioactive compounds. The release kinetics of natural bioactive compounds, as well as their degradation rate, are also analyzed to see the positive or negative effects of microencapsulation on the active ingredients of these compounds.

There are several food industries with potential products for the inclusion of microencapsulated ingredients, including dairy products, meat products, bakery products. and beverages (Table 2). Pan et al. [193] studied the encapsulation of thymol essential oil in sodium caseinate and reported that encapsulated essential oil was more effective than free form (unencapsulated) for inhibiting food-borne pathogens (*Listeria*) in milk. It might be due to enhanced distribution and solubility through encapsulation. Chen et al. [194] encapsulated eugenol and thymol in zein/casein complex nanoparticles; the authors reported that encapsulated essential oils showed higher bactericidal activity than unencapsulated essential oils. Controlled release of eugenol and thymol was also seen in the encapsulated samples. Kfoury et al. [195,196] investigated the encapsulation of essential oils in cyclodextrins to provide long-term effects by increasing their retention, as well as controlling their release in the food matrix. They found higher ABTS scavenging capacity, a reduction in the volatility, and a lesser degradation of essential oil microcapsules during the storage period [196]. Giroux et al. [72] encapsulated vitamin B_{12} in water-in-oil-in-water double emulsions to produce functional cream for cheese milk standardization. They observed higher encapsulation efficiency (96%) and a lower loss of vitamins during in vitro gastric digestion. The encapsulation of vitamin B_{12} in double emulsions also reduced its losses in the whey portion and increased the retention from 6.3 to more than 90% in the cheese samples. The encapsulation of vitamin E using octenyl succinic anhydride modified starches has been carried out by Hategekimana et al. [66]. It was concluded that the capsules could be used in drug and beverage applications. Xu et al. [197] used dextrin for the encapsulation of polyunsaturated fatty acids to control their release and to improve their stability. Rasti et al. [155] used liposome-based delivery systems for the microencapsulation of omega-3 fatty acids, and they reported that encapsulated bioactive compounds showed higher oxidative stability.

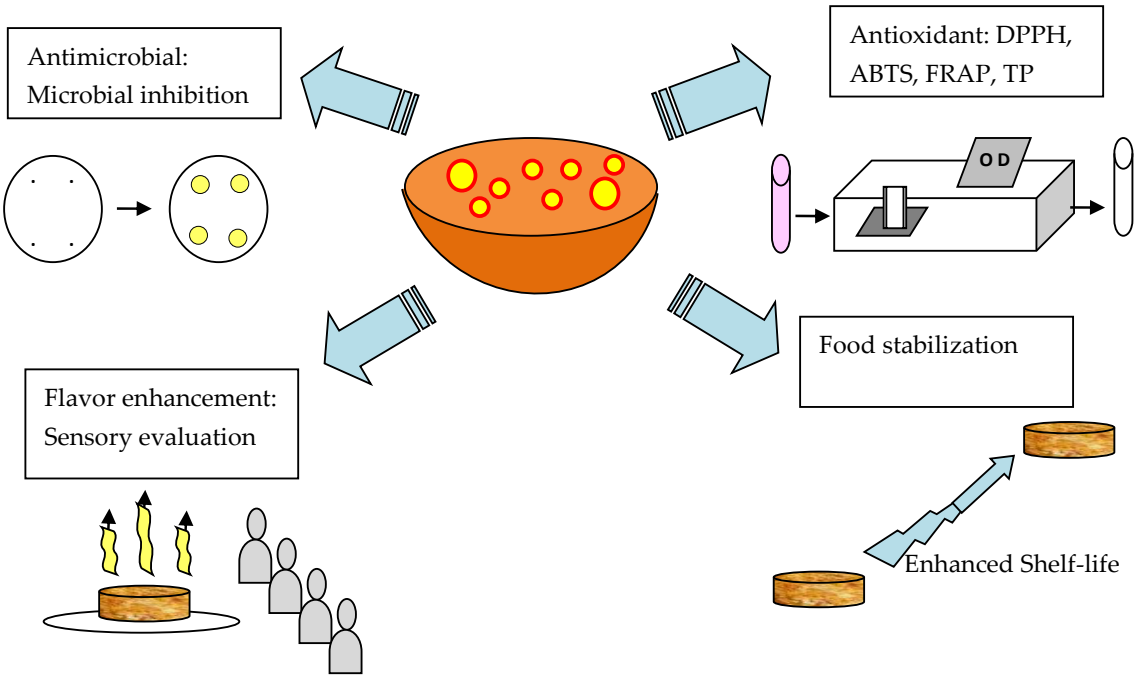

Figure 3. Assessment of microencapsulated bioactive compounds for their efficiency.

Çam et al. [198] microencapsulated the phenolics of pomegranate peel (*Punicagranatum*) by spray drying for use in ice cream formulations. They reported that enrichment with microencapsulated polyphenols from the peel improved antioxidant and α-glucosidase inhibitory activities; more than 75% of sensory evaluation panelists accepted the final product. A formulation of omega-3 fatty-acid-enriched soup powder was optimized by Rubilar et al. [199] using microcapsules of linseed oil. The linseed oil was encapsulated in a matrix of maltodextrin and gum arabic, and thus a controlled release of core bioactive compound was also achieved.

Garcinia fruit extract, a rich source of hydroxycitric acid that possesses various health benefits, has been microencapsulated and incorporated in bread [200] and pasta [201]. These authors reported that the products having microencapsulated bioactive compounds had higher quality attributes including sensory properties. Casein and inulin were used for the microencapsulation of citric acid powder [202], and the microcapsules were incorporated in the formulation of chewing gums. The samples containing microcapsules had higher sensory characteristics than control samples. Pasrija et al. [179] encapsulated green tea polyphenols by two techniques, viz., freeze drying and spray drying using different wall materials. The bread samples containing these microcapsules had similar characteristics in comparison to the control samples. The color and taste of bread samples containing microcapsules were slightly better than the control (free polyphenols) samples. These authors concluded that the fortification of bread with microcapsules of green tea polyphenols can retain the quality characteristics of bread along with the functionality of polyphenols more efficiently.

The flavor is one of the most important sensory attributes of any food product. It is easily disturbed by various processing and storage conditions as described earlier. Thus, the microencapsulation of flavor compounds and their application in real food systems are important challenges for the food industry. The encapsulation of cardamom oleoresin by co-crystallization in a sucrose matrix to formulate flavored sugar cubes has been performed by Sardar et al. [203] for tabletop use in tea. Similar to natural flavor compounds, natural food colorants are also preferred by consumers. A natural food colorant was obtained by spray drying *Opuntiastricta* fruit juice as a potential source of

betacyanin pigments [204]. This colorant was used in the preparation of yogurt and soft drinks; it was noticed that this addition resulted in an attractive tonality for the consumers, which was even maintained after one month of storage under refrigerated conditions. In a more recent study, yogurts and soft drinks were also fortified with encapsulated (spray drying; maltodextrin) betaxanthin-rich extract obtained from cactus pear fruits [205]. This water-soluble natural yellow color improves the color stability of both foods' models, although this stability was dependent on the storage temperature and light. Additionally, the foods were fortified in betaxanthins, which exert a clear and important health benefit for humans [205]. In yogurts reformulated with β-carotene microparticles, the authors observed that the incorporation of these microparticles did not drastically affect yogurt characteristics [206], while the co-encapsulation with α-tocopherol increased the oxidative stability of the product. Moreover, the sensory evaluation indicated that it is a strategy to produce nutritional fortification and allow the replacement of artificial dyes in the dairy industry. Similarly, the use of freeze-dried pomegranate encapsulated in maltodextrin presented a fantastic opportunity to develop a functional beverage with high antioxidant and antibacterial properties, while this inclusion did not affect sensory acceptance in amounts up to 2% [82]. Finally, other authors proposed the use of encapsulated (ionic gelation) betalain-rich extract of *Opuntia-ficus-indica* fruit as a colorant for the development of gummy candies [207]. In this case, the betanin stability color was confirmed, since after 30 days of refrigerated storage (4 °C), there were no significant color variations in the candies. In fact, this food product exhibited a vivid red-purple color; thus, the use of these microcapsules represents a promising application for the food industry [207].

On the other hand, hydroalcoholic extracts of two mushroom species, *Suillus luteus* and *Coprinopsis atramentaria*, were encapsulated and incorporated in cottage cheese [208]. They found that the addition of free extracts resulted in products with higher DPPH antioxidant activity but the activity declined after the seventh day. However, the cottage cheese enriched with the microencapsulated extracts had a lower antioxidant activity at the initial stage but an improvement in the activity was observed after seventh day. This might be due to the controlled release of antioxidant compounds of natural extracts in the food systems.

Another important application of encapsulated bioactive molecules is their use for extending meat products' shelf-lives. In a very recent study, the authors proposed the encapsulation of garlic essential oil using the spray drying technique and maltodextrin and gum Arabic as wall materials to prevent minced meat deterioration [209]. In this case, the use of microencapsulated essential oil at a concentration of 20% exerted a strong antimicrobial activity, which increased the minced-meat's shelf-life. Similarly, the use of casein–maltodextrin thyme essential oil encapsulated using spray drying showed a strong antioxidant and antimicrobial activity [210]. Additionally, these authors conclude that the essential oil encapsulation by spray drying with casein and maltodextrin as wall materials was effective in producing capsules with high encapsulation efficiency, excellent thermal stability, and regular morphology [210].

However, not only the use of essential oils was proposed to preserve meat products. In another study, the authors use encapsulated prickly pear extracts to extend the shelf-life of beef burgers [211]. In this research, the authors observed a significant antimicrobial effect in burgers with the addition of prickly pear extracts, and among them, the use of encapsulated extract presented higher activity than unencapsulated extract. Additionally, the encapsulation of the extract determined more desirable color and texture features [211]. The use of pitaya peel extract, encapsulated using spray drying technique and maltodextrin as wall material, also presented promising results. The use of these microcapsules delayed the protein oxidation and also demonstrated a protective effect against the alterations promoted by the treatment with high-pressure process [212].

Table 2. Microencapsulation of natural bioactive compounds for their incorporation in food matrices.

Industry	Bioactive Compound	Bioactivity/Nutrients	Encapsulation Technique	Wall Material	Food Matrix	Ref.
Dairy	Thymol	Antimicrobial activity	High shear homogenization	Sodium caseinate	Milk	[193]
	Flaxseed oil	Fortification omega-3	Spray drying	Whey protein concentrate, sodium caseinate, and/or lactose	Milk	[213]
	Pomegranate peel phenolics	Antioxidant and α-glucosidase inhibitory activities	Spray drying	Maltodextrin	Ice cream	[198]
	Flaxseed oil	Fortification omega-3	Patent (Application number: 2030/DEL/2014)		Ice cream	[214]
	Opuntia stricta fruit juice	Colorant/betacyanin retention	Spray drying	Dried glucose syrup	Yogurt and soft-drinks	[204]
	Cactus pear fruits extract	Colorant	Spray drying	Maltodextrin	Yogurt and soft-drinks	[205]
	β-Carotene and α-tocopherol	Colorant/carotene fortified/antioxidant activity	Emulsion	Soy protein isolate, palm stearin, and xanthan gum.	Yogurt	[206]
	Rapeseed oil and hibiscus extract	Colorant/Fortification omega-3	Ionic gelation	Pectin and $CaCl_2$	Yogurt	[215]
	Mushroom extracts	Antioxidant activity	Spray drying	Maltodextrin	Cheese	[216]
	Folic acid	-	Hydrogel	Alginate and pectin	Cheese	[217]
	Vitamin B_{12}	-	Emulsion	Sodium, caseinate	Cheese	[72]
	Vitamins A, E and CoQ_{10}	Fortification vitamins	Emulsion	Lecithin	Cheese	[218]
	CoQ_{10}	Fortification CoQ_{10}	Emulsion	Calcium caseinate, flaxseed oil, and lecithin	Cheese	[219]
	Chia oil	Fortification omega-3	Spray drying	Chitosan	Butter	[220]
Cereal and Bakery	Green tea polyphenols	Antioxidant activity	Freeze drying and spray drying	Maltodextrin, β-cyclodextrin, and combination	Bread	[179]
	Garcinia fruit extract	Hydroxycitric acid	Freeze drying	Whey protein isolate and maltodextrin	Bread	[200]
	Flaxseed oil and garlic oil	Fortification omega-3	Ionotropic gelation	Sodium alginate and whey protein concentrate and soy lecithin for emulsion stability	Bread	[221]
	Flaxseed oil	Fortification omega-3	Freeze drying	β-Glucan and *Saccharomyces cerevisiae* Yeast Cells	Bread	[222]
	Chia oil	Fortification omega-3	Freeze drying	Soy protein isolate	Bread	[223]
	Lycopene	-	Complex coacervation	Gelatin and gum arabic	Cake	[224]

	Chia oil	Fortification omega-3	Freeze drying	Sodium caseinate and carnauba wax	Cookies	[225]
	Hydrocitric acid	Antioxidant activity	Spray drying	Whey protein	Pasta	[201]
	Chia oil	Fortification omega-3	Freeze drying	Soybean protein isolate	Pasta	[226]
Meat	*Allium sativum* essential oil	Antimicrobial activity	Spray drying	Maltodextrin and gum Arabic	Minced meat	[209]
	Thyme essential oil	Antioxidant and antimicrobial activities	Spray drying	Casein and maltodextrin	Hamburgers	[210]
	Prickly Pear Extract	Antioxidant and antimicrobial activities	Ionic gelation	Sodium alginate	Beef burger	[211]
	Pitaya peel extract	Colorant and antioxidant activity	Spray drying	Maltodextrin	Pork patties	[212]
	Flaxseed oil and Vitamin E	Fortification omega-3 and vitamin E	Freeze drying	Gelatine and gum arabic/genipin	Chicken sausages	[227]
	Tigernut, chia, and linseed oils	Healthy meat product (replacement of SFA by MUFA/PUFA)	Spray drying	Lactose and caseinate	Deer pâté	[228]
	Fish oil	Fortification omega-3	Freeze drying	Whey protein, tragacanth gum, and carrageenan	Chicken nuggets	[229]
	Fish oil	Fortification omega-3	Complex coacervate	Soy protein isolated and inlulin	Beef burger	[230]
	Fish oil	Fortification omega-3	Spray drying	Lecithin, maltodextrin, and chitosan	Cooked and cured sausages	[231]
	Fish oil	Fortification omega-3	Spray drying	Maltodextrin, gum arabic, and caseinate	Frankfurter-type sausages	[232]
	Fish oil	Fortification omega-3	Spray drying	Lecithin, maltodextrin, and chitosan	Chicken nuggets	[233]
Others	Linseed oil	Fortification omega-3	Spray drying	Gum arabic and maltodextrin	Soup	[199]
	Citric acid	-	Microwave	Casein and inulin	Chewing gum	[202]
	Opuntia ficus-indica betalains	Colorant	Ionic gelation	Calcium alginate	Gummy candy	[207]
	Cardamom oleoresin	Flavor	Co-crystallization	Sucrose	Tea	[203]
	Pomegranate flavedo phenolics	Antioxidant and antimicrobial activities	Freeze drying	Functional beverage	Functional beverage	[82]

On the other hand, the use of microencapsulated nutrients is a good option for the development of nutrient-enriched food products. Madziva et al. [217] used edible gums i.e., alginate, pectin, and their mixtures as a wall material for folic acid encapsulation. They reported higher encapsulation efficiencies of alginate (216 μg/100 g), pectin (196 μg/100 g), and alginate–pectin mixture (360 μg/100 g of folic acid). The stability of alginate–pectin folic acid capsules was tested in the milk system. The retention of folic acid was 360 μg/100 g, which indicates their ability to remain intact in the milk system. The addition of folic acid capsules in milk resulted in a more even distribution of capsules than the incorporation after milling the curd and injection into the pressed block of raw cheese. In

cheddar cheese, the encapsulated folic acid showed more stability (360 μg/100 g) than the free folic acid (109 μg/100 g) during the 3 month ripening period.

Similarly, the encapsulation of vitamins E, A, and coenzyme Q_{10} (CoQ_{10}) in two flaxseed oil emulsion formulations which were stabilized with calcium caseinate in the presence or absence of lecithin was studied [218]. The microcapsules were used to develop cheese with higher vitamin content. In the presence of lecithin, the recovery level of vitamin E, A, and CoQ_{10} in cheese was 92%, 90%, and 93%, respectively. Higher retention of these nutrients in the cheese, as well as higher stability of the final product, was also observed during this study. CoQ_{10} was also encapsulated in a nutraceutical formulation composed of calcium caseinate, flaxseed oil, and lecithin [234]. The emulsion samples with CoQ_{10} were found to be more stable and were used as a functional cream in the cheese-making process. The retention rate of CoQ_{10} in the cheese matrix was 93% and equivalent to the total lipid retention, while no changes in the protein retention and cheese yield were noticed. Thus, the microencapsulated CoQ_{10} provided protection to lipids against oxidation in the cheese samples.

The encapsulation of different healthy oils in order to improve the nutritional quality or enrichment foods with biologically active fatty acids (e.g., omega-3, DPA, DHA, etc.) was also a strategy used by multiple researchers [25]. Moreover, microencapsulation not only prevents the degradation of nutrients but also enhances the bioavailability of these nutrients at their site of absorption. Several dairy products were reformulated with this strategy. Vitamin D_3 was encapsulated in two flaxseed oil emulsion formulations [234] to fortify the cheese with omega-3 and other polyunsaturated fatty acids**.** Emulsions were stabilized with calcium caseinate in the presence or absence of lecithin; these were used to standardize cheese milk. It was noticed that the recovery level of vitamin D_3 in the cheese samples was 91% and 84% in the presence or absence of lecithin, respectively. The higher retention and stability of the encapsulated vitamin in the curd were also observed. The same oil (flaxseed oil) was also encapsulated and added to ice cream at various levels (3%, 4%, and 5%) [214]. In this case, the authors reported a fortification in α-linolenic acid (omega-3) and high oxidative stability; thus, the nutritional characteristic of this product was improved without comprising its shelf-life or organoleptic properties during the studied period of storage (120 days). The same research group also proposed the fortification of milk using microcapsules of flaxseed oil (spray drying) [213]. Similar to the other study, the authors reported that fortified milk was oxidative stable and sensorially acceptable, while high amounts of α-linolenic acid were found in the reformulated milk, which demonstrated their nutritional improvement. Another research uses a combination of encapsulated rapeseed oil and *Hibiscus sabdariffa* L. anthocyanins to produce a healthy and functional yogurt [215]. In this case, the use of these microparticles in the yogurt matrix presented technical feasibility, providing color and functionality to the product, while the yogurt had high appearance acceptability [215]. Finally, the use of 8% encapsulated chia oil in butter manufacture produces a significant improvement of nutritional quality (omega-3), with reasonable oxidative stability [220]. In fact, these authors conclude that the omega-3 concentration in butter up to 8% could be increased using these microcapsules without any effect on the sensory characteristics.

In the bakery industry, the fortification of bread with flaxseed oil was also conducted by Kairam et al. [221]. The authors proposed the development of a functional bread using encapsulated garlic and flaxseed oils and reported that these functional breads were stable against oxidation and simultaneously received a high sensory rating and acceptability. Thus, the authors pointed out that the use of these microcapsules served as a potential carrier for the development of functional foods to enhance human health. In addition, the use of encapsulated flaxseed oil to fortify bread was studied recently by Beikzadeh et al. [222]. In this research, the encapsulation with β-glucan increased dough rheological properties, firmness, and density, while the use of yeast cells improved oxidative stability, and in both cases, higher α-linolenic acid content was observed. In conclusion, the results suggest the possible use of oil-loaded yeasts and β-glucan microcapsules for the

fortification of food products with no effects on bread sensory properties [222]. Other authors proposed the omega-3 fortification of different bakery products using encapsulated chia oil. In this regard, the use of freeze-drying encapsulated chia oil using soy proteins as a wall material did not affect the oxidative stability, specific volume, average cell area, firmness, and chewiness of fortified bread [223] while producing a significant improvement in the nutritional quality (fatty acid profile) of this bakery product. In addition, the use of microencapsulated chia oil was used for the reformulation of pasta [226]. The authors reported a significant increase in omega-3 fatty acids, while no significant variations were observed in the cooking parameters, cooking loss, and texture of cooked pasta, although in raw pasta, some influence was described for texture and water retention parameters. Additionally, encapsulation also protects highly unsaturated oil (chia oil) against oxidative deterioration [226]. Finally, encapsulated chia oil was also proposed as a partial (15% and 30%) margarine replacer during cookies manufacture [225]. In this case, the replacement produced a nutritional improvement in the cookies (high α-Linolenic acid concentrations), which reduced both atherogenicity and thrombogenicity indices. Moreover, the highest color stability during storage (30 days) was obtained in samples with 30% substitution, while the cookies produced with the 15% margarine substitution were well-accepted sensorially [225].

In the meat industry, the use of encapsulated oils to replace animal fat (high cholesterol and saturated fatty acids) with healthy marine or vegetable oils were widely studied. In this sense, encapsulated flaxseed oil was employed to design a functional (fortified with omega-3) chicken sausage [227]. In this study, the authors reported that the addition of encapsulated oil produces a significant impact on the physical characteristics of the sausages (mainly on texture and rheological parameters), with concurrent improvement in the nutritional profile in comparison to control samples [227]. The partial (50%) replacement of animal fat by microencapsulated healthier oils (tigernut, chia, and linseed oils) to enhance the physicochemical and nutritional properties of deer pâté was studied [228]. The authors found a significant improvement in the nutritional properties (decreased total fat, cholesterol, and saturated fatty acids and increased mono- and/or polyunsaturated fatty acids, depending on the formulation). However, the reformulation with high PUFA oils (chia and linseed) produced an increase in lipid oxidation and a reduction in sensory acceptability [228]. In contrast, the use of tigernut oil microcapsules did not promote oxidation and presented the same acceptability to control pâtés, which demonstrated that the reformulation is possible using this oil [228].

In a very recent study, the authors proposed the use of different encapsulated fish oils in the formulation of chicken nuggets [229]. A strong fortification of EPA and DHA fatty acids was observed, which had benefits in human health, and the encapsulation also improved the fatty acid stability (protective action against lipid and protein oxidation). Moreover, these authors also highlighted that the addition of unencapsulated fish oil in chicken nuggets led to a decrease in sensory acceptance, while the use of encapsulated oil did not affect sensory quality, which demonstrated the high potential to use encapsulated fish oil to produce healthy meat products [229]. Similar results were obtained in cooked and dry-cured sausages, in which fish oil microcapsules (mono and multilayered) did not influence physicochemical characteristics, oxidative stability, or acceptability of usual changes that take place during the culinary heating of dry-cured processing [231]. In agreement with these findings, the inclusion of microencapsulated fish oil for the fortification of chicken nuggets [233] affected neither sensory traits nor the acceptability of the enriched products. Moreover, the encapsulation of fish oil provides protection from both lipid and protein oxidation [233]. In contrast, other authors which used encapsulated fish oil to produce long-chain omega-3 enriched burgers found opposite results [230]. In this case, although a significant improvement of nutritional quality was observed, the inclusion of microparticles into burgers impaired the oxidative stability and increased the release of lipid-derived volatiles, which negatively impacted the sensory profile and overall liking of the burgers. Thus, these authors conclude that the use of fish oil

microparticles for incorporating long-chain omega-3 in meat products is not recommended [230]. In line with these findings, the use of monolayered fish oil microcapsules in the reformulation of frankfurter-type sausages increases the nutritional quality (high EPA and DHA contents, improve nutritional indices) but produces a significant increase in the lipid oxidation (both, TBARs and lipid-derived volatiles) and changes in color parameters, which could impair the sensory properties of sausages [232].

6. Conclusions, Opportunities, and Challenges

The concept of microencapsulation has been developed by the pharmaceutical industry for improving the drug delivery system. Microencapsulation has several advantages in the controlled and targeted delivery of functional ingredients through foods to the human digestive tract. It not only controls the release of active ingredients/drugs but also minimizes the side effects and masks the bitter taste, astringency, and odor. It also protects sensitive ingredients by the formation of a protective barrier, ensures the controlled release of active ingredients as per the desired concentration, changes liquids to solid-state, increases the solubility of insoluble constituents, and improves the bioavailability of active ingredients at their particular site of action. This controlled and targeted release of active ingredients is caused by heating, pH change, enzymatic action, shearing, or solubilization. Eun et al. [109] and Arenas-Jal et al. [102] summarized the purpose of microencapsulation as the control of the transfer rate of active ingredients, the protection of sensitive ingredients from environmental conditions, the precision and control of the reactivity and incompatibility of active core ingredients, change in the physical state of core materials (such as increasing melting point increasing, dry powder form, flowability, stickness, and hygroscopicity), masking of the off-odor, bitterness, and color of active ingredients, the controlled release of core materials at the desired place at desired concentrations, and increasing the handling storage stability of core ingredients such as in powdered form.

This technology is gaining popularity among consumers due to the inherent advantages of the delivery of active ingredients, associated health benefits and increasing awareness. As per one projection by the Global Market Insights survey of 2017, the market of functional food with encapsulated active ingredients is projected to reach up to USD 45 billion dollars by 2024. The current trend in the industry is to prepare convenient and ready-to-eat or ready-to-prepare food products and increase their nutritional value and functional properties by incorporating various functional bioactive compounds such as essential oils, polyphenols, herbs, omega-3 fatty acids with the 'fat is back label', plant extracts, etc. In the food industry, the research is mainly oriented towards avoiding unwanted interactions, enhancing bioavailability, and reducing undesired flavors. With increasing education and awareness leading to an increasing interest by consumers in how foods are prepared and ingredients added, this technology will be immensely helpful in the success of the food industry, and microencapsulated food with natural active ingredients will play important role in the future.

At present and in the future, the availability of suitable wall materials that fit within the requirement of personal beliefs, food habits, and clean label requirements poses a challenge in the food industry. For example, lactose, casein, milk fat, etc., in wall materials may not be suitable for vegans, and by chance, these materials are some of the most commonly used materials in the food industry for encapsulating active ingredients [102,234]. The food industry has explored the use of plant proteins as encapsulating materials such as soy protein, pea proteins, prolamins obtained from barley, sorghum, and corn, etc. Gomez et al. [4] observed the need for in-depth research at microscopic and nanoscopic levels to identify various health and safety issues and alleviate them to make this method more acceptable and compatible with green food processing.

Thus, the success and application of microencapsulation technology in the food industry depends largely on the possibility to scale up the operation and devise simple and economic processes to be used at industrial scale. It can be certainly achieved with

the introduction of more advanced methodologies, the use of suitable and compatible wall materials, and ensuring compliance with various food safety and trade regulations.

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