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Review

Food Polysaccharides as Stabilizers and Carriers for the Delivery of Polyphenols and Pigments

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Abstract

Polysaccharide-based microparticles have emerged as suitable carriers and stabilizers of active substances, showing potential to stabilize bioactive compounds during storage and gastrointestinal digestion, thereby improving their bioaccessibility and bioavailability. This narrative review provides a comprehensive overview of the main polysaccharides employed as wall materials, including starch, maltodextrin, alginate, pectin, inulin, chitosan, and gum Arabic, and discusses how structural interactions and physicochemical properties can positively influence the microencapsulation of polyphenols and pigments. The principles and main findings of the main microencapsulation techniques, including spray-drying, freeze-drying, extrusion, emulsification, and coacervation, are briefly described. Polysaccharides can entrap both hydrophilic and hydrophobic compounds through physical interactions, forming a barrier around the nucleus or binding to the bioactive compound. Intermolecular binding between polysaccharides in the wall matrix, polyphenols, and pigments in the nucleus can confer up to 90% encapsulation efficiency, primarily governed by hydrogen bonds and electrostatic interactions. The mixture of wall polysaccharides in the microparticles synthesis favors the encapsulation solubility, storage stability, bioaccessibility, and bioactivity of the microencapsulate compounds. Clinical trials regarding the bioefficacy of polyphenols and pigments loaded in polysaccharide microparticles are scarce and require further evidence to reinforce the use of this technology.

Keywords: microencapsulation; polysaccharide carriers; polyphenols; pigments; bioaccessibility; bioavailability

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1. Introduction

Microencapsulation refers to the use of different materials to entrap and stabilize solid, liquid, or gaseous substances within a nucleus, resulting in microscopic particles that protect, transport, and release active compounds in a controlled manner, according to

properties of the coating agent [1]. Microencapsulated compounds may exhibit enhanced nutraceutical and therapeutic effects due to increased stability against environmental conditions, resulting in enhanced bioaccessibility and bioavailability after intake [2,3]. Microencapsulation can be employed for enriching food and nutraceuticals by encapsulating bioactive compounds, extending their half-life, avoiding sensory alterations, and preventing anti-nutritional interactions [3,4].

Several polysaccharides from plants and agro-industrial residues are utilized in food processing and formulated products due to their low cost, biodegradability, and biocompatibility. Additionally, these polysaccharides are components of the typical diet and are generally recognized as safe (GRAS) by the United States Food and Drug Administration (FDA) [5]. Starch, maltodextrin, pectin, gum Arabic, and sodium alginate, among others, can protect and facilitate the delivery of bioactive compounds, justifying their broad application as food-based carrier systems [6].

Bioactive compounds exhibit biological or physiological roles that confer health benefits and mitigate the development of chronic diseases by modulating endothelial function, blood pressure, and oxidative homeostasis [7,8]. These benefits align with epidemiological studies, which corroborate the inverse association between regular fruit and plant intake and chronic diseases [9].

Naturally occurring antioxidants, such as the polyphenols and pigments found in plant foods, are secondary metabolites that promote human health by modulating multiple cellular processes [10]. Polyphenols comprise an extensive group of compounds, including phenolic acids, flavonoids, stilbenes, and lignans. In addition, secondary metabolites include natural dyes such as carotenoids, which impart red, yellow, and orange hues; yellow-colored flavonoids; green-colored chlorophylls; purple-colored anthocyanins; red-violet and yellow betalains [11]. The health benefits of polyphenols and pigments stem from their ability to neutralize reactive oxygen and nitrogen species, as well as induce the expression of genes encoding antioxidant and anti-inflammatory enzymes [3,8]. These compounds are marketed mainly, and their addition to foods and nutraceutical products is regulated by the FDA and the European Food Safety Authority (EFSA) [12]. To benefit from these natural compounds, it is necessary to overcome their limited physiological solubility, instability under gastrointestinal enzymatic and bacterial metabolism, and instability in the bloodstream against plasma enzymes, resulting in rapid elimination [10].

Polysaccharide microparticles have been developed to stabilize polyphenols and pigments using techniques common in the food industry, including spray-drying, freeze-drying, extrusion, coacervation, and emulsification. Optimized conditions are necessary to achieve satisfactory microencapsulation, including the appropriate technique, wall polymer, and optimizing processing conditions [1]. Furthermore, physicochemical interactions between the functional groups of polysaccharides and bioactive compounds, such as hydrogen bonds, hydrophobic and electrostatic interactions, influence the properties of microparticles and are critical factors to consider in the synthesis of polysaccharide–bioactive delivery systems [13]. This narrative review presents the main findings on the synthesis of polysaccharide microparticles, highlighting their role in physicochemical properties of bioactives, such as solubility, stability, bioaccessibility, and bioactivity. It also discusses the main techniques applied in microparticle synthesis, the potential chemical interactions underlying microencapsulation, and perspectives on biological effects.

2. Polysaccharides as Vehicles for the Entrapment of Polyphenols and Pigments

Several approaches have been developed using food polysaccharides alone or blends for the synthesis of delivery systems, demonstrating their high capacity to retain both

hydrophilic and hydrophobic bioactive compounds. Interactions between the functional groups of wall polymers and the active nucleus of microparticles confer desirable physico-chemical properties, enabling their use for enriching food formulations and nutraceuticals.

2.1. Starch

Starch is a polysaccharide composed of amylose and amylopectin polymers, which make up the amorphous and crystalline areas of the starch structure, respectively (Figure 1). Amylose is a predominantly linear sequence of D-glucopyranose units linked by α-1,4-glycosidic bonds, and amylopectin presents an essentially branched structure formed by α-1,6-glycosidic bonds [14].

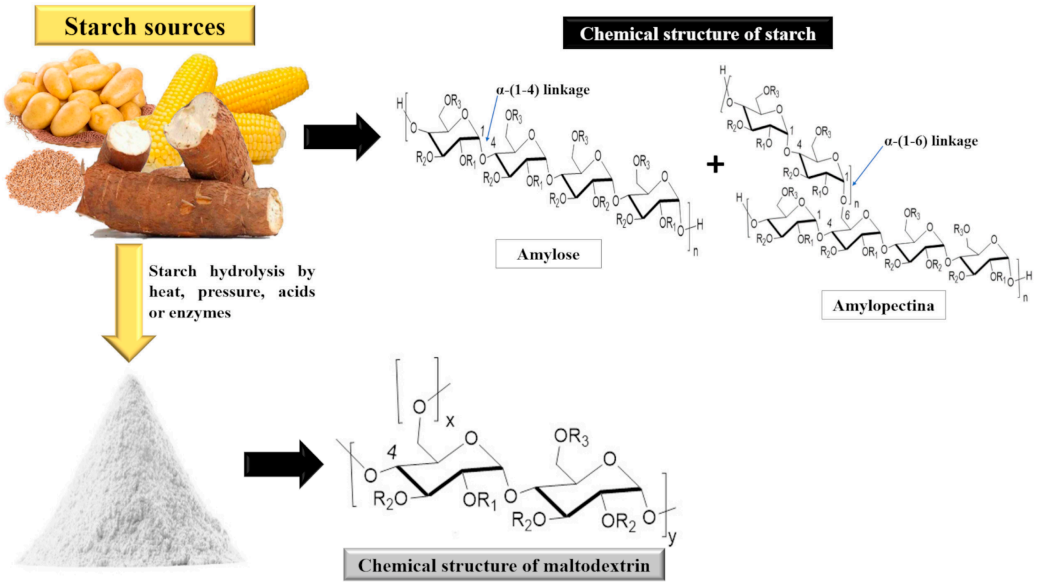


Figure 1. Starch sources, amylose and amylopectin polymers, and maltodextrin are produced by their hydrolysis.

Starch is primarily extracted from corn in the food industry, where it serves as a thickening, emulsifying, gelling, and encapsulating agent [15,16]. However, starch exhibits low thermal stability, retrogradation resistance, and poor water solubility, motivating the use of modified starches to achieve the desired properties [16,17]. Starch can be modified through physical, chemical, or enzymatic processing of the native granules. The chemical modification of starch results from alkaline or acid hydrolysis, cross-linking, and oxidation, as well as the introduction of functional groups, like acetyl-, carboxyl-, ethyl-, or octenyl-, to starch –OH groups [18]. Physical modifications are carried out through thermal processes, such as pre-gelatinization, extrusion, heat-moisturizing, and microwaving, or non-thermal processes, including ultra-high pressure, ultrasonication, and pulse electric field exposure, which alter the granular starch size or arrangement.

It is important to highlight that the use of modified starches has been considered safe by the Joint FAO/WHO Expert Committee on Food Additives [19], which stated an acceptable daily intake (ADI) as 'not specified'. Regarding this, the EFSA Panel on Food Additives and Nutrient Sources added to Food (ANS) issued a scientific opinion on the reassessment of safety issues [20], reporting that modified starches are not a concern for genotoxicity based on in silico analysis. The data on long-term toxicity and carcinogenicity of modified starches are considered sufficient, and there are no safety concerns within the reported levels [20].

Eleven modified starches, included as carriers, are approved as food additives in the European Union list in *quantum satis* maximum level (Regulation EC 1333/2008),

including oxidized starch (E 1404), monostarch phosphate (E 1410), distarch phosphate (E 1412), phosphated distarch phosphate (E 1413), acetylated distarch phosphate (E 1414), acetylated starch (E 1420), acetylated distarch adipate (E 1422), hydroxypropyl starch (E 1440), hydroxypropyl distarch phosphate (E 1442), starch sodium octenyl succinate (E 1450), and acetylated oxidized starch (E 1451). However, the Panel stated that sodium octenyl succinate in dietary foods for special medical purposes and special formulas for infants requires additional evaluation data on the potential health effects, as well as the oxidized, monostarch phosphate, distarch phosphate, phosphate distarch phosphate, acetylated distarch phosphate, acetylated, and acetylated oxidized starches in dietary foods for babies and young children for special medical purposes.

Modified starches such as β-cyclodextrin, maltodextrin, and octenyl succinic anhydride (OSA) are suitable for coating purposes due to their physicochemical properties that protect volatile compounds. Modified starches exhibit emulsifying characteristics, forming oil–water interfaces that enable the entrapment of hydrophobic components and increase their water solubility [21,22]. Starch-based microparticles provide controlled release, gastrointestinal stability, and preserve the biological activity of encapsulated bioactive compounds [16,23].

Hydrolyzed starch (2.5% hydrolysis grade) has been shown to encapsulate 76% of carotenoids from red araçá (*Psidium cattleianum Sabine*) extracts at a 1:1 wall–core ratio (*w*/*w*) using the spray-drying technique. It also improves total phenolic, carotenoid, and anthocyanin retention and maintains antiradical activities of loaded compounds, as demonstrated by 2,2-diphenyl-1-picrylhydrazyl (DPPH•) and 2,2′-azinobis (3-ethylbenzothiazoline-6-sulphonate (ABTS•+) assays. The half-life of starch microencapsulated compounds ranges from 23 to 37 days following microencapsulation at a 1:1 (*w*/*w*) starch: taro gum ratio [24].

OSA–starch microparticles were found to retain 67% of the anthocyanins from jussara (*Euterpe edulis Martius*) pulp, promoting 83% water solubility at a 1:0.5 core-to-wall ratio (*w*/*w*) by spray-drying. This is superior to inulin–maltodextrin microparticles, demonstrating the potential of modified starch alone to retain and confer polyphenol solubility [25]. However, a wall material mixture comprising 66% OSA–starch, 16% maltodextrin, and 16% inulin appears to be the ideal composition to ensure anthocyanin stability and antioxidant polyphenol power following 38 days of storage under unfavorable conditions, such as 50 °C and exposure to light [25]. Encapsulation efficiencies (*EE*) >80% were achieved employing porous corn starch as the coating material for curcumin and resveratrol, both at a 1:10 (*w*/*w*) core-to-wall ratio. However, saturation of the porous starch surface area can restrict polyphenol loading and affect encapsulation efficiency, requiring additional experimental data to better develop this microencapsulation system [26].

Starch derived from taro has also been modified by OSA esterification to coat phytosterol and polyphenols from pomegranate (*Punica granatum* L.) seed oil employing β-cyclodextrin (β-CD) as an emulsifier. The OSA–starch microparticles produced by spray-drying at a 1:3 (*w*/*w*) core-to-wall ratio were shown to encapsulate 61% of total phytosterols and polyphenols. The microencapsulated pomegranate oil released 6.6% and nearly 50% of bioactive compounds under simulated gastric and intestinal conditions, respectively [27]. The bioaccessibility of polyphenols coated in native taro starch by spray-drying was higher, especially at pH 8, where these compounds can become unstable. However, 12% of the encapsulated polyphenols remained stable following intestinal digestion compared to only 2.7% of unencapsulated compounds [28].

A microencapsulated and ready-to-eat beetroot (*Beta vulgaris* L.) soup was formulated using native starch to coat the beetroot matrix, a source of polyphenols, betalains, and nitrates. The encapsulation of beetroot in starch by freeze-drying at a 2:1 starch-to-core

ratio (*w*/*w*) resulted in small and spherical particles capable of encapsulating 55% of the total betalain content, the main bioactive compound (Patent BR1020230151965) [29].

Starches as carriers of polyphenols are found to provide very sustained release under gastrointestinal conditions and other fluids governed mainly by the controlled polyphenols diffusion through the swollen starch matrices, indicating a Fickian diffusion kinetic release mechanism [26,30].

These findings show that starch is a suitable microencapsulation material for polyphenols and pigments that could be applied to the development of new foods that confer health benefits. The studies described here demonstrate that no single starch microparticle composition can be considered superior for delivering active compounds, as each formulation offers specific advantages for the bioactive compound microencapsulated, such as retention, solubility, and stability. This is because the microencapsulation process of bioactive molecules depends on the variability of methodological approaches. Therefore, it is worthwhile to prioritize comparative evaluation under standardized conditions between native and modified starches, as well as their different sources and combinations with other polysaccharides.

2.2. Maltodextrin

Maltodextrin is obtained through acid or enzymatic starch hydrolysis (Figure 1), generating D-glucose polymers linked by α-1,4 and α-1,6 glycosidic bonds, representing about 3% glucose and 7% maltose (*w*/*w*). As hydrolysis extends along the starch chain, maltodextrin polymers with variable molecular weights and reducing-end groups can be generated [31].

Maltodextrins are classified by their dextrose equivalents (DE), which range from 3 to 20 and display variable physicochemical properties, such as water solubility, freezing point, and viscosity, characteristics desirable for use as an additive in food. High DE maltodextrins ranging from 16 to 20 provide significant stability to volatile compounds in foods due to their smoother surfaces, the presence of vacuoles, and more –OH groups interacting through hydrogen bonds [31–33].

High concentrations of maltodextrins form a soluble network of double helical chains and long-chain aggregates, allowing their complexation with bioactive compounds. In addition, maltodextrin has a high glass transition temperature (*Tg*), which contributes to desirable properties as a food additive and microencapsulation, including thermal and acid stability, freezing resistance, and prevention of volatile oxidation. Maltodextrin is usually combined with gum Arabic, modified starch, or inulin to enhance its limited emulsifying capacity, resulting in more stable microencapsulating emulsions [31].

The remarkable capacity of maltodextrin as microparticles for loading polyphenols and pigments has been exploited. Annatto (*Bixa orellana* L.) extracts were encapsulated with an 86% *EE*, achieving 97% solubility, higher bixin and norbixin contents, low hygroscopicity, and 60 days of storage stability [34]. A similar microencapsulation efficiency was reported in maltodextrin microparticles loaded with freeze-dried beetroot (*Beta vulgaris* L.), which displayed 88% betalains *EE* and higher concentrations of phenolic acids, catechin, epicatechin, and myricetin [35].

The encapsulation of black carrot (*Daucus carota* L.) juice in DE 20 maltodextrins through spray-drying and freeze-drying resulted in 96% water solubility and 98% anthocyanins *EE* due to the greater amount of –OH available from the hydrolyzed maltodextrin chain stabilizing anthocyanins. This bioactive stabilization conferred higher antioxidant activity (329 mmol Trolox/g) compared to non-encapsulated juice [36]. The ability of maltodextrin to form a protective film on the surface of the microcapsules is also proposed as a mechanism for the properties conferred by the microparticles [31].

On the other hand, maltodextrins with a lower degree of hydrolysis, as found in DE10 maltodextrins, were able to encapsulate 87% of stevia extract (*Stevia rebaudiana*) by spray-drying, which is higher than the 76% found for DE19 maltodextrin. The authors hypothesized that maltodextrins with greater DE have many free –OH that do not interact sufficiently with each other in the polymer chain, which can result in greater exposure of the active nucleus and affect the encapsulation [37].

In general, there is a lack of studies predicting the behavior and release kinetics of microparticles composed solely of maltodextrins, because this polysaccharide, when used as a carrier of bioactive compounds, may be more effective combined with other polymers. Given these limitations, future experiments should focus on the correlation between the degree of maltodextrin hydrolysis and general microparticle properties, such as release mechanism, standardizing processing parameters, such as techniques of obtention, co-encapsulation, and loaded compounds.

2.3. Alginate

Alginate is a hydrophilic and anionic polysaccharide formed by β-D-mannuronic (M) and α-L-guluronic acid (G) residues arranged in a linear chain through 1,4-glycosidic bonds with specific regions termed M-blocks, G-blocks, and MG-blocks (Figure 2), in which M and G contents are variable according to the source [38–40]. The cell walls of *Phaeophyceae* brown algae are the source of alginate extraction, which exists as a mixture of calcium, sodium, potassium, and magnesium salts [38,41].

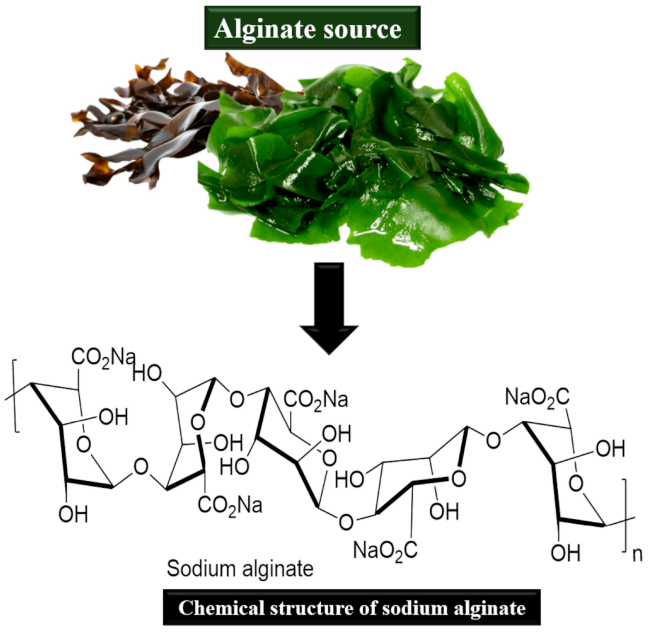


Figure 2. Alginate sources and chemical structure.

Alginate has great application in the food industry due to its ability to form a stable gel-like structure through ionic cross-linking of α-L-glucuronic carboxylate groups in the presence of divalent ions, such as calcium chloride, providing consistency to food preparations and stability to volatile compounds. The gelling property of alginate enables the development of edible films and stable formulations employed in commercial food products and drug delivery systems [42]. The physicochemical properties of alginate have stimulated its employment for therapeutic purposes as an oral delivery vehicle due to its stability in the gastric environment and gradual dissolution under alkaline fluids from the small intestine, making this polysaccharide a suitable vehicle for bioactive compounds stability in oral administration [43].

Alginate can be combined with chitosan through ionic cross-linking between alginate carboxylate moieties and protonated amines of chitosan, forming a three-dimensional entrapment matrix that displays valuable physicochemical characteristics for active microencapsulation carriers [44]. For example, alginate, chitosan, and calcium chloride microparticles have been developed to load phenolic acids obtained from orange peels. These polysaccharides generate 252 μm carriers capable of encapsulating 89% of active compounds, thereby improving antioxidant capacity as evaluated by IC_{50} values. Additionally, with 55% of all compounds are released after two h in a simulated gastric fluid, and 82.5% within 24 h in intestinal fluid [45]. Alginate–chitosan microparticles obtained by extrusion have also been employed to encapsulate a black locust flower extract, with an 92% *EE* and a sustainable release reaching about half the amount present in gastric and intestinal simulated fluids after 2 h and 6 h, respectively. In both assays, antioxidant activity maintenance following gastrointestinal digestion was a result of polyphenol, organic acid, and saponin stabilization from the microcapsule-loaded extracts alongside the synergistic antioxidant power inherent to both chitosan and alginate molecules [46].

Alginate as a microencapsulation coating protects bioactive compound structures in physiological fluids, ensuring their delivery, and guarantees volatile compound stability during storage under harsh environmental conditions concerning light, oxygen, humidity, and temperature. In this sense, flavonoids from the herbal galactagogue were stable at 4 °C and 25 °C for 120 days after being loaded in a water/oil emulsion when covered by sodium alginate microparticles and encapsulated in chitosan. The optimal conditions for flavonoid stability were determined as 1.49% sodium alginate, 0.84% calcium chloride, 1.58% herbal extract, and loading alginate–microemulsions in 1% chitosan. These carrier systems generated 123 μm-sized microparticles capable of encapsulating 77% of all flavonoids and promoting controlled simulated gastrointestinal release for 24 h [47]. Interestingly, alginate-based microbeads can also be employed as β-carotene nanoemulsion stabilizers for 12 days at 55 °C. Alginate microparticles protect lipid-derived structures from lipase activity, conferring up to 87% stability in a simulated gastrointestinal environment by encapsulating nanoemulsions in 0.5% alginate cross-linked with 10% calcium chloride at a 1:1 ratio (core-to-matrix) [48]. Other polyphenols, such as catechin, astaxanthin, and quercetin, are efficiently encapsulated in alginate, alone or combined with chitosan, resulting in an improved release and chemical stability under physicochemical conditions [49–51].

Pure alginate carriers have low mechanical strength, resulting in a rapid release of the loaded compound under harsh environments. In addition, the release of ionically cross-linked alginates is influenced by the hydration rate due to the exchange of Ca^{2+} ions with monovalent cations in the physiological fluids. The swelling of alginate microparticles is highly influenced by the pH of the medium, being greater at pH 6.8 compared to pH 1.2 [52,53]. Although under simulated conditions, the current scientific evidence shows that, through electrostatic interactions, alginate, mainly combined with oppositely charged polysaccharides, displays desirable behavior for carriers intended for the oral delivery of nutraceuticals, as it has exhibited high gastric resistance and sustained intestinal dissolution, with the potential to increase the bioaccessibility of orally administered active compounds.

2.4. Pectin

Pectin is a complex heteropolysaccharide formed by at least 17 kinds of monosaccharides, including D-galacturonic acid as the most prevalent, comprising nearly 70% of monomers, D-galactose, L-arabinose, among others (Figure 3) [54–56]. Pectin refers to a family of polysaccharides from primary cell walls and middle lamella, which contribute to

plant maintenance and present structural variations composed mainly of homogalacturonan, known as the 'smooth' region, and rhamnogalacturonan II, the 'hairy' region. The backbone is composed of D-galacturonic acid units linked by α-1,4 glycosidic bonds [57].

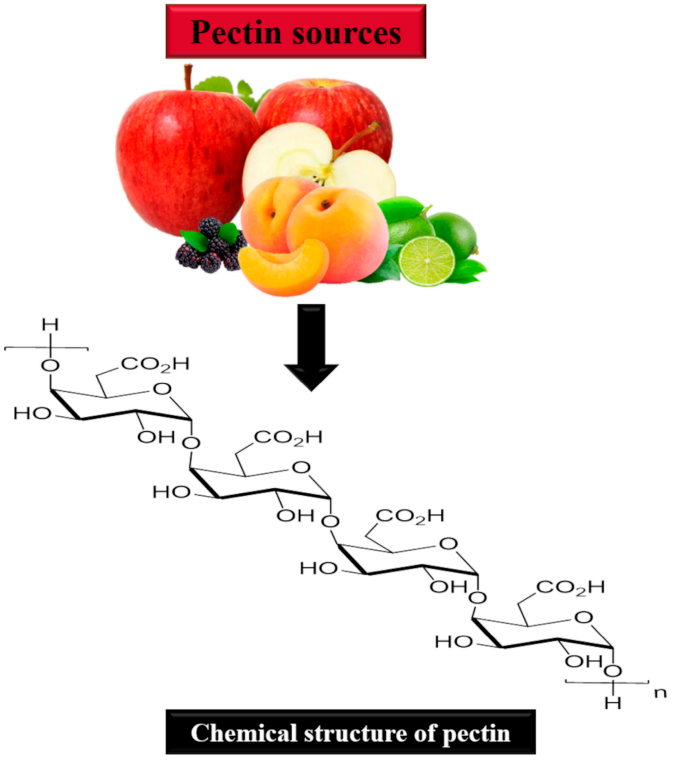


Figure 3. Pectin sources and their general chemical structures.

Pectin is extracted from agro-industrial residues, i.e., sugar beet pulp, citrus peel, and apple pomace, and employed as a food additive thickener, stabilizer, and gelatinizer, due to its rheological and textural properties that improve sensorial food product features [57–59]. The physicochemical properties of pectin are influenced by its structural variability, mainly concerning the esterification degree (ED), the percentage of carboxyl (–COOH) groups esterified with methyl groups, in which ED >50% is considered high methoxylated, and ED < 50% is low methoxylated.

Pectins exhibiting high ED interact with hydrophobic molecules, providing drug loading and controlled release [60–62]. The formation of pectin carriers is underlined by ionic interactions between its –COOH groups in the presence of divalent ions, such as Zn^{2+}. The divalent cross-linking controls the release of encapsulated compounds and, alongside polymers like alginate, promotes the formation of an "egg box" structure, making microstructures very resistant due to the linkage between galacturonate and guluronate blocks [63].

Several reports have demonstrated pectin's structural properties and physicochemical interactions with loaded compounds, which could result in stable complexes capable of protecting and transporting bioactive compounds under environmental and physiological conditions, making them reliable for application in food and nutraceutical formulations.

Pectin microparticles designed for resveratrol entrapment were synthesized by extrusion and cross-linking with Zn^{2+}, resulting in particles with a diameter of 900–950 µm that retained 94% of polyphenols and demonstrated 90 days of storage stability at 25 °C and 40 °C. The pectin microparticles conferred protection to resveratrol greater than 90% in simulated gastric conditions (pH 1.2); in other words, only a small amount of resveratrol is released after 2 h in gastric digestion fluid [64]. Similarly, increased gastroin-

testinal resistance of resveratrol can be achieved through its entrapment in pectin/alginate blend microparticles (polymer ratio 2:1 *w*/*w*) formed by ionotropic gelation with calcium chloride, followed by atomization in a peristaltic pump. This combination stabilizes resveratrol for 2 h in gastric fluid (pH 1.2), releasing less than 10% of the polyphenol [65].

Pectin combined with alginate (55:45 *w*/*w* polymer ratio) and calcium chloride can generate 180 µm microparticles that provide sustained gallic acid release and stability, resulting in 40% and 30% release at pH 1.8 and pH 6.5, mimicking gastric and intestinal fluids, respectively, and complete release after 4 h, a timeframe close to human digestion [66]. Gallic acid microencapsulation of up to 89% efficiency was obtained by air drying, forming 1300 µm pectin–alginate microparticles (1:1 *w*/*w* polymer ratio), with cumulative release percentage ranges of 15–35%, 55–85%, and 50–85% at pH 4, pH 7, and pH 10, respectively [67]. Pectin microparticles were found to release the loaded compounds via matrix swelling and erosion, with very low release (7%) at pH 1.2, and the remaining gallic acid was predominantly released at pH 6 [64]. These characteristics are desirable for intestinal absorption, reinforcing the colon-specific formulation properties.

In short, the physicochemical behavior and compatibility with food components allow the development of pectin carriers based on intermolecular linkages, such as cross-linking and hydrogen bonds with calcium and alginate. These interactions generate a stable network microstructure around dispersed bioactive compounds in the nucleus with no chemical modifications, promoting high gastrointestinal resistance, controlled release, and bioactive content stability, enabling them to be absorbed in the gastrointestinal mucosa. Cross-linking and polymer interactions achieved by using pectins enhance the protection and can improve the bioavailability of bioactive compounds.

2.5. Inulin

Inulin is a polymer comprising β-d-fructose units linked by β-(2-1) glycosidic bonds and a terminal glucose residue. Inulin can exhibit linear or slightly branched chains and a degree of polymerization (DP) from 2 to 60 monomers, categorized as short-chain (DP < 10) and long-chain (DP > 10) inulin, which varies according to plant source and cultivar conditions (Figure 4). The DP variation in inulin underlies its physicochemical behavior, affecting solubility, gelation, and functional roles. In addition, inulin has gastrointestinal resistance and the ability to form a wall matrix, encouraging its use for bioactive delivery [68–71]. Plant members from the Asteraceae family, particularly chicory (*Cichorium intybus* L.), Jerusalem artichoke (*Helianthus tuberosus*), dahlia (*Dahlia* spp.), and agave (*Agave* spp.) roots are the main sources of inulin [70,71].

Inulin is effective as an encapsulating agent when the physicochemical properties and interactions of the entrapped compounds in the inulin structure are understood. Short-chain inulin is particularly suitable for encapsulating hydrophilic polyphenols, such as flavonoids, catechins, epicatechins, and rutin, as well as natural pigments, such as anthocyanins and betalains, due to its lower molecular weight and high –OH content, which make it highly soluble [72]. The number of hydrophilic groups in inulin favors hydrogen bonding, improves encapsulated compound dispersibility, and solubility in aqueous media, being ideal for water-based mixtures and functional foods. In contrast, long-chain inulin exhibits lower water solubility but superior gelling and emulsifying properties, forming a structured polymeric network. This denser matrix protects both lipophilic and amphiphilic compounds, such as resveratrol, curcumin, and carotenoids, from oxidation, light, and temperature degradation, making long-chain inulin useful for conferring storage stability and sustained release of its loaded compounds [68,71,73].

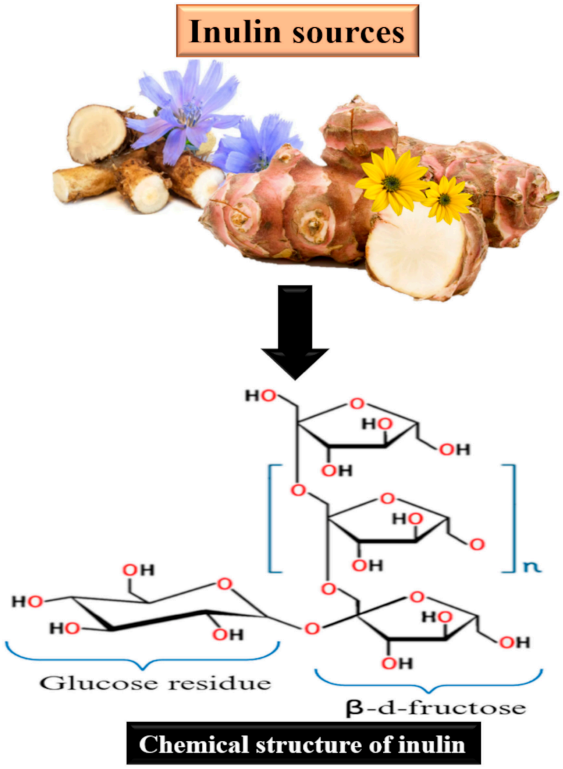


Figure 4. Inulin sources and general chemical structure.

Although inulin has varied physicochemical properties, it is necessary to consider the type of inulin based on the polarity of the encapsulated bioactive to avoid low solubility or poor retention, reducing the performance of the particles as a delivery system [68,73]. The polyphenols hydroxytyrosol and oleuropein from Olive leaf (*Olea europaea*) display strong antioxidant and anti-inflammatory properties, but they are highly susceptible to enzymatic hydrolysis and gastric digestion. However, inulin microparticles retain ~80% of them, improve their stability, intestinal availability, and promote controlled and sustained intestinal release [74]. According to FTIR analyses, the role of inulin in encapsulating polyphenols is attributed to hydrogen bonding and hydrophobic interactions, which inhibit premature degradation of polyphenols. Other studies have confirmed the thermal stability of oleuropein in inulin microparticles, which retained 78% and conferred a 60% release under in vitro digestion [74,75].

Due to its prebiotic properties, inulin microparticles were evaluated as a replacement for modified starch for the encapsulation of lipophilic phenolics from oregano extract (*Origanum vulgare* L.), displaying *EE* for up to 66%, increased thymol retention reaching 84%, and enhanced thermal stability up to 220 °C, evidencing the suitability of inulin as a prebiotic and encapsulant to lipophilic bioactive compounds [76].

Inulin combined with other polymers such as maltodextrin or alginates results in hybrid and stable microparticles that demonstrate enhanced retention, thermal resistance, and bioavailability of the loaded compounds [71,73]. Quercetin encapsulation employing a combination of inulin, alginate, and chitosan to target colonic release has been successful. The presence of inulin enhances microstructural integrity by filling polymeric pores, resulting in improved gel strength and controlled quercetin release. In vitro simulated digestion experiments demonstrated that the encapsulated quercetin obtained by freeze-drying displayed a 50% increase in bioavailability compared to free quercetin, with encapsulation efficiencies ranging from 70% to 85%, followed by sustained release up to 24 h. Fur-

thermore, FTIR analyses revealed strong hydrogen bonding between quercetin and the wall material [73].

Another study demonstrated that employing different ratios of inulin and maltodextrins in the synthesis of spray-dried microparticles loaded with blackcurrant (*Ribes nigrum* L.) compounds confers better phenolic retention and maintenance of antioxidant activity after 12 months of storage at 8 °C and 25 °C, reinforcing the evidence regarding the greater oxidative stability and biological effects maintenance of bioactive compounds encapsulated in inulin microparticles [77].

Inulin microparticles also exhibit high pigment retention, improving thermal, gastrointestinal, and antioxidant stability. In this sense, the microencapsulation of natural pigments, such as betalains, anthocyanins, and carotenoids, within inulin alone or combined with biopolymers has proven effective in terms of thermal stability and color retention. Betalains loaded in inulin spray-dried microparticles, for example, resulted in a 58% *EE* and promoted improved color stability to sorbets over six months at −18 °C [78]. Similarly, betalain encapsulation using inulin alone, or combined with maltodextrin (IN-MD) or whey protein isolate (IN-WPI) by spray-drying resulted in over 88% retention of betalains. While the different polysaccharide coatings did not significantly affect retention, the IN-WPI mixture exhibited the highest powder stability [79]. However, when betalains and other related pigments (betacyanins and betaxanthins) were encapsulated by freeze-drying in inulin or maltodextrin as carrier agents, maltodextrin-based microparticles retained the highest betalains content (15.7 mg/100 g) compared to inulin (10 mg/100 g). This lower retention has been attributed to the relatively low glass transition temperature ($Tg \approx 27$ °C) of inulin-based powders, which makes them more prone to collapse and degradation under warm or humid storage conditions. In contrast, maltodextrin delivery systems exhibit a *Tg* of approximately 60 °C and greater stability. Therefore, inulin may perform better in frozen or refrigerated products; however, its application in thermally processed foods often requires blending with higher *Tg* polymers to ensure structural stability [80]. An inulin-alginate blend has been shown to improve *EE* from 79% to 89% upon increasing the inulin content from 10% to 20% (*w*/*w*), reinforcing the potential of inulin combined with other polysaccharides for pigment stabilization [81]. The potential of inulin microparticles combined with polymers was found to improve the bioaccessibility of anthocyanins from maqui (*Aristotelia chilensis*) through inulin complexation with alginate by spray-drying, which confers 79% *EE* and anthocyanins bioaccessibility enhancement [82]. Inulin can be considered for betalain encapsulation, although its performance is improved by combination with proteins or maltodextrin to achieve consistent stability across storage conditions. Although hybrid systems enhance stability, they also complicate the standardization of wall formulations, since performance will depend on inulin chain length and the physicochemical interactions with co-polymers. Such variability challenges the reproducibility across batches and processing conditions [71,73].

Encapsulation by physical methods also affects the encapsulation properties of inulin, as assessed, for example, by comparing spray-drying and freeze-drying in the encapsulation of a Roselle (*Hibiscus sabdariffa*) extract in an inulin–maltodextrin microparticle. In this mixture, the spray-drying technique favors the total polyphenol content, anthocyanin retention, and maintains the antioxidant activity [83]. In addition, spray-dried inulin–maltodextrin microparticles encapsulated 81% of anthocyanins from a mangosteen pericarp extract (*Garcinia mangostana*) and increased half-life (t1/2) of 3 days when incorporated as a yogurt ingredient stored at 4 °C [84].

Other pigments, such as carotenoids, have been shown to benefit from inulin encapsulation. Inulin–fructooligosaccharides (FOS) microparticles confer 47% bioaccessibility to carotenoids from mango peel (*Mangifera indica*) compared to 30% bioaccessibility of

carotenoids in FOS microparticles. Ultramicroscopy revealed post-digestion micelle-like formations, which facilitated β-carotene solubilization and increased its uptake in Caco-2 cells [85]. Similarly, the encapsulation of lycopene from tomato (*Lycopersicon esculentum*) in inulin microparticles synthesized by spray-drying led to a 26% final release of microencapsulated lycopene during simulated intestinal digestion, whereas only 15% of free lycopene was recovered, suggesting that encapsulation significantly enhances intestinal lycopene delivery and consequent bioaccessibility [85,86]. Inulin microparticles are characterized by displaying short release of loaded compounds in aqueous media, due to their low solubility compared to other polysaccharides. Previous data reported the release of less than 30% of the entrapped compound after 2 h in the gastric conditions, followed by the simulated intestinal digestion, suggesting its effectiveness for gradual release and protection of bioactive compounds [86]. Inulin emerges as a versatile biopolymer with effective capacity to stabilize and deliver as a microparticle. Its structural variations and combinations with other polymers have a positive influence on its behavior.

2.6. Chitosan

Chitosan is derived from the alkaline deacetylation of chitin, a biopolymer found in exoskeletons of crustaceans, fungi cell walls, insect cuticles, and certain microalgae. It consists of a non-branched chain composed of β-(1,4)-linked D-glucosamine (deacetylated units) and N-acetyl-D-glucosamine (acetylated units), exhibiting over 60% of deacetylation degree (DD) and molecular weight (Mw) from 50 to 2000 kDa (Figure 5) [87–90].

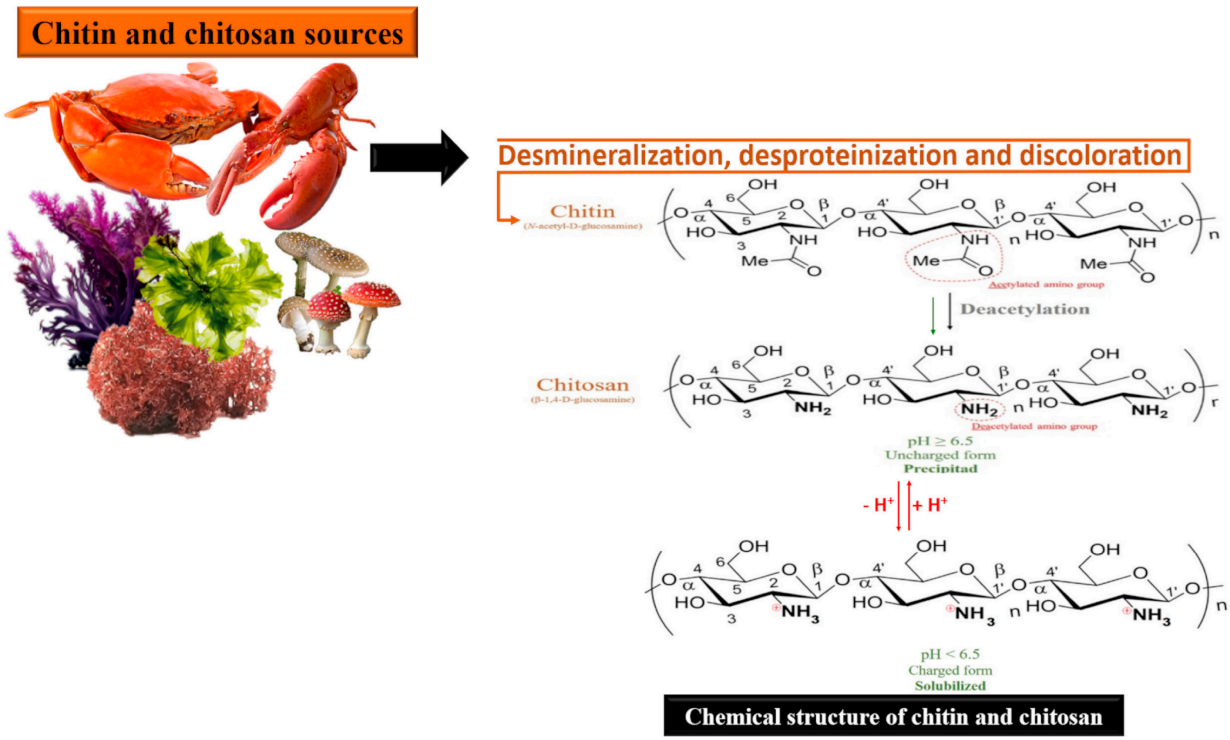


Figure 5. Chitin sources and deacetylation forming chitosan.

Chitosan undergoes chemical, enzymatic, and/or physical degradation, generating oligosaccharides with specific biological properties. Moreover, its reactive $–NH_2$ groups enable the synthesis of derivatives displaying improved water solubility, antioxidant, and anti-inflammatory activities. These physicochemical characteristics enhance the stability and bioavailability of entrapped molecules while also enabling chitosan formulations as solutions, films, hydrogels, fibers, nanoparticles, and microparticles. The versatility of chi-

tosan enhances its employment for the development of pharmaceutical, food, agricultural, and environmental products [91–95].

The physicochemical and biological roles of chitosan are influenced by its DD, that are its molar fraction of repeating units of free amino groups ($–NH_2$) and the proportion of N-acetylated units, respectively, expressed as a percentage of total units. Chitosan Mw, ionic strength, pH, temperature, and solvent polarity also induce different behaviors of chitosan.

Lower DA of chitosan results in a high content of protonatable $–NH_2$ groups, which under acidic conditions (pH < 6.5) become positively charged ($–NH_3^+$) and more water-soluble. Conversely, high DD promotes strong hydrogen bonding and hydrophobic interactions due to the presence of N-acetyl-D-glucosamine units, leading to increased crystallinity and reduced solubility [93,96]. The Mw significantly affects the impact on chitosan functionality, since low Mw chitosan displays enhanced gastrointestinal permeability and absorption through epithelial barriers, while high Mw ones contain a higher density of cationic charges, which tend to exhibit stronger interactions with cell membranes [89,97]. The cationic nature of chitosan allows its interaction with various biomolecules via electrostatic attraction, hydrogen bonding, and hydrophobic interactions [93,98,99].

Chitosan properties make it prone to form polyelectrolyte complexes, enabling its use as an agent for drug entrapment and synthesis of carriers for bioactive delivery [89,90,97,99]. In addition, chitosan provides mucoadhesion, which enhances the permeability of encapsulated compounds and their controlled release [93,97,100]. These mucoadhesive properties are valuable in oncology, as they enhance the therapeutic effectiveness of anticancer compounds by improving drug retention in tumor sites. Moreover, chitosan exhibits antibacterial properties contributing to reducing post-treatment infections, further supporting its biomedical relevance [99,101–103].

Chitosan can co-encapsulate bioactive compounds alongside other encapsulating biopolymers, forming more stable delivery systems. Microparticles based on chitosan–polysaccharide mixture, such as inulin, alginate, or pectin, can enhance the bioavailability of loaded compounds [104,105]. The co-encapsulation of blackcurrant (*Ribes nigrum*) anthocyanins in a blended matrix composed of chitosan, whey protein isolate (WPI), and inulin through freeze-drying produced a complex microstructure encapsulating 95% of the anthocyanins, conferring resistance to gastric fluids and 94% sustained release after 2 h at the intestine [106]. In addition, anthocyanin cargo load was stable for 90 days at 4 °C due to electrostatic interactions between functional groups of chitosan and anthocyanins. Chemical interactions, along with hydrogen bonding between anthocyanin –OH and $–NH_2$ and –C=O groups between chitosan/WPI, and hydrophobic interactions with WPI and inulin, underlie enhanced anthocyanin properties [106].

Glutaraldehyde–cross-linked chitosan microparticles are suitable for the encapsulation of polyphenols from *Thymus serpyllum* by emulsion cross-linking, in which chitosan concentrations ranged from 1.5% to 3% (*w*/*v*), and the glutaraldehyde-to-chitosan mass ratio varied from 0.15 to 1.20 (*w*/*w*), tailoring the physicochemical properties of microparticles. Interactions between the polyphenol compounds and the chitosan matrix took place mainly through hydrogen bonding between the phenolic compound –OH groups and the $–NH_2$ and –OH chitosan groups, as demonstrated by X-ray diffraction analysis, which showed that chitosan crystallinity increased following glutaraldehyde cross-linking and polyphenol encapsulation [107]. Blueberry anthocyanins loaded in chitosan microparticles cross-linked with either cellulose nanocrystals or sodium tripolyphosphate (TPP), displaying 94% *EE* of anthocyanins in the chitosan–cellulose particles and favorable distribution within the matrix [108].

There are few studies evaluating the release profile of loaded compounds on chitosan microparticles, but it has been proposed that the release occurs through swelling of the wall material, which is inversely proportional to the concentration of the cross-linking agent used in the formulation. In other words, the higher the cross-link density, the less flexibility and free volume between the polymer chains. Water diffusion into the chitosan wall matrix occurs at a zero-order Fickian mechanism [108,109]. During gastrointestinal digestion, for example, low pH values of gastric fluids lead to repulsion of polymer chains, eliciting water diffusion and swelling. The mathematical–empirical Weibull model for polyphenol release kinetics has also been suggested.

Chitosan represents a multifunctional biopolymer with suitable physicochemical and biological properties that support its wide applicability in food, pharmaceutical, and biomedical fields. Its cationic nature, tunable Mw, and DD enable interactions with diverse bioactive compounds, conferring greater retention, protection, and controlled release.

2.7. Gum Arabic

Gum Arabic is a complex and highly branched polysaccharide containing magnesium, calcium, and potassium salts. The main chain is formed by β-D-galactopyranosyl units linked by 1,3-linkages, and side chains containing different arabinofuranose, galactopyranose, rhamnopyranose, and uronic acid (glucuronic or galacturonic) units linked by 1,6-glycosidic bonds. Uronic acid and galactose are found in β-D form, whereas arabinose and rhamnose are observed in α-L form (Figure 6). The structure of gum Arabic contains small amounts of amino acids, mainly hydroxyproline, aspartic acid, proline, and serine [110–113].

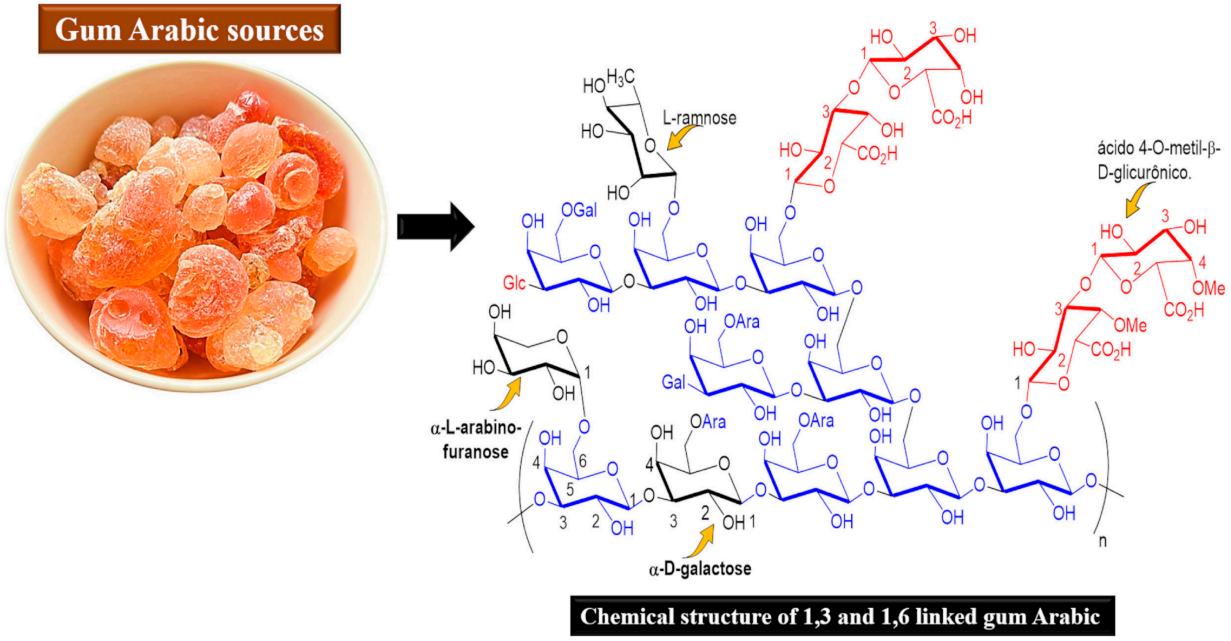


Figure 6. Gum Arabic sources and general chemical structure.

Gum Arabic is the oldest and most well-known exudate gum produced by the Acacia Senegal tree and commercially available in granular, flake, and powdered forms exhibiting odorless, hard, and tasteless after processing [111,112]. As a food additive, gum Arabic acts as a stabilizer for oil-in-water emulsions and an enhancer of sensory attributes of food formulations and cosmetic products. As a soluble dietary fiber, it is absorbed intact in the small intestine, allowing intake of substantial daily doses with no adverse effects. These

properties make gum Arabic widely employed as a wall matrix for stabilization of active molecules designed for oral delivery [113].

Gum Arabic stability in carrier systems is influenced by pH due to the amphoteric nature of the polysaccharide chain. At high pH values, the –COOH group can lose a proton (H^+), becoming $–COO^-$ with a negative charge, while at low pH, the amino groups within the protein fraction are protonable, forming a positive charge. Therefore, when these groups ionize, gum Arabic becomes more water-soluble [114]. Several assays carried out using gum Arabic as a carrier of bioactive compounds are found in the literature.

Leaf extracts from *R. Tuberosa* L. and *T. Diversifolia*, sources of polyphenols and terpenoids, can be loaded under optimal conditions in gum Arabic 4% (*w*/*v*), pH 5, and 60 min of stirring, followed by freeze-drying. The *EE* of gum Arabic was found to increase at pH 5, as some gum Arabic acid groups are not fully ionized, resulting in low viscosity and optimal interactions [114]. Flavonoids from ponkan peel extracts were microencapsulated by spray-drying using 5% (*w*/*v*) soybean oil in aqueous gum Arabic solution (10%, *w*/*v*) and/or 2.5% (*w*/*v*) soybean oil, 0.5–5% (*w*/*v*) WP concentrate in aqueous gum Arabic dispersion (5%, *w*/*v*). When 100 mg of ponkan peel extracts was added to gum Arabic-stabilized oil- and WP-in-water emulsions, the microparticle sizes increased to 1 μm and 1.4 μm, respectively. However, when the pH increased from 2 to 7, the average size of the microparticles reached approximately 1 μm, while the droplet diameter measured around 0.87 μm. At pH 7, gum Arabic with a stronger negative charge was adsorbed onto the oil–water droplet surfaces, enhancing the stability of the interfacial layer through electrostatic repulsion and preventing droplet flocculation. The microencapsulation maintains the antioxidant activity of the flavonoids across all emulsions, with this activity closely tied to the concentration of flavonoids. Notably, emulsions stabilized solely with gum Arabic-stabilized oil-in-water emulsions exhibited higher antioxidant activity compared to gum Arabic-stabilized in oil- and WP-in water emulsions, likely due to the reduced flavonoid concentration—approximately half—in the WP-based formulations [115].

Spray-dried curcumin microencapsulation using coconut milk whey and/or gum Arabic (5, 10, and 15%) demonstrated that increasing gum Arabic concentrations significantly improved *EE*. The resulting microcapsules exhibited a rough surface, providing a greater effective encapsulation area. The gum Arabic added to the curcumin–coconut milk whey solution occupies empty spaces in the carrier matrix, increasing wall material quality and stabilizing this component by reducing oxygen permeability. Smaller particle sizes increased the available surface area for curcumin encapsulation, leading to higher curcumin retention with increasing gum Arabic concentrations. Gum Arabic combined with coconut milk whey may improve powder retention compared to a single carrier material, thereby enhancing the attributes and offering a suitable alternative for encapsulating polyphenols. Furthermore, the curcumin degradation rates are directly proportional to temperature and can be decreased by increasing the gum Arabic concentration in the coconut whey–gum Arabic complex [116].

Anthocyanins from barberry (*Berberis vulgaris*) extract were easily degraded during storage and processing; however, their microencapsulation by spray-drying employing an optimized combination of gum Arabic and maltodextrin at a core/wall ratio of 25% inhibits anthocyanin instability, even compared to anthocyanin microencapsulated only in maltodextrin. The branched structure of gum Arabic and its covalent linkages to amino acids, which are closely associated with the carbohydrate chain, may act as a film-forming agent for the entrapment of bioactive molecules. Therefore, gum Arabic interactions make the flavylium cation of anthocyanins less vulnerable to nucleophilic attack by water molecules, in addition to flavylium interactions with dextrins, thereby preventing anthocyanins transformation to less stable forms [117]. Buriti (*Mauritia flexuosa*) oil pulp, composed mainly of

β-carotene, was loaded in different gum Arabic–inulin mixture ratios. The microparticles composed of gum Arabic–inulin mixture (50% *w*/*w*) displayed 93% *EE*, 91% β-carotene retention, and 71% water solubility, being superior to microparticles composed of gum Arabic–inulin 25/75% (*w*/*w*) and 75/25% *w*/*w* [118].

In general, the synthesis of gum Arabic microparticles is combined with other wall polymers. However, gum Arabic as the sole wall material can exhibit *EE* greater than 80% for loading flavonoids from plant extract and inhibitory activity on alpha–amylase enzyme and DPPH radical under optimal conditions already mentioned. An interesting finding is that the release profile of gum Arabic microparticles may not be influenced by pH fluids between pH 2.2 and pH 7.4, but perhaps above that range [114]. On the other hand, significant degradation in acidic pH and resistance to degradation in alkaline pH have been reported [119]. The release of compounds occurs by swelling and follows a second-order kinetic rate. This lack of pH influence can be an important finding considering the stability of bioactive compounds encapsulated in gum Arabic in different physiological fluids, but these behaviors require further investigation.

In short, gum Arabic demonstrates significant potential as an encapsulating agent for polyphenols, pigments, and oils, owing to its amphoteric nature, pH-dependent solubility modulation, and film-forming capacity. Its performance and versatility as a drug carrier are further enhanced when combined with other biopolymers, conferring a broad applicability in the development of functional foods and nutraceutical formulations.

3. A Brief Overview of the Putative Interactions Between Polysaccharides and Bioactive Compounds in Drug Carrier Systems

Polysaccharides, as bioactive carriers, can promote health benefits by enhancing the stability of bioactive compounds and displaying synergistic effects through their functional roles in obesity, type 2 diabetes, hypercholesterolemia, and gut microbiota health [120–122]. Based on the health benefits, there is considerable interest in developing polysaccharide microparticles and understanding the chemical interactions underlying bioactive retention, which provides a basis for choosing suitable wall materials for bioactive delivery [123].

Polysaccharides form delivery systems by binding with bioactive compounds through hydrogen and ionic bonds, Van der Waals forces, and electrostatic and hydrophobic interactions [124]. On the other hand, covalent linkages between polysaccharides and bioactive compounds usually require conjugation with proteins [125]. The supramolecular interactions between polysaccharides and bioactive compounds are still underexplored and restricted to polyphenol–polymer chemical interactions, although physical linkage has been found to enhance the retention of the microencapsulate compound [31,35,76,117,126].

The *EE* is one of the main properties of encapsulation systems, and can result from intermolecular interactions between specific functional groups of the wall material and the active nucleus. The availability of –OH groups in polysaccharides has been proposed to influence the retention of bioactive compounds, explaining, for example, the different behaviors and retention capacities of maltodextrin microparticles related to the DE degrees. Maltodextrins with high DEs contain more –OH groups available for interactions, as evidenced by DE20 maltodextrin microparticles encapsulating 98.5% of anthocyanins [24,36]. Similarly, short-chain inulin exhibits greater polyphenol and hydrophilic pigment retention due to its higher –OH contents compared to long-chain ones [71]. Hydrolyzed starch was found to retain higher total polyphenols and anthocyanins, which was attributed to the high number of –OH hydrogen bonds, and dipole–dipole interactions with free –OH in polyphenols and anthocyanin flavylium cations [24]. It is worth noting that the increased hydrophilicity of the wall matrix favors the retention of hydrophilic compounds within the amylose helix structure of maltodextrin, as

evidenced by microparticles composed of inulin and maltodextrin mixtures, which display greater anthocyanin retention compared to starch alone [25]. A schematic illustration of starch interactions with polyphenols based on the availability of –OH groups is represented in Figure 7.

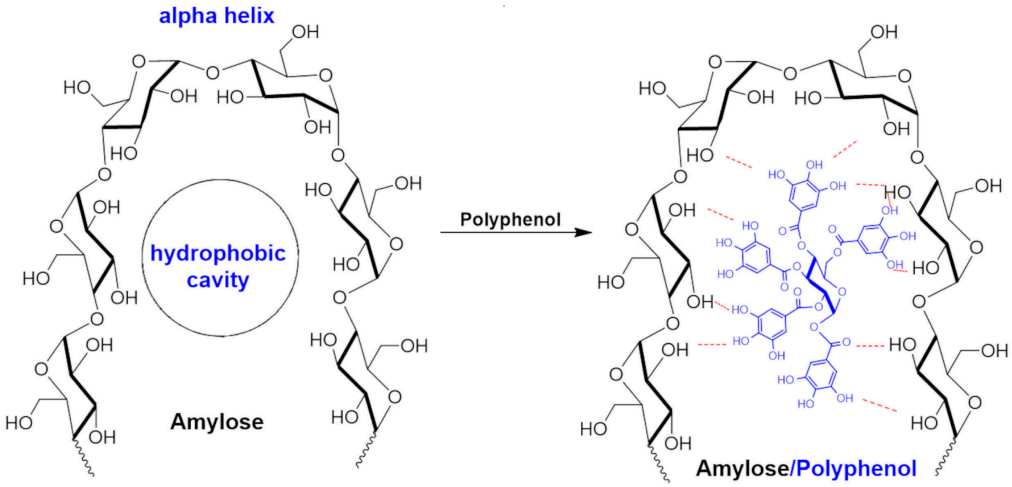


Figure 7. Hydrophobic interaction of beta–carotene with the amylose helix structure cavity.

The increased DE degree also favors encapsulation governed by hydrophobic interactions since polysaccharides with higher DE have more exposed hydrophobic regions available for binding with hydrophobic bioactive and emulsions [127,128]. Corroborating this, hydrolyzed starch was found to retain more than 70% of β-carotenes inside the amylose helix cavity [24], and an illustrative hydrophobic interaction is suggested in Figure 8. Although many studies suggest a strong intermolecular relationship influenced by the number of –OH groups, hydrogen bonding, and hydrophobic interactions between polysaccharides and bioactive, specific analyses—usually carried out by FTIR spectroscopy—confirming these hypotheses are lacking.

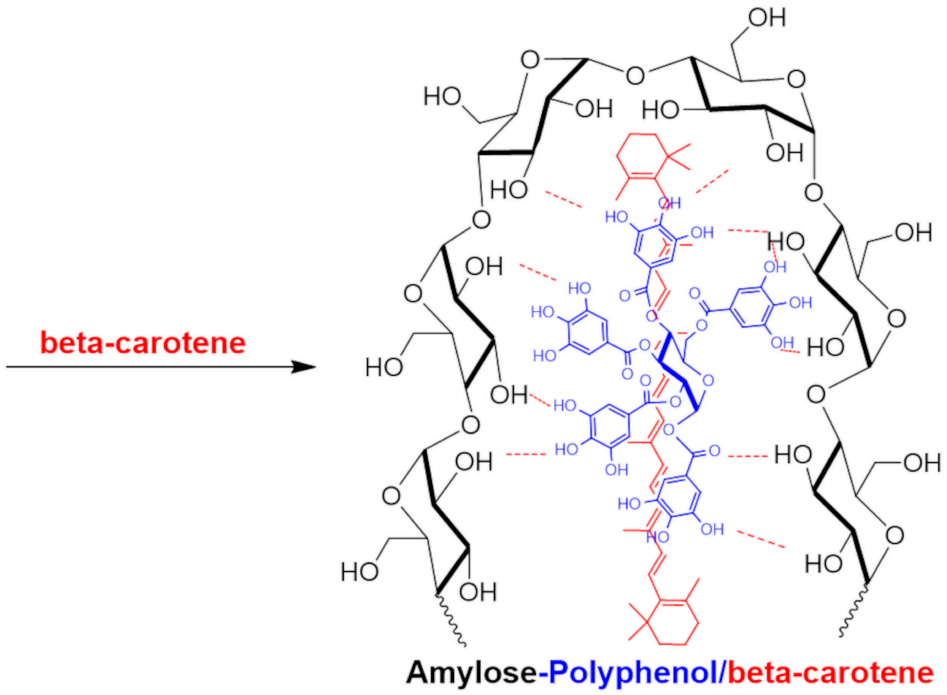


Figure 8. Interactions between polyphenols and the amylose helix structure cavity.

On the other hand, in the inulin–alginate microparticles co-encapsulated with polyphenols and betacyanins, FTIR spectroscopy showed hydrogen bonds between the polar groups of the wall materials, such as –OH and –COOH groups of the bioactive compounds [81]. Hydrogen-bonded OH bands have also been evidenced between bioactive compounds from pink pepper (*Schinus terebinthifolius*) extract loaded in maltodextrin, as well as in the alginate–agar microparticles loaded with green tea extract, exhibiting five hydrogen bond-donating OH groups from catechin, influencing the hydrogen-bonding pattern of intermolecular interaction between wall polysaccharides [129,130].

In addition to the polysaccharide–bioactive interaction, bioactive encapsulation can be achieved through the synthesis of microparticles formed by bonds between polysaccharide chains combined in the polymeric wall surrounding the nucleus. Polysaccharide delivery systems based on the mixture of two or more polysaccharide chains have been obtained mainly by ionotropic gelation, in which oppositely charged wall components, such as negatively charged sodium alginate, pectin, and inulin, are bonded to the positively charged chitosan, as represented in Figure 9.

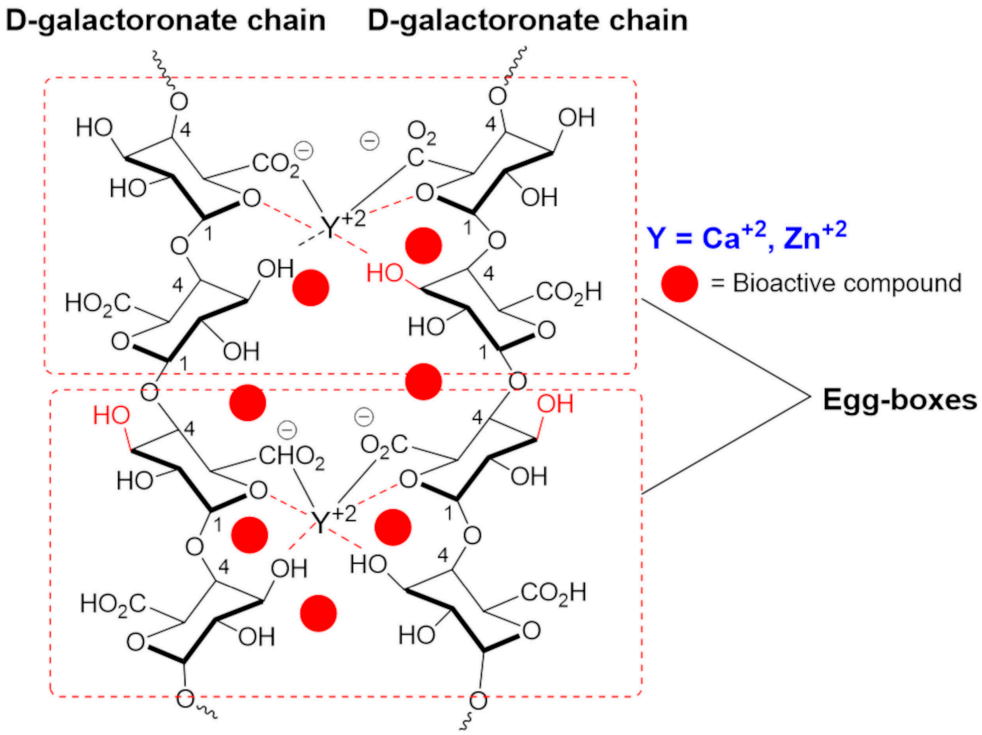


Figure 9. Egg-box structure formed by alginate D-galacturonate and D-guluronate chain interactions in the presence of divalent ions.

Polysaccharides can be cross-linked to themselves or to reticulant agents such as polyanion TPP, divalent ions Ca^{2+} or Zn^{2+}, through electrostatic interactions, leading to the formation of a hydrogel network like an "egg-box" around the bioactive nucleus, as represented by alginate D-galacturonate and D-guluronate chains, forming a network through divalent ions cross-link, schematized in Figure 10 [44,63,104,131]. A resistant microstructure composed of a polysaccharide blend was formed through electrostatic interactions of –COO groups from alginate to protonated $–NH_2$ chitosan groups, capable of encapsulating 89% and 92% of orange peel and black locust flower bioactive extracts, respectively [45,46]. In addition, blending alginate and pectin resulted in 90% gallic acid encapsulation through physical interactions between the alginate –COOH group and the pectin carbonyl groups (–C=O), according to FTIR spectra, with no signal of linkage between the wall matrix and gallic acid, showing that the bioactive compound can be

only surrounded by physical interaction between the polysaccharides [67,132]. In addition, through the formation of a network microstructure by electrostatic interactions, polyphenol and betacyanin were efficiently co-encapsulated at 79% and 89%, respectively, in inulin–alginate microparticles through external ionotropic gelation reaction with Ca^{2+}, although interactions between polysaccharide and bioactive were also observed [81].

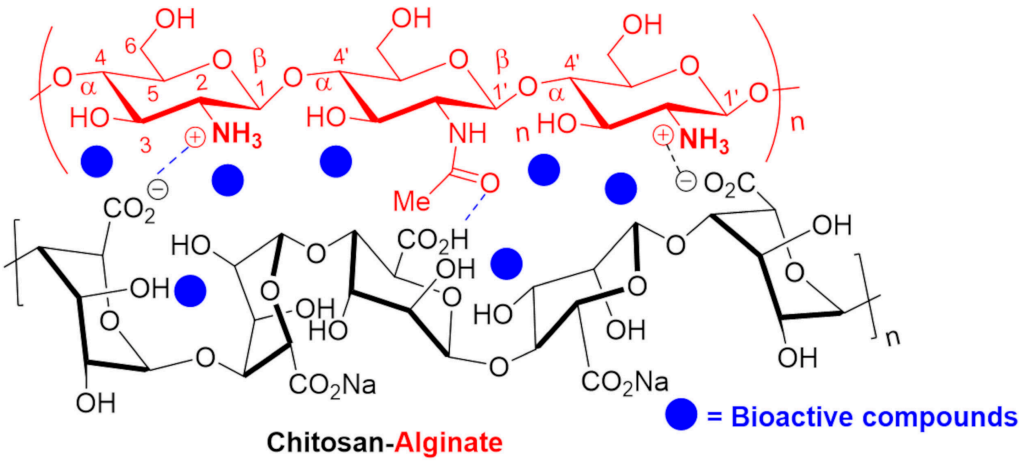


Figure 10. Egg-box structure formed by electrostatic interactions between sodium alginate and chitosan.

It is important to note that polysaccharide–bioactive linkages take place in ionic gelation-based microparticles alongside polysaccharide blends around the nucleus. Hydroxyl groups from alginate, inulin, and gum Arabic form intermolecular hydrogen bonds with bioactive core molecules [71,129,133]. Chitosan can retain active molecules through several intermolecular linkages, including hydrogen bonds between its $–NH_2$ groups and polyphenol –OH groups, electrostatic reactions between opposite-charged chitosan and polyphenol groups, and hydrophobic interactions between hydrophobic chitosan surface areas and the hydrophobic moieties of polyphenols [134]. On the other hand, interactions between pectin and bioactive molecules are poorly understood and contradictory. The absence of binding between pectin and polyphenols in microparticles was evaluated through FTIR spectra analyses, indicating no covalent or other type of bonds to pectin [135,136]. Therefore, other studies suggest the occurrence of hydrophobic interactions with polyphenols, influenced by the high methylation of pectin (>60%), through bonds between methyl groups and C-rings, as well as hydrogen bonds between carboxyl groups and phenolic hydroxyls, as evidenced by their lower methoxyl content [137,138].

In summary, several alternatives for the development of microparticles based on edible polysaccharides to entrap polyphenols and pigments have been proposed, showing the ability of those delivery systems for coating hydrophilic and hydrophobic compounds through polysaccharide–polysaccharide and polysaccharide–bioactive physical linkages, with potential to transport, chemical structure, and biological activity at a physiological environment following oral intake.

4. Microencapsulation Techniques

Several technologies have been developed to load polyphenols and pigments into polysaccharide carriers. The encapsulation methods described herein are the most employed for the development of polysaccharide–bioactive delivery systems—the focus of this review. The correct choice of polysaccharide, either single or combined with others, is a crucial step for successful microencapsulation, preservation of loaded compounds under harsh physicochemical conditions, and sustained release. The most commonly employed

physical methods include spray-drying, freeze-drying, and extrusion, while the most commonly employed physicochemical methods, such as coacervation and emulsification, are generally used in conjunction with physical methods [139]. It is plausible to state that the ideal technique depends on the balance between the characteristics of the active compound, the interaction with the encapsulating polymer, and the functional role intended for the final product.

4.1. Freeze-Drying

Freeze-drying, also known as lyophilization, involves dehydration in which a solvent or suspension medium is frozen at reduced temperatures and then undergoes direct sublimation from the solid state to the gaseous state. This process comprises three main steps: freezing, water/solvent sublimation under vacuum in the primary drying phase, and desorption of the small amount of bound water in solids during secondary drying, resulting in a dry material [140].

In this process, temperature and pressure are associated with the solid, liquid, and gaseous states of water, at which the intersection of these three phases (known as the triple point) occurs at 0.0098 °C and 4.58 mmHg (or 0.00603 atm), allowing all states to occur simultaneously. Freeze-drying occurs below the triple point, resulting in the direct conversion of ice into vapor [141]. This process promotes the retention of chemical constituents and the stability of bioactive compounds derived from plant, microbial, or animal sources, ensuring a high-quality final product. Regarding structure, lyophilized material exhibits a porous surface and retains its sensory attributes, particularly texture [102]. Due to this, freeze-drying is one of the most widely employed techniques for encapsulating bioactive compounds from edible plants, thereby maintaining their preservation [142]. A beetroot soup rich in polyphenols and dietary nitrate was microencapsulated by freeze-drying in starch or maltodextrin, producing spherical particles ranging from 7.9 to 636 μm. The microparticles remained stable at room temperature for over 90 days, and the sensory characteristics of the beetroot were preserved, resulting in high consumer acceptance. This novel product has been patented according to the Brazilian National Institute of Industrial Property standards under number BR 1020230151965 [29]. The freeze-drying methodology has also been employed for the microencapsulation of anthocyanins from juçara fruit (*Euterpe edulis*) in maltodextrin and gum Arabic, retaining 151 mg/100 g and preserving over 80% of polyphenol content [143].

Freeze-drying, however, presents some disadvantages, such as a higher price compared to conventional drying methods, due to operating costs, as the equipment's cold chamber and vacuum pumps consume high energy. Furthermore, microencapsulation by this method can lead to polyphenol degradation after thawing, as cells disrupted during freezing extrude their hydrolytic enzymes [144,145]. However, the whole process can release bioactive compounds from food matrices, as described for flavonoids obtained from onions [146]. A similar effect was observed in anthocyanin-rich powders obtained by freeze-drying, where anthocyanin retention was 1.5-fold higher than in atomized powders. The freeze-drying effect was compared to spray-drying for encapsulating coffee powder extract in maltodextrin, gum Arabic, or a combination of both at the same ratio. Polyphenol and flavonoid retention were optimized following freeze-drying in maltodextrin [147].

The antioxidant power of encapsulated compounds can be preserved by freeze-drying [1]. Inulin, combined with modified starch, whey protein, and gum Arabic, has been employed to encapsulate geranylgeraniol from annatto (*Bixa orellana*) oil seeds through ultrasound-assisted emulsification followed by freeze-drying. All wall material combinations retained at least 80% of geranylgeraniol antioxidant activity after freeze-drying, with the gum Arabic–inulin combination preserving 96% of its antioxidant

power [148]. In another study, maltodextrin–gum Arabic microparticles loaded with sugar maple exhibited an antioxidant capacity up to 241 μM TE/g (Trolox equivalents) by the cupric reducing antioxidant capacity (CUPRAC) method throughout storage, preserving over 95% of phenolic and flavonoid compounds after freeze-drying [149]. These findings reinforce the effectiveness of the freeze-drying methodology combined with polysaccharides for stabilizing antioxidant compounds and preserving their bioactivities.

4.2. Spray-Drying

The spray-drying methodology transforms liquids into powders by atomizing them using a hot gas injector for drying [150]. The process consists of three main steps: (i) liquid homogenization by an atomizer, (ii) liquid drying using a hot gas carrier to evaporate the solvent, and (iii) collection of the dried particles by cyclones or filters [151]. The liquid is a solution, emulsion, or suspension injected into the drying container through a nozzle or atomizer, producing droplets that evaporate the solvent, resulting in dry particles [152–155].

Spray-drying is widely used for the microencapsulation of bioactive food compounds due to its practicality, low operating volume, and favorable cost–benefit ratio [155]. The most common encapsulating agents used in this process include polysaccharides, gum Arabic, cyclodextrins, maltodextrins, gellan gum, chitosan, whey protein, soy protein, gelatin, and sodium caseinate [156].

The most significant concerns associated with spray-drying are the high temperatures employed, which can lead to thermal product damage, surface cracks, and loss of volatile compounds [157]. Concerning the encapsulation of polyphenol, the oxygen exposition facilitates oxidative reactions, while high temperatures accelerate thermal degradation and polymerization, resulting in loss of biological properties [158]. Additionally, the rapid water outflow for evaporation at high temperatures can lead to microparticle surface damage and the premature release of core compounds [159].

However, spray-drying has been successfully applied to microencapsulate several food components, including flavorings, dyes, vitamins, minerals, fats, and oils, and its limitations do not appear to override the protective encapsulation effects regarding bioactive compound stability [160]. The encapsulation of phenolic extracts from apple peel (*Malus domestica*) by spray-drying in maltodextrin–WP microparticles coated by gum Arabic resulted in spherical microparticles, from 315 to 719 μm, and *EE* up to 83%, designed for yogurt enrichment. The microparticles exhibited high probiotic activity for up to 15 days of storage, indicating the stability of encapsulated polyphenols during spray-drying and maintenance of probiotic viability [161]. In another study, an acerola extract (*Malpighia emarginata*) rich in carotenoids and polyphenols was encapsulated in gum Arabic and maltodextrin by spray-drying, generating highly soluble microparticles retaining 1000 mg GAE/100 g (Gallic acid equivalent) and 80 to 99% of anthocyanins from maple grape skin (*Vitis labrusca*) in gum Arabic microparticles [162]. The speed and effectiveness of the method ensure the production of small microparticles with satisfactory morphology, no microbial contamination, and low cost [152,162,163].

4.3. Coacervation

Coacervation is widely employed for the microencapsulation of food matrix compounds due to its low-temperature operating conditions, which attenuate thermal degradation and prevent volatile compound loss. This process separates liquid–liquid phases in colloidal solutions containing oppositely charged polymers that bind through electrostatic and hydrophobic interactions, hydrogen bonds, and polarization effects, or through chemical reactions and cross-linking agent employment [164]. Encapsulation by coacervation can be carried out using a single polymer, such as gelatin, which undergoes phase

separation in response to changes in temperature, pH, or electrolyte addition, forming a coacervate layer around the core. Alternatively, it can be employed with two or more oppositely charged coatings, typically proteins and polysaccharides, to form a coacervate phase encapsulating the active compound. The formation of a stable carrier with suitable loading and release properties is influenced by physicochemical conditions, such as pH, ionic strength, and polymer ratios [165,166]. Carrier systems obtained by coacervation display high *EE*, including hydrophobic compounds, desirable release behavior, and versatility for microencapsulation of several nutraceuticals [167]. One of the limitations of coacervation may be related to the instability and degradation of particles during drying and storage, which requires the employment of cryoprotectants to enhance the stability and dispersibility [168].

Microparticles formed by gelatin and gum Arabic, ranging from 35 to 80 µm, obtained by coacervation to anthocyanins from black raspberry extract encapsulation, resulted in higher stability of polyphenols after two months at 37 °C [166]. This methodology has also been used to microencapsulate quercetin in a gelatin–carboxymethylcellulose and curcumin in chitosan microparticles, conferring approximately 90% *EE* for quercetin and curcumin, maintaining 87% of curcumin antioxidant capacity against the DPPH radical [169,170].

4.4. Extrusion

Microencapsulation by extrusion involves the mixing of encapsulating materials with active compounds in a homogeneous solution, which is subsequently transferred to specialized extrusion equipment through a pointed nozzle with a specific diameter, in which the solution is dripped in a controlled manner. A solution containing the wall material and the active core passes through the nozzle or needle, forming a droplet of controlled shape and size that falls into a hardening solution, usually containing $CaCl_2$, where it then solidifies as microbeads. Calcium chloride enables ionic gelation by interacting with negatively charged encapsulating agents, forming cross-links that strengthen the microcapsule structure and enhance its stability. The process is fast, allowing for the production of 100–300 microcapsules per second, as determined by adjustments to the nozzle vibration. It is worth mentioning that this method has also been reproduced on a small laboratory scale through simple dripping using a syringe and needle [171–173]. Thus, extrusion is a simple technique to microencapsulate polyphenols into food matrices, forming small and spherical microparticles. However, this technique generates particles with high water content, which can compromise their stability and increase their susceptibility to microbial contamination. This limitation can be solved by drying the microparticles, extending their stability for up to 60 days at room temperature [171].

As stated with other microencapsulation techniques, the efficacy of extrusion depends on the choice of polysaccharide coating and adequate processing parameters for the preservation and enhancement of the functional properties [174,175].

The synthesis of hydrogel microparticles combining alginate to different polysaccharides, such as pectin, carrageenan, or chitosan, was found to be very efficient in maintaining the chemical stability of polyphenols and preserving the antioxidant capacity of chokeberry (*Aronia melanocarpa*) juice and fruit extracts. Microcapsules prepared with alginate and carrageenan retained 345 mg of polyphenols per 100 g of juice and 939 mg per 100 g in the extract-containing capsules. Under these conditions, only 37% and 24% decreases in phenolic content were observed, respectively. When alginate alone was used, about 42% of bioactive compounds were preserved, according to remaining antioxidant activity analyses. Microparticles prepared using alginate and carrageenan showed antioxidant activity superior to 97%, and using alginate and chitosan, reached around 70% of preserved antioxidant activity [176].

Anthocyanins from *Hibiscus sabdariffa* L. were encapsulated into pectin employing gelling combined with drip extrusion. The 788–1100 μm microparticles retained over 60% of anthocyanins and other antioxidants, even after 35 days of storage at low temperature [177]. Extrusion was also employed to encapsulate *Aronia melanocarpa* extracts into hydrogel microparticles prepared using sodium alginate combined with guar gum and chitosan. The microcapsules retained up to 158 mg/100 g of polyphenols and preserved 87% of storage stability after 28 days [175].

4.5. Emulsification

The emulsification method is based on a colloidal system composed of two immiscible hydrophobic and hydrophilic liquid phases stabilized by an interfacial layer composed of an emulsifier that elicits the interaction between phases, forming small droplets dispersed in a non-miscible continuous phase [178]. Oil-in-water (O/W) and water-in-oil (W/O) are the most commonly employed emulsions, although multiple emulsion systems, such as double emulsions formed by an internal aqueous phase (W) trapped as small drops within oil droplets (O), which are subsequently dispersed in another aqueous phase (W/O/W), can also be employed [179]. Droplet emulsions are classified as 'conventional' or macroemulsions when diameters are over 1 μm, while microemulsions range from 10 to 50 μm and droplets or nanoemulsions, between 20 nm and 500 nm [180].

Although emulsion systems designed for microencapsulation and bioactive delivery have demonstrated efficient performance in protecting bioactive compounds and controlling their release to specific sites, conferring stability and bioavailability, recent data have reinforced that emulsification for microencapsulating food bioactive compounds can be enhanced when associated with polysaccharide coatings that improve loaded compound stability and enhance their controlled release, favoring their absorption and potential bioactivity in the human body [181].

Polysaccharides, such as chitosan, carrageenan, starches, and alginate, play a key role in gastrointestinal modulation when included as emulsion-based microparticle components, as they act as structural stabilizers by coating microemulsions while, at the same time, improving digestibility and allowing for the controlled release of bioactive compounds, affecting the absorption and bioavailability of loaded nutrients [182].

Double-gelled emulsions (W/O/W) formed by a combination of chia mucilage with κ-carrageenan, locust bean gum, and commercial gum acacia plus xanthan blend have been employed for the encapsulation of green tea, demonstrating stable polyphenol compound content for 35 days by maintaining at least 75% of the initial antioxidant activity. The emulsions displayed static stability with no phase separation, and the κ-carrageenan, gum acacia, and xanthan blend formulation demonstrated the best results, promoting the controlled release of green tea during in vitro digestion, where about 80% of polyphenols were released under simulated intestinal physicochemical conditions [183].

Similarly, W1/O/W2 double emulsions have been employed to enhance the stability and bioavailability of tea polyphenols using a composite wall material of modified gluten proteins (MEG) and β-cyclodextrin (β-CD) at different ratios, with the best performance observed for MEG: β-CD (2:1) combined with 0.2% xanthan gum or gum Arabic. The emulsions were stabilized with 5% polyglycerol polyricinoleate, resulting in an *EE* of up to 94% and improved thermal stability and protection under simulated gastric conditions. Furthermore, this system enabled controlled release and achieved a bioavailability of about 60% [184].

Chitosan microspheres have been employed to deliver thyme (*Thymus serpyllum* L.) polyphenol compounds by emulsification followed by cross-linking with glutaraldehyde. Microparticles measuring 70–230 μm were obtained using 2% chitosan (*w*/*v*) and 0.4% gum

Arabic (*v*/*v*), enabling 67% polyphenol *EE*. The microemulsion protected the polyphenols, minimizing their degradation and prolonging their release by 3 h under simulated physicochemical conditions mimicking the gastrointestinal tract. The use of gum Arabic provides higher microsphere surface roughness, which may influence their stability and interaction with the gastrointestinal environment [185].

Betalains and polyphenols from cactus–pear extract have also been encapsulated in a W/O/W emulsion coated with 60% gum Arabic and 40% maltodextrin and used for yogurt enrichment, at 30% of the final mixture. The 20% polysaccharide-based emulsion conferred 22% total phenolic compound retention and 81.8% betalain retention after 36 days of storage. Flavonoids were some of the main identified compounds, although not all phenolics were retained. Furthermore, total polyphenols displayed solid stability, and up to 80% of the antioxidant capacity was maintained when exposed to a simulated gastrointestinal environment [186].

Microparticle production techniques considered herein have proven effective for the proposed applications, but the feasibility of producing these delivery systems at an industrial scale needs further exploration. Spray-drying may be one of the most suitable techniques for large-scale microencapsulation due to its continuous operation, high production capacity, and relatively low cost compared to other methods, such as freeze-drying. Although it can be detrimental to thermolabile compounds, the spray-drying technique is widely employed for food and pharmaceutical products due to its cost-effectiveness, which outweighs its limitations [152].

5. Polyphenols and Pigments in Polysaccharide Microparticles: Clinical Perspectives

As described here, the development of polysaccharides as bioactive carriers through microencapsulation is a growing technology. The main advantage of the polysaccharide is its biocompatibility, which reduces the risk of adverse reactions. It also exhibits proper permeability, facilitating the transport of bioactive molecules, and enables its large-scale use as a carrier due to its low cost. Polysaccharides have been employed in the development of nutraceutical products and have demonstrated relevant potential in the development of medical devices, drug delivery systems, and regenerative materials, promoting advances in biomedicine [187].

Polysaccharides can transport polyphenols, which exhibit antioxidant and anti-inflammatory properties that contribute to the prevention and treatment of chronic diseases, including cardiovascular and neurodegenerative conditions and certain types of cancer [188]. These compounds are nutraceuticals permitted for free marketing by health organizations. However, due to limited gastrointestinal stability and absorption, their properties can be improved through polymer encapsulation. In short, when ingested, the polyphenols are hydrolyzed and subjected to chemical modifications such as methylation, alkylation, sulfation, and glucuronidation in the small intestine and liver before entering the circulatory system. Unabsorbed polyphenols, in turn, reach the large intestine and colon, where they can be metabolized by the microbiome into distinct bioactive substances or eliminated in feces and urine [10]. In addition, the interaction of polyphenols with other nutrients, such as proteins, fiber, and minerals, as well as the covalent binding of esterified or conjugated polyphenols to matrix components, can reduce their bioaccessibility and bioavailability, circumventing potential health benefits. These challenges highlight the importance of encapsulation techniques to increasing the biological roles of bioactive compounds [123,189,190]. However, the number of in vitro, preclinical, and mainly clinical trials is low and should be conducted to evaluate the behavior of microencapsulated polyphenols in the gastrointestinal tract and their biological effects.

In a preclinical assay aimed at improving the stability and bioavailability of oral resveratrol, pectin–alginate microparticles synthesized by ionotropic gelation exhibited resistance to acidic pH, with a slight release of 10% resveratrol in the gastric phase, ensuring its delivery into the small intestine. Microencapsulated resveratrol was tested in mature 3T3-L1 adipocytes to evaluate its influence on triacylglycerol cell content. Both free and encapsulated resveratrol reduced triacylglycerol levels, indicating that stomach-resistant microparticles may represent a promising strategy for oral administration of resveratrol, enabling its use as a dietary supplement and functional food for the treatment of obesity. In addition to protecting resveratrol, gastro-resistant encapsulation favors its release in the distal intestinal tract, where absorption is optimized, thereby preserving its bioactivity on lipid regulation [65]. Resveratrol also exhibited post-digestion antioxidant activity in Caco-2 and HT29-MTX cells, with no cytotoxic effects when loaded onto hydroxymethylcellulose microparticles [191].

Preclinical experiments also show increased antiangiogenic effects of caffeic acid loaded in chitosan-β-cyclodextrin microparticles on the reduction in tubular structures formation in human endothelial cells after exposure to hydrogen peroxide (H_2O_2)-induced oxidative stress. Microencapsulated caffeic acid looks very promising in the treatment of chronic diseases due to the combination of antioxidant and antiangiogenic activities [192].

Chitosan microsponges incorporated into pectin tablets to deliver resveratrol prevent polyphenol degradation in the upper gastrointestinal tract, maximizing its bioavailability at the target site, showing a remarkable therapeutic effect in the treatment of acid-induced ulcerative colitis. In vivo evidence demonstrated a significant reduction in colonic lesions, necrosis, and inflammation when compared to tissues treated with free resveratrol. In addition, histological analyses demonstrated the preservation of mucosal architecture, reduced infiltration of inflammatory cells, and lower ulcer development, indicating that resveratrol loaded in chitosan–pectin microsponges confers cell integrity and promotes health in the colonic environment. This innovative microencapsulation strategy also shows promise in the treatment of inflammatory bowel diseases, offering controlled release and enhanced therapeutic efficacy [193].

Peanut skin extracts encapsulated in maltodextrin by spray-drying significantly reduced postprandial glucose spike in healthy human subjects, as observed in an oral glucose tolerance test. This effect is probably associated with the antioxidant properties of polyphenols and their potential interaction with glucose transport and metabolism. Although the exact mechanisms have not yet been elucidated, microencapsulation appears to preserve the bioactivity of the compound and improve its delivery. This strategy may offer promising applications for the dietary management of metabolic conditions such as diabetes while contributing to overall health promotion [194].

Studies show that the effects of encapsulation of bioactive compounds go beyond simple physical protection from the gastrointestinal environment, being strongly influenced by the interactions between polymers and active molecules. In pectin and alginate systems, selective release is mainly due to enzymatic degradation, favoring bioavailability in the colon. In hybrid associations that combine cyclodextrins and chitosan, the increase in biological activity is due to the complexation and modulation of the release profile, which maintains more stable concentrations and reduces the loss of the compound. Microspongy structures modulate kinetics in a way that depends on porosity and degradability, expanding the local therapeutic action. Maltodextrin microparticles, on the other hand, act mainly in the modulation of absorption time, without significantly altering bioavailability. Thus, it is understood that the choice of polymer is relevant for physiological performance, as it defines the quality of the interaction with the compound and, consequently, the biological effects obtained.

Although a large number of in vitro and preclinical trials have shown greater bioavailability and improved biological activity of bioactive compounds microencapsulated in polysaccharides, the confirmation of these findings through human ingestion is lacking, unable to confirm whether food polysaccharide microparticles actually can increase bioavailability and confer health effects on the human organism [195].

The biological effects of polyphenols from green tea loaded in chitosan microparticles were evaluated in a randomized and placebo-controlled study with 29 volunteers through topical application during 8 weeks. The microparticles conferred a significant improvement in skin elasticity and reduction in fine wrinkles, an effect attributed to the protection of catechins against degradation associated with the improvement in skin permeation through chitosan microencapsulation [196]. These preliminary findings reinforce the potential of polysaccharide microparticles as effective carriers of bioactive compounds intended for non-drug therapies and health maintenance.

Current research regarding the use of polysaccharides as encapsulating agents, including production techniques and the biological behavior of the resulting microparticles, is summarized in Table 1. However, future research investigating the clinical outcomes of both acute and chronic ingestion is essential to address the existing knowledge gap in microencapsulated bioactive delivery systems.

Table 1. Edible polysaccharides for polyphenols and pigments delivery, and the improvement of bioactive physicochemical characteristics.

Encapsulating Polysaccharide	Loaded Polyphenols and Pigments	Chemical or Physical Methods	Particle Size (μm)	Encapsulation Efficiency (%)	Improvements on Bioactive Compound	Study
Starch	Carotenoids	Gelatinization and freeze-drying	63	75	- 98% water solubility - Maintenance of antioxidant capacity at 30 °C and under light with a half-life of 37 days	Rosário et al. [24]
	Curcumin/ Resveratrol	ND	ND	80/88	- 10% release of curcumin in SGD for 2 h - 86% release of resveratrol in SID for 10 h - 60% of sustained release of antioxidants in SGD and SID for 24 h	Wahab & Janaswamy [26]
	Anthocyanins	Spray-drying	ND	ND	- 16% release of anthocyanins in SGD and SID for 1 h and 12% in intestinal fluid at 2 h	Rosales-Chimal et al. [28]
Starch + β-cyclodextrin	Phenolic compounds from pomegranate (*Punica granatum* L.)	Emulsification and spray-drying	10.16	61	- 49% release of bioactive compounds for 180 min in SID	Cortez-Trejo et al. [27]
Starch + Inulin + Maltodextrin	Anthocyanins	Spray-drying	10	67	- Greater antioxidant power and stability for 38 days under 50 °C and light exposure	Lacerda et al. [25]
Maltodextrin	Bixin	Emulsification and spray-drying	ND	86	- 97% water solubility - 60 days of storage stability at 4°C and 28 °C - >80% of heat stability at 160 °C for 15 min	Shridar et al. [34]
	Betalains/phenolic acid/catechin/ epicatechin/ myricetin	Freeze-drying	ND	88	- Higher encapsulation of phenolic acids (44 μg/g)	Mkhari et al. [35]
	Anthocyanins	Spray-drying and freeze-drying	23.14	98	- 96% water solubility - High antioxidant activity (329 mmol TE/g)	Murali et al. [36]
	Phenolic compounds of stevia extract (*Stevia rebaudiana*)	Emulsification and spray-drying	ND	87	- 94% water solubility - Enhanced stability - Stability of phenolic compounds in SGD after 60 min	Zorzenon et al. [37]
Maltodextrin + Gum Arabic	Polyphenols	Spray-drying	27	83	- High retention of total polyphenols and anthocyanins - >97% water solubility	Kuck et al. [162]

Table 1. *Cont.*

Encapsulating Polysaccharide	Loaded Polyphenols and Pigments	Chemical or Physical Methods	Particle Size (μm)	Encapsulation Efficiency (%)	Improvements on Bioactive Compound	Study
Inulin	Hydroxytyrosol and oleuropein from olive leaf extract	Spray-drying	ND	80 of OH-droxytyrosol 79 of oleuropein	- Facilitated colonic delivery (60% release) - Enhanced oleuropein retention after baking (≈77%) and boiling (≈63%) - Improved protection in SID	Duque-Soto et al. [74]
	Betacyanins from beetroot (*Beta vulgaris* L.) extract	Spray-drying	ND	58	- Improved color stability in sorbet for 150 days at 18 °C - Partial protection of betacyanin in aqueous media - Increased half-life at 4 °C	Omae et al. [78]
	Oleuropein from olive leaf (*Olea europaea*) extract	Spray-drying	10	79	- Retention of oleuropein during baking and boiling - Reduced release in the small intestine (27.5%) indicates delayed bioaccessibility and enhanced oleuropein delivery to the colon - Enhanced stability in intestinal fluid	Pacheco et al. [75]
	Black currant berries (*Ribes nigrum* L.)	Spray-drying	ND	ND	- Increased storage stability of anthocyanins and polyphenols for 12 months at 8 °C and 25 °C - Higher antioxidant retention with maltodextrin DE11	Bakowska-Barczak & Kolodziejczyk [77]
	Betalains from beetroot (*Beta vulgaris* L.) extract	Freeze-drying	ND	ND	- Improved water solubility - Reduced hygroscopicity - Promote thermal stabilization - Preservation of antioxidant activity and polyphenols during storage	Flores-Mancha et al. [80]
	Anthocyanins from chokeberry (*Aronia berries*) extract	Spray-drying	17	88	- 90% water solubility - Reduced anthocyanin degradation during storage - Improved stability under light and oxygen	Pieczykolan & Kurek [197]
Inulin or Acetylated Inulin	Gallic acid	Spray-drying	ND	83 inulin 75 acetylated inulin	- Acetylated inulin reduced burst release effect and slowed gallic acid release, non-Fickian (anomalous) diffusion behavior	Robert et al. [198]
Inulin + Modified Starch	Oregano (*Origanum vulgare* L.) extract	Emulsification and spray-drying	151	66	- Thymol retention (84%) - High entrapment efficiency (92%) - Improved thermal stability up to 220 °C	Zabot et al. [76]
Inulin + Maltodextrin	Anthocyanin from mangosteen (*Garcinia mangostana*)	Spray-drying	13	81	- High thermal stability (<200 °C mass loss) - 80% anthocyanin retention in 120 min at 70 °C - Increased stability and shelf-life when incorporated into yogurt	Sakulnarmra et al. [84]
Inulin + Sodium Alginate	Betacyanins (*Bougainvillea glabra*) bracts	External ionic gelation and extrusion	4.3 mm (Feret's diameter)	89	- Increasing inulin content from 10% to 20% enhanced the rheological properties of dispersions - Spherical shape - Increased mechanical resistance - Thermal stability up to 200 °C and antioxidant protection	Azevedo & Noreña [81]
Inulin or gum Arabic + Maltodextrin	β-carotene extracted from mango peel (*Mangifera indica*)	Spray-drying	ND	87	- Enhanced β-carotene stability (>68% recovery after SGI) - 2-fold increase in bioaccessibility with inulin addition - Improved cellular uptake of β-carotene in Caco-2 cells	Cabezas-Terán et al. [85]
Inulin + Maltodextrin + WPI	Betalains from beetroot (*Beta vulgaris* L.) extract	Spray-drying	ND	88–95	- Increased antioxidant activity - Enhanced thermal stability (~200 °C)	Carmo et al. [79]
Inulin + Maltodextrin + gum Arabic	Anthocyanins from Roselle (*Hibiscus sabdariffa* L.)	Spray-drying and freeze-drying	ND	92 by spray-dried, 95 by freeze-dried	- Higher retention of anthocyanins and antioxidant activities with MD/GA - >94% solubility in all delivery systems	Nguyen et al. [83]
Inulin + Maltodextrin + gum Arabic	Lycopene from tomato (*Lycopersicon esculentum*) extract	Spray-drying	ND	ND	- Improved lycopene stability under storage - Enhanced intestinal delivery >50% bioaccessibility when incorporated into yogurt	Corrêa-Filho et al. [86]
Alginate	Catechins	Ionic gelation, emulsification, and extrusion	131	42	- 63% sustained catechins release after 2 h in a pH 7 solution	Kim et al. [49]
Alginate + Carrageenan	Phenolic compounds	Extrusion	ND	97	- Retention of 939 mg of phenolic compounds per 100 g in microcapsules containing phenolic extract - High antioxidant capacities after one month of storage at 4 °C	Stach & Kolniak-Ostek [176]

Table 1. *Cont.*

Encapsulating Polysaccharide	Loaded Polyphenols and Pigments	Chemical or Physical Methods	Particle Size (μm)	Encapsulation Efficiency (%)	Improvements on Bioactive Compound	Study
Alginate + Pectin	Resveratrol	Ionic gelation by atomization in a peristaltic pump	1.4	41	- >90% of stability for 2 h in pH 1.2 solution; - 70% of sustained release for 24 h in tri-sodium phosphate at pH 7.4	Gartziandia et al. [65]
Alginate + Chitosan	Black locust flower extract	Ionic gelation and extrusion	228	92.5	- 60% sustained antioxidants release for 24 h in gastric and intestinal fluids - Increased antioxidant activity in SGD and SID	Boškov et al. [46]
	Phenolic acids from orange peel	Ionic interaction, ionic gelation, and extrusion	252	89	- 82% of sustained antioxidants release for 24 h and increased antioxidant activity in SGD and SID	Savic et al. [45]
	Quercetin	Electrostatic complexation and extrusion	ND	86	- Intact microparticle structure after 8 h in a pH 1.2 solution - Sustained release for 24 h in a pH 7.4 solution	Frent et al. [51]
	Quercetin	Emulsion-assisted external ionic gelation and freeze-drying	25–79	53 (with the combination of inulin and chitosan)	- Retention of 80% quercetin after SID - Delayed release during colonic fermentation - Increased structural integrity of microspheres	Liu et al. [73]
Chitosan	Polyphenols from *Thymus serpyllum* L. extract	Emulsion and cross-linking oven processing at 50 °C	68–230	ND	- pH-responsive prolonged release - Higher release in SGD and SID vs. water - Swelling-controlled release - Maintenance of antioxidant activity - Increased matrix crystallinity	Trifkovic et al. [107]
	Anthocyanin from blueberry (*Vaccinium caesariense*) extract	Ionic gelation and spray-drying	0.06	94	- High anthocyanin recovery - Improved pH stability (especially at pH 7.4) - Reduced release at neutral pH	Wang et al. [108]
Chitosan + Inulin + WPI	Anthocyanins from black currant (*Ribes nigrum*) and Lactic acid bacteria (*Lactobacillus casei* 431®)	Spray-drying	<5 (small spheroSomes) ≈20–337 (large coaserVates)	95.46	- Stability in gastric fluid and sustained release in intestinal fluid - Stability over 90 days at 4 °C - Antioxidant and antidiabetic activity preservation	Enache et al. [106]
Gum Arabic	*Ruellia tuberosa* L. and *Tithonia* diversifolia leaves powder	Spray-drying	145	84	- Alpha-amylase inhibition (IC_{50}: 54 μg/mL) - Increased antioxidant activity (DPPH)	Almayda et al. [114]
Gum Arabic + WP	Flavonoids from ponkan peel (*Citrus reticulata Blanco*)	gum Arabic emulsion in soybean oil and WP in water Spray-drying	1.42	97	- ABTS·⁺ and DPPH inhibition	Hu et al. [115]
Gum Arabic + Coconut (*Cocos Nucifera L.*) + milk Whey	Curcumin	Spray-drying	<22	94.5	- Over 88% water solubility - 72 days of storage stability at 50 °C (15%)	Adsare & Annapure [116]
Gum Arabic + Maltodextrin	Anthocyanins from barberry (*Berberis vulgaris*) extract	Spray-drying	ND	96	- 91% water solubility	Mahdavi et al. [117]
	Flavonoids	Freeze-drying	7.2	95	- Retention of phenolic and flavonoid for one month of storage under refrigeration and room temperature - >89% water solubility	Yeasmen & Orsat [149]
Gum Arabic + Inulin	Buriti (*Mauritia flexuosa*) oil	Freeze-drying	7.5	93	- 92% β-carotene retention - 72% water solubility	de Oliveira et al. [118]
	Geranylgeraniol	Freeze-drying	1.1	96	- 96% of antioxidant activity stability maintenance (ORAC and DPPH•)	Silva et al. [148]
Gum Arabic + Gelatin	Anthocyanins	Freeze-drying	63	ND	- 48–70% of anthocyanins retention after 60 days at 7 °C	Shaddel et al. [166]
Gum Arabic + Chitosan	Polyphenols	Emulsification	ND	67	- Reduced polyphenols degradation and 3 h prolonged release under simulated digestion	Trifkovic et al. [185]
Pectin	Resveratrol	Ionic interaction and cross-linking of pectin-glutaraldehyde by extrusion	902	98	- 93% of stability for 5 h in SGD and SID - >90% of storage stability at 4°, 25° and 40 °C for 180 days - 5 h increment to reach the plasma of Sprague–Dawley rats	Das et al. [64]
Pectin + Alginate	Gallic acid	Ionic gelation and air-drying	1.3	89	- 15–35% of sustained cumulative release in pH 4 of HCl solution	Nájera-Martinez et al. [67]

ND—not determined, $CaCl_2$—calcium chloride, D_h—hydrodynamic diameter, DPPH—2,2-diphenyl-1-picrylhydrazyl, HCl—hydrochloric acid, IC_{50}—half-maximal inhibitory concentration, Zn^{2+}—zinc ion, LC—loading capacity, SGD—simulated gastric digestion, SID—simulated intestinal digestion, TE—Trolox equivalents, WP—whey protein, WPI—whey protein isolate.

6. Conclusions

Polysaccharides are a promising edible coating for polyphenols and pigments, mainly due to their non-toxicity and low cost. The current literature indicates that polysaccharide–polysaccharide matrices and polysaccharide–bioactive compound interactions primarily occur through hydrogen bridges and electrostatic physical interactions, which influence the *EE*, the physicochemical, and bioactive properties of the loaded compounds. In general, the content of polyphenols or pigments microencapsulated in polysaccharides is stable during the gastrointestinal route, as evidenced in in vitro conditions and in cell culture models. Regarding microparticle production, spray-drying appears to be more advantageous due to its practicality and cost-effectiveness, while freeze-drying is more suitable for preserving heat-sensitive compounds, albeit with its high-energy and time-consuming processing.

Chemical techniques offer greater control over particle structure, particularly the emulsification combined with polymers like chitosan and alginate, which reduces digestibility and enables the controlled release of bioactive compounds. Investing in hybrid techniques may be an interesting and promising direction, although more experimental support is needed. Most studies focus on *EE*, release, and bioaccessibility, but few have addressed issues such as in vivo bioavailability and pharmacological effects. Compiling and reproducing this knowledge and process on an industrial scale remains challenging, primarily due to encapsulation protocols that employ multiple coatings or require several chemical reaction steps. In this sense, it seems preferable to explore polysaccharide combinations that exhibit better solubility in aqueous media, such as modified starches and maltodextrins, which do not require the use of chemical agents for solubilization during microparticle synthesis.

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