AGRO PRODUCTIVIDAD

Colegio de
Postgraduados

Encapsulation of bioactive compounds of food interest: applications, current advances, challenges, and opportunities

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ABSTRACT

Objective: The encapsulation of bioactive compounds of food interest provide protection against ambiental factors and degradation reactions. Therefore, the encapsulation of these compounds, was studied and analyzed considering the applications, current advances, challenges, and opportunities on the topic.

Design/methodology/approach: Wall materials, bioactive compounds of food interest, encapsulation methods, applications, current advances, challenges, and opportunities in encapsulation of bioactive compounds were explored, described, and discussed considering the principal literature on the topic, and scientific databases were used for the bibliographic research.

Results: Encapsulation process is a novel technology that allows the increasing the stability of aromas, flavors, pigments, and microorganisms, beside of improve the sensory, physical chemical and functional properties, quality, and the extend the shelf-life.

Limitations on study/implications: Foods contain bioactive compounds that are susceptible to oxidation and degradation, which can reduce their quality and shelf life. To preserve these compounds, is important to develop other encapsulation systems considering alternative wall materials from different sources that can be applied under different process conditions from laboratory, pilot to industrial scale.

Findings/conclusions: Encapsulation process provide protection to bioactive compounds enhancing the sensory, physical chemical and functional properties, quality, and extend the shelf-life considering the integral and sustainable use of agricultural products.

Keywords: encapsulation, bioactive compounds, food interest, applications

Citation: Fabela-Morón, M. F., Pérez-Ruíz, R. V., Ruíz-Hernández, R., Arce-Vázquez, M. B., Aguilar-Toalá, J. E., Jiménez-Guzmán, J., & García-Garibay, J. M. (2022). Encapsulation of bioactive compounds of food interest: applications, current advances, challenges, and opportunities. *Agro Productividad*. https://doi.org/10.32854/agrop.v15i10.2407

Academic Editors: Jorge Cadena Iñiguez and Libia Iris Trejo Téllez

Received: May 21, 2022.
Accepted: October 24, 2022.
Published on-line: December 20, 2022.

Agro Productividad, 15(11). November. 2022. pp: 31-43.

INTRODUCTION

Food systems are susceptible to degradation making them unacceptable for its consumption and shelf-life. Bioactive compounds from foods require to be protected to extend the shelf-life, to improve its physical chemical, and sensorial properties, quality and stability in food science and technology. Exist an increasing interest in the development of food products containing natural compounds with antioxidant activity and other valuable properties. However, due to their structure and nature certain bioactive compounds such as polyphenols, flavonoids, carotenoids, among others, are not stable when are incorporated in food systems. The stability and shelf-life of bioactive compounds increase when are protected against chemical and physical factors prior to their application. In this sense, encapsulation process provides protection due to its potential for stabilization and controlled release of sensitive bioactive compounds, increasing the bio accessibility of bioactive molecules during digestion, and raising the water solubility of bioactive ingredients of food interest in different applications (Hcini *et al.*, 2021; Jayaprakash *et al.*, 2023; Komijani *et al.*, 2022).

Encapsulation technology

Encapsulation process is the technology by which a material or mixture of materials is entrapped with another material in the form of micro- and/or nanostructures through entrapment of a bioactive core with another substance called wall materials to provide protection to bioactive compounds as flavors, colorants, among others (Finney *et al.*, 2002; Jayaprakash *et al.*, 2023). This technology is widely used in the pharmaceutical, chemical, cosmetic and food industries. In the food industry, is used to encapsulates oils, fats, aromatic compounds, oleoresins, vitamins, minerals, colorants, flavors, antioxidants, and enzymes (Madene *et al.*, 2006). The material inside the microcapsule is called “core”, “inner phase” or “filler”, while the wall is referred to as “shell”, “coating”, “wall material” or “membrane”. Wall material does not react with the encapsulated compound (Gharsallaoui *et al.*, 2007).

Wall materials as encapsulant agents

Several food grade biopolymers have been used as wall materials in encapsulation of bioactive compounds, such as gum arabic, alginates, carrageenan, mesquite gum, proteins (milk or whey proteins, gelatin, soy, pea), maltodextrins with different dextrose equivalents, waxes and their blends (Mohan *et al.*, 2015; Wandrey *et al.*, 2010; Yang & McClements, 2013).

Physical chemical properties such as solubility, molecular weight, glass transition, crystallinity, diffusivity, film formation and emulsion properties, compatibility with the food product, dissolution release, among others, are very important to select the encapsulant agents (Madene *et al.*, 2006). Carbohydrates such as starches, corn syrup solids and maltodextrins are frequently employed as encapsulating agents. These materials exhibit low viscosities at high solids concentrations and good solubility (Da Costa *et al.*, 2012; Madene *et al.*, 2006), due to reduced interfacial properties of these materials are employed with mixtures of proteins or gums (Jayasundera *et al.*, 2011; Vélez-Erazo *et al.*, 2021). Starch and starch-based ingredients (modified starches, maltodextrins and cyclodextrins) act as carrier agents for flavor encapsulation, fat replacers and emulsion stabilizers (Falcão *et al.*, 2022; Madene *et al.*, 2006). Maltodextrin is a polysaccharide obtained by partial hydrolysis of corn, potato, or rice flour by means of acids or enzymes. Its chemical composition consists of D-glucose units linked with bonds (1-4) and with a low number of bonds (1-6) in random position. It has been determined that Maltodextrin with 10-20 DE are more effective for microencapsulation (Šturm *et al.*, 2019; Vidović *et al.*, 2014). Pectin is a polymer that produces stable emulsions due to its emulsifying properties related to the protein residues present with the pectin chains and to its chemical composition characterized by a high content of acetyl groups (Braccini & Pérez, 2001; Popoola-Akinola *et al.*, 2022). Gums and thickeners decrease sweetness in encapsulation process (Madene *et al.*, 2006), being used in encapsulation for their emulsion-stabilizing and film-forming properties; the most used is gum arabic (Krishnan *et al.*, 2005). Gum arabic is a branched heteropolysaccharide consisting of D-glucoronic acid, L-rhamnose, D-galactose and L-arabinose (F. C. da Silva *et al.*, 2013). This material has emulsifying properties due to the presence of arabinogalactan structure (Archaina *et al.*, 2019; Mcnamee *et al.*, 1998). Proteins as natural encapsulant matrix have functional properties (solubility, film formation, ability to interact with water, emulsion formation and stability properties), proteins play a role as potential ingredients for the development of novel encapsulant systems. In recent years, protein-based carrier agents like whey protein and soy protein isolate are reported to be more effective and to produce higher product yields even when used at low concentrations (Jayasundera *et al.*, 2011; Nesterenko *et al.*, 2013).

Chitosan is other wall material derived from chitin, that can be found in the cell wall of fungi, exoskeleton of arthropods, and crustaceans. Is environmentally friendly, non-toxic, and has excellent antimicrobial and fil-forming properties to be used in encapsulation process (Ashfaq *et al.*, 2022; Chausali *et al.*, 2022; Ré, 2006). Hydrogels and nanocarriers, also can be used as natural carrier agents for encapsulation as three-dimensional (3D) colloidal systems contained of physically or chemically bonded linear or branched polymer chains that can retain and absorb considerable amounts of water or bioactive compounds owing to hydrophilic features in the substitutional gaps between chains with amide ($-CONH-$), carboxyl ($-COOH$), hydroxyl ($-OH$), and sulfonic acid ($-SO_3H$) inside chains (Do *et al.*, 2022). Principal coating materials that can be used in hydrogels are polysaccharides, proteins, and lipids (Amiri *et al.*, 2022; Wen *et al.*, 2022). On the other hand, when polymers are used in mixtures of two or more encapsulating agents, the encapsulate structure can exhibit excellent physical characteristics and properties. The synergistic effect resulting from the mixture of carbohydrates, gums and proteins offers a significant application in bioactive compounds encapsulation, cost reduction in price and to create new macro, micro and nanostructures (Carneiro *et al.*, 2013; Du *et al.*, 2014; V. M. Silva *et al.*, 2014). For example, mixtures of whey proteins with maltodextrin, corn syrup and lactose; soy proteins with maltodextrin; sodium caseinate with lactose; whey protein concentrates with maltodextrin, increase the retention of volatile compounds and the efficiency of encapsulation of food oils by spray-drying (Rodea-González *et al.*, 2012).

Bioactive compounds of food interest

Among the bioactive compounds of food interest to encapsulate, stand out ingredients that provide flavor, color, beside of antioxidants, polyunsaturated fatty acids, polyphenols, etc. Table 1 indicates the principal bioactive compounds of food interest to be encapsulated:

Flavors. Flavors whether liquid or solid, are a complex mixture of volatile substances and labile components, susceptible to oxidation, chemical interactions, or volatilization and consequently affect sensory perception. To minimize damage, microencapsulation is used to trap liquid flavor substances: essential oils, oleoresins, aroma blends and acidulants. In this regard, research has been conducted to evaluate the wall material composition and operational conditions in relation to the retention and control of the release of encapsulated flavors (Buffo *et al.*, 2002; Goula & Adamopoulos, 2010). Enzymes. One of

Table 1. Principal bioactive compounds of food interest

Bioactive compounds	Source	Application	References
Bioactive peptides	Spirulina	Peptide based foods.	(Ovando *et al.*, 2018)
Phenolic compounds	Agroindustrial waste of mango	Food products with antioxidant activity	(Castro-Vargas *et al.*, 2019)
Antioxidants, phenolics, flavonoids carotenoids, anthocyanins, pectin	Tropical fruit, agri-food by-products	In functional foods and nutraceuticals	(Cádiz-Gurrea *et al.*, 2020; Gullón *et al.*, 2020)
Aminoacids, phenolics	Beans (*Phaseolus vulgaris*), (*Phaseolus lunatus*), (*Phaseolus coccineus*)	Development of functional foods with nutraceutical potential.	(Alcázar-Valle *et al.*, 2021)
Polyphenols	*Citrus pomace*	As nutraceuticals in functional foods and beverages	(Caballero *et al.*, 2021)
Phenolics, pectin, oils	Pomegranate fruit biowastes	Industrial application	(El-Shamy & Farag, 2021)
Vitamins, carotenoids, polyphenols	Kiwi fruit, byproducts	Development of novel valuable foods	(Sanz *et al.*, 2021)
Phenolics, oils	Olive byproducts	Foods packaging systems.	(Khwaldia *et al.*, 2022)
Carotenoids, pigments	Agrowastes from fruits, vegetables	Red, orange, and yelow colorants in food industry	(Cassani *et al.*, 2022)
Unsaturated fatty acids, amino acids, carotenoids, chlorophylls, phycocyanins, phenolic compounds	Spirulina	For use as colorants, antioxidants and functional ingredients in food products.	(Bortolini *et al.*, 2022)
Capsaicinoids, phenolics, pigments	Habanero chili pepper, Bell pepper wastes	Ingredient in food products.	(Fabela-Morón *et al.*, 2020; Razola-Díaz *et al.*, 2022)
Anthocyanins, ellagic acid, tocopherols, tannins, polyphenols	Jabuticaba (*Myrciaria* spp. or *Plinia* spp.)	As a food supplement in beverages, bakery products, and biodegradable film	(Fernandes *et al.*, 2022)
Flavonols, phenols and anthocyanins	Berries agroindustrial waste	Development of protective films.	(Romero *et al.*, 2022)
Amino acids, phenolic, flavonoids compounds	*Moringa oleifera* Lam.	Agroindustrial and food products uses.	(Ruiz-Hernandez *et al.*, 2022)
Antioxidants	Legumes	Chemopreventive agents in foods	(Sánchez-Chino *et al.*, 2022)

Source: own elaboration.

the main advantages of enzymes encapsulation by spray drying is the short times of this process. Due to the thermosensitivity of enzymes, the duration of this process is used to microencapsulate without causing severe damage to the enzyme structure. In addition, the incorporation of proteins as encapsulant agents increases porosity, surface flexibility, mechanical strength, and water evaporation (Jayaprakash *et al.*, 2023; Wen *et al.*, 2022).

Antioxidants. In food industry, antioxidant compounds have become of great interest due to their potential benefits as important nutraceuticals ingredients, because they have a protective effect on oxidative process (Fang & Bhandari, 2010).

Polyphenols. The consumption of polyphenols (anthocyanins, catechins, tannins and flavonoids) reduces cardiovascular damage and the risk of cancer. The effectiveness of these compounds depends on the stability, bioactivity, and bioavailability of the active compound. Due to their sensitivity to environmental factors such as light, heat and oxygen, encapsulation is an effective process for their preservation (Bakowska-Barczak & Kolodziejczyk, 2011; Koop *et al.*, 2022)to a large extent, due to its content of bioactive nutrients and their importance as dietary antioxidants. There is a growing demand for delivery of antioxidants through functional foods with the related challenge of protecting their bioactivity during food processing and subsequent passage through the gastrointestinal tract. This study focuses on the evaluation of concentration of bioactive compounds in black currant berries (*Ribes nigrum* L.).

Carotenoids. Composed of the hydrophobic carbon chain and are defined as natural pigments synthesized by plants, algae, bacteria, and fungi sources, which are related to the yellow, orange, and red colors (Luana Carvalho de Queiroz *et al.*, 2022).

Polyunsaturated fatty acids. Encapsulation of polyunsaturated fatty acids as omega-3 and omega-6 fatty acids, is commonly used in food fortification for their considerable health benefits to preserve their characteristics such as odor, flavor and oxidative stability (Carneiro *et al.*, 2013; Khwaldia *et al.*, 2022; Quintanilla-Carvajal *et al.*, 2010).

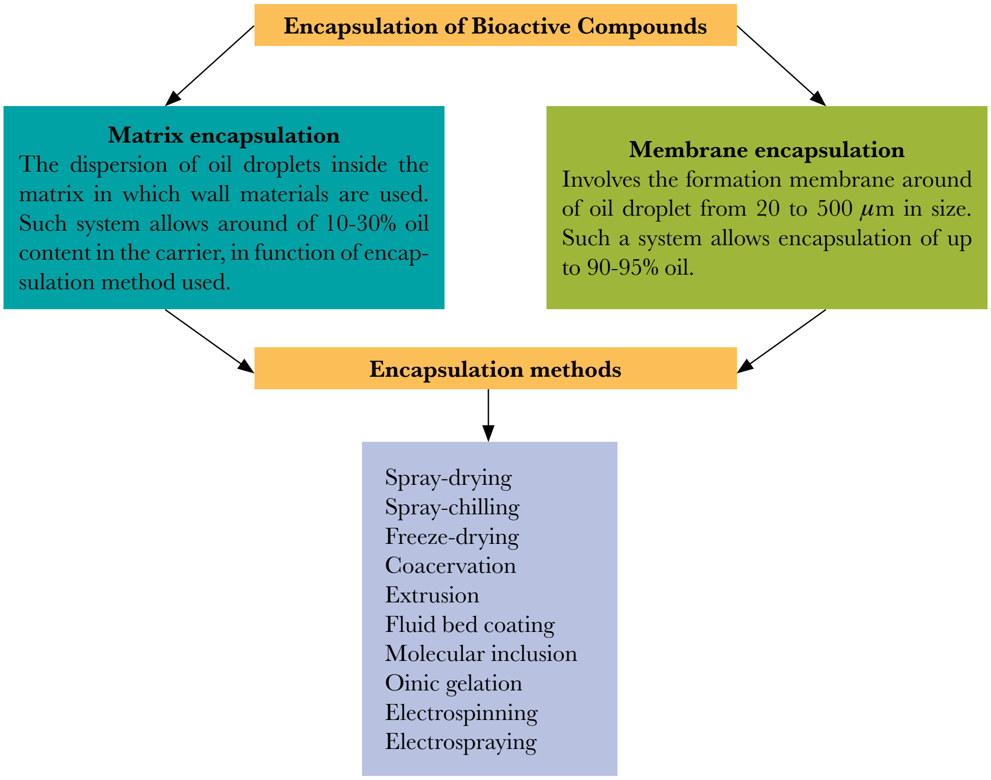
Encapsulation methods

There are different encapsulation methods that can be used to encapsulate bioactive compounds of food interest, which are indicated in Figure 1:

Spray drying. Is an encapsulation method that provides maximum protection of the encapsulated compounds in dried and stable form (Fang & Bhandari, 2010), improving flavor, aroma, stability, nutritional value and appearance, and allowing the complete release of the bioactive ingredient from wall material (Adamiec & Kalemba, 2006; Krishnan *et al.*, 2005). These process achieves microparticles or microcapsules with particular characteristics and properties (Burgain *et al.*, 2011).

Spray chilling. Is a variant of spray drying, which consists of cooling or freezing, where the material to be encapsulated is mixed with the carrier and atomized by means of nebulization of the emulsion or suspension containing the wall material and the solid or liquid active substance (sensitive pigments, flavors, and aromas, antioxidants, and natural preservatives) in cold air (Figueiredo *et al.*, 2022; Yáñez *et al.*, 2002). The coatings usually used are vegetable oils in the case of cooling spraying or hydrogenated vegetable oil for freeze spraying; thus, heat-sensitive liquids and materials that are not soluble in conventional solvents can be encapsulated.

Freeze drying. Is the widely used method for the encapsulation of thermosensitive substances, because minimizes thermal degradation reactions for carotenoids encapsulation (Šeregelj *et al.*, 2021). Coacervation. involves the separation from solution of colloid particles which then agglomerate into separate, liquid phase called coacervate, the core material



Encapsulation of Bioactive Compounds

Matrix encapsulation
The dispersion of oil droplets inside the matrix in which wall materials are used. Such system allows around of 10-30% oil content in the carrier, in function of encapsulation method used.

Membrane encapsulation
Involves the formation membrane around of oil droplet from 20 to 500 μm in size. Such a system allows encapsulation of up to 90-95% oil.

Encapsulation methods

Spray-drying
Spray-chilling
Freeze-drying
Coacervation
Extrusion
Fluid bed coating
Molecular inclusion
Oinic gelation
Electrospinning
Electrospraying

Figure 1. Classification of encapsulation methods. Source: own elaboration.

used must be compatible with the receiver polymer and be insoluble or partially soluble in the coacervation medium. Simple coacervation involves only one type of polymer with the addition of strongly hydrophilic agents to the colloidal solution. For complex coacervation, two or more kind of oppositely charged polymers are used to protect bioactive compounds (Madene *et al.*, 2006; Ré, 2006).

Extrusion. is based on the gelation of an anionic polysaccharide, when in contact with calcium or any other multivalent ion, immobilizing microorganisms or bioactive compounds. Alginate, k-carrageenan and whey proteins can be used to obtain capsules (beads) by this method.

Fluid bed coating. Consists of suspending solid particles in air at high velocity in a temperature and humidity controlled chamber, where the wall material is atomized with hot air to encapsulate bioactive ingredients using hydrogenated vegetable oils, stearins, fatty acids, emulsifiers, waxes, starches, gums and maltodextrins (Yáñez *et al.*, 2002).

Molecular inclusion. Encapsulation of compounds is performed using cyclodextrins and maltodextrin as biopolymer matrix (Yáñez *et al.*, 2002).

Ionic gelation. Process that encapsulates by means of extrusion dripping, is an efficient and low-cost method that does not require specialized equipment, high temperature, or organic solvents, making it appropriate for hydrophobic or hydrophilic compounds using calcium alginates (Arriola *et al.*, 2019).

Immobilized cell technology. In this process, the material to encapsulate as probiotics, enzymes, is trapped throughout the matrix, but not necessarily inside it by extrusion or emulsification. Involves the entrapment of living cells in spherical gel beads and was initially designed to improve continuous fermentation processes, especially in the dairy industry, as well as to enhance cell protection against undesirable conditions (Frakolaki *et al.*, 2021).

Electrospraying. Is used in food and nutraceutical applications, based on electrohydrodynamic process using high voltage electrical field without heating, offers high product quality, encapsulation efficiency, short drying time and economical as novel alternative method (Jayaprakash *et al.*, 2023). Electrostatic spray drying has recently been introduced as novel process, which uses electrostatic charge technology, and achieve low drying temperatures, higher encapsulation efficiency, and reduced bioactive compounds degradation (Jayaprakash *et al.*, 2023).

Applications and current advances in encapsulation of bioactive compounds

Table 2 shows the applications and current advances of encapsulated bioactive compounds using different wall materials. The advantages of encapsulating these compounds are related to retard auto-oxidation, increase stability, enhance flavor, mask the bitter taste of lipidic substances, and to reduce the risk of oxidation.

Challenges and opportunities in the encapsulation of bioactive compounds of food interest

Meat and seafood products contain compounds like polyunsaturated fatty acids and fat-soluble vitamins that are susceptible to oxidation, which can reduce their quality and shelf-life. Therefore, there is interest in developing antioxidant packaging materials to preserve these sea products as part of principal challenges in encapsulation technology for their preservation. Respect to fruits and vegetables, have been protected with nanostructured wall materials, alginate-based films, cellulose nanocrystals, and polylactic acid/gelatin/polylactic acid multilayer films, among others. Consequently, in food products is being of interest the development of another encapsulant systems to improve their protection, properties, and shelf-life considering the different alternatives that encapsulation methods and wall materials provide.

In addition, the development of encapsulation process suitable for different applications in the food industry has some challenges and opportunities to be investigated and improved, which need to be addressed in the future research of encapsulation of bioactive compounds.

The current development of novel encapsulation systems is mainly focused on the preparation step in the laboratory, and most of the research developed does not take into consideration the cost and production conditions process. Also, other challenges and opportunities in the encapsulation of bioactive compounds include the exploration of alternative wall materials from different sources that can be used in different foods stuffs and food matrices to improve the performance in the creation of microparticles. Therefore, is necessary to extend the research developing of manufacturing processes to encapsulate bioactive compounds with different wall materials and selecting the kind of encapsulation method from pilot-scale to industrial scale. Additionally, is important to evaluate and establish the possible effects of encapsulant agents and process conditions

Table 2. Applications of bioactive compounds encapsulated.

Bioactive compounds	Wall materials	Encapsulation method	Application	References
Aqueous extract of pink-fleshed guava fruit	Maltodextrin, gum arabic, and their mixtures.	Spray-drying	Product that can be incorporated into different food products in powder form.	(Osorio *et al.*, 2011)
Paprika oleorresin	Modified starch Capsul®	Spray-drying	Carotenoids, antioxidants protection.	(Rascón *et al.*, 2015)
Phenolic and antioxidant compounds from Roselle (*Hibiscus sabdariffa* L.)	Maltodextrin, gum arabic	Spray-drying	Instant beverage powder to extend shelf-life.	(Archaina *et al.*, 2019)
Non-dewaxed Propolis and antioxidants from honey	Maltodextrin, gum arabic, inulin	Freeze-drying Spray-drying	Water-dispersible propolis powder and phenols protection.	(Šturm *et al.*, 2019)
Antioxidants from Bottle Gourd (*Lagenaria siceraria*) juice	Maltodextrin, whey protein isolate, soy protein isolate	Spray-drying	Beverage powder to extend shelf-life and bioactive compounds protection.	(Bhat *et al.*, 2021)
Antioxidants from food sources	Gelatin, whey, soy proteins, cellulose, starch, chitosan, alginate, lipids, beeswax, palm wax, fats, and oils.	Nanotechnology process	Active food materials as nanoparticles, nanofibers, nanocrystals, and nanoemulsions with antioxidants properties.	(Cheng *et al.*, 2022)
Phenolic compounds	Tunisian Rosemary (*Rosmarinus officinalis* L.) Extracts	Coprecipitation	Nanoparticles for use in food tecnology.	(Hcini *et al.*, 2021)
Capsicum oleoresin	Gum arabic, modified corn starch (EMCAP^{TM}), modified malt (MALT)	Spray-drying	*Capsicum oleoresin* protection with capsaicin retention, and antioxidant activities.	(da Silva Anthero *et al.*, 2022)
Asthaxantin	Carrageenan, chitosan, lupin protein isolate	Spray-drying	Functional ingredient in Foods.	(Morales *et al.*, 2021)
Cinnamon esential oil	Sodium alginate, chitosan powder,	Droplet-based millifluidic technique	Food engineering.	(Farahmand, Emadzadeh, *et al.*, 2022)
Probiotics	Prebiotics matrices	Immobilized cell technology, Extrusion, Spray-drying, Freeze-drying	For food products to enhance microbial balance.	(Frakolaki *et al.*, 2021)
Lycopene	Basil gum, zein	Electrospinning	Functional nanofibers.	(Komijani *et al.*, 2022)
Probiotics	Low molecular chitosan powders	Ionic gelation	Probiotics protection and controlled release.	(Farahmand, Ghorani *et al.*, 2022)
Phenolics, probiotics	Chitosan, fructans, whey protein, alginate, gelatin, oils	Electrospraying, spray-drying	Use in food products.	(Jayaprakash *et al.*, 2023)

Source: own elaboration.

on sensory, physical chemical, and tecno-functional properties, human health, and the environmental impact in a sustainable perspective (Cassani *et al.*, 2022; Cheng *et al.*, 2022; Figueiredo *et al.*, 2022; Šeregelj *et al.*, 2021).

CONCLUSIONS

Encapsulation process is a novel technology with many advantages for its applications in a food science and technology, that allows the increasing the stability of aromas, flavors, pigments, and microorganisms, cover undesirable odors and flavors, allow controlled release, and increase the bioavailability of bioactive compounds, with health benefits for consumers. This process has avoided the development of wall materials for different uses in food science and technology. The physical and chemical properties of wall materials can improve the antioxidant and functional properties of active ingredients encapsulated, thus inhibiting the oxidative and degradation reactions in foods. Consequently, encapsulation processes of bioactive compounds can improve the sensory, physical chemical and functional properties, beside of quality, in addition to extend the shelf-life considering the integral and sustainable use of agricultural products within the applications, challenges and opportunities to improve the encapsulation of bioactive compounds of food interest from laboratory, pilot to industrial scale.

ACKNOWLEDGMENTS

Authors would like to thank the Department of Food Science from Universidad Autónoma Metropolitana, Unidad Lerma, for the support provided in the publication of this research work.

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