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Natural Hydrocolloids as Wall Materials for Microencapsulation of Bioactive Compounds in Food Systems: A Critical Review

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Abstract: ***Bioactive food ingredients rarely survive intact the journey from formulation to absorption. Polyphenol oxidase degrades phenols, vitamins photodegrade, probiotic cells lose viability in the stomach and mineral salts react with matrix components long before they reach the small intestine. Microencapsulation in natural hydrocolloids is the food industry's principal answer to this problem, and the literature on it has grown quickly. That growth has not been matched by consistency. This review examines six established wall materials (Sodium Alginate, Pectin, Chitosan, Gum Arabic, Carrageenan, and Guar gum) together with five that remain underused (Xanthan gum, Locust Bean gum, Gellan gum, Konjac Glucomannan, and Tamarind seed polysaccharide), and evaluates the five techniques most commonly paired with them. Rather than treating materials and processes as separate subjects, a single causal chain was followed: how wall chemistry sets gel mesh size, how mesh size and core charge together determine what can actually be retained, and how the same structural features govern release in the gut. Two challenges recur throughout. Encapsulation efficiency is reported by at least two incompatible calculations, making cross-study comparisons unreliable and pooled estimates meaningless. Moreover, small cationic species, iron and zinc above all, are systematically poorly retained in anionic gels; some of the low values reported for them are analytical artifacts rather than real losses, because an intact alginate matrix shields the ions from colorimetric reagents.***

Keywords: Microencapsulation, natural hydrocolloids, sodium alginate, ionic gelation; bioactive compounds

1. Introduction

Functional foods- defined broadly as foods that deliver demonstrable health benefits beyond basic nutrition- have expanded considerably in both research output and commercial relevance over the past two decades. The scientific rationale for incorporating bioactive compounds such as polyphenols, carotenoids, vitamins, probiotics, polyunsaturated fatty acids, and essential minerals into food matrices is well established. However, translating this rationale into stable, bioavailable, and organoleptically acceptable products is considerably more difficult than laboratory efficacy data alone would suggest.

The problem is that most biologically active compounds are inherently unstable under the conditions of food processing and storage. Polyphenols oxidise; vitamins decay under heat and light; minerals interact with food matrix components to form insoluble complexes of poor absorbability; probiotics lose viability during storage and gastric transit; omega-3 fatty acids undergo rancidity within days upon exposure to air. Each of these compounds, added to a food product without protection, may suffer a significant amount of its bioactivity before it gets to the human body.

Microencapsulation addresses this problem by enclosing the active compound within a protective shell- the wall material- which physically separates the core from the external environment. The first microencapsulation patent was granted in the United States in 1954 for a carbonless copying paper application [1]. What has changed dramatically since then is the range of materials considered food-safe and the sophistication of techniques available. Natural hydrocolloids have emerged as the wall materials of choice for food applications because they are biodegradable, non-toxic, widely available, compatible with food-grade processing conditions, and, in many cases, have established regulatory approval as food additives.

The global microencapsulation market was valued at approximately US Dollars 9.8 billion in 2023 and is expected to cross US Dollars 17 billion by 2030 owing to the growth of functional food, nutraceutical, and pharmaceutical industries [2]. Despite this growth, several fundamental challenges remain inadequately resolved. Among these, the retention efficiency of small, water-soluble, or ionically charged core materials within hydrogel matrices- including mineral micronutrients critical for addressing nutritional deficiencies in populations across South Asia- presents a specific challenge that has been insufficiently addressed in review literature.

This review is organized to discuss (i) the origin, composition and functional properties of traditional and emerging natural hydrocolloid wall materials, (ii) the criteria for suitable wall material selection, (iii) main microencapsulation techniques applicable to food systems, (iv) main determinants of encapsulation efficiency, (v) in vitro release behavior, (vi) reported applications in functional food products, (vii) characterization methods, and (viii) challenges and future research directions. Only food-grade natural hydrocolloids are considered and not synthetic polymer systems.

2. Review Methodology

The present review was developed from a systematic search of peer-reviewed literature available in the databases of Scopus, Web of Science, PubMed, Science Direct and Google Scholar. The main search was for the period January 2010 to June 2025, with a particular focus on studies published between 2018 and 2025. Foundational references from before 2010 were retained where they described structural mechanisms or physicochemical properties that remain authoritative.

Boolean search queries were constructed using the following primary keywords and combinations: "microencapsulation", "natural hydrocolloids", "food-grade encapsulation", "alginate", "pectin", "chitosan", "gum Arabic", "carrageenan", "guar gum", "xanthan gum", "konjac glucomannan", "gellan gum", "tamarind seed polysaccharide", "encapsulation efficiency", "ionic gelation", "spray drying", "controlled release", "bioactive compounds", "functional food". Specific search terms were additionally applied for application-focused sections: "probiotic encapsulation", "polyphenol stability", "mineral encapsulation", "iron bioavailability".

Inclusion criteria were: (i) peer-reviewed English-language articles; (ii) studies involving food-grade natural polysaccharides or gums as primary wall materials; (iii) articles reporting quantitative encapsulation efficiency, morphological characterization, or bioavailability data; (iv) review and original research articles focused on food, nutraceutical, or food-biomedical interface applications. Studies exclusively involving synthetic polymers, non-food-grade materials, or clinical pharmacological trials were excluded. Titles and abstracts were independently screened, followed by full-text review of relevant papers

3. Natural Hydrocolloids: Classification, Sources, and Physicochemical Properties

3.1 Sodium Alginate

Sodium alginate is derived from the cell wall of brown seaweeds (mainly *Laminaria hyperborea, Macrocystis pyrifera* and *Ascophyllum nodosum*) and commercially extracted by alkaline treatment and precipitation as the sodium salt [3]. Chemically it is a linear polysaccharide made up of 1,4-linked beta-D-mannuronic acid (M) and alpha-L-guluronic acid (G) residues arranged in blocks, i.e. MM blocks, GG blocks and alternating MG blocks. The ratio of M to G units (M/G ratio) is important: high-G alginates form stiffer, more brittle gels when crosslinked with divalent cations, whereas high-M alginates form softer, more elastic gels. Commercial sodium alginates generally have M/G ratios in the range 0.5-1.5 [4] Fig 1& 2.The most important property of sodium alginate for microencapsulation is the rapid gelation property in contact with divalent cations, especially Ca2+. This ionic crosslinking- the egg-box model- involves Ca2+ binding in the cavities formed by GG blocks [5]. This gelation takes place at room temperature without heat treatment and organic solvents and is suitable for heat-labile bioactives. Sodium alginate has been declared as “generally regarded as safe” (GRAS) by the US FDA and approved by FSSAI as a thickener and stabilizer (INS 401).

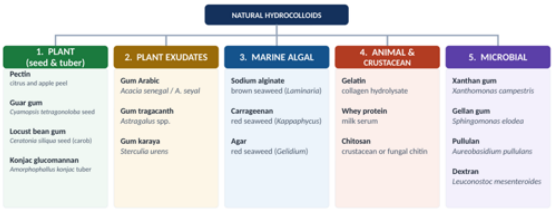

Figure 1. Classification of principal natural hydrocolloids used as wall materials in food-grade microencapsulation systems, organized by biological source. FSSAI = Food Safety and Standards Authority of India; INS = International Numbering System for Food Additives [32].

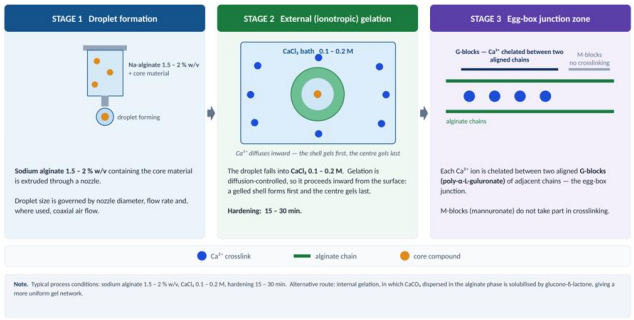

Fig. 2 Ionic gelation of sodium alginate and the egg-box junction zone. Stage 1: sodium alginate at 1.5–2% w/v containing the core material is extruded through a nozzle, droplet size being governed by nozzle diameter, flow rate and, where used, coaxial air flow. Stage 2: droplets fall into a $CaCl_2$ bath at 0.1–0.2 M; gelation is diffusion-controlled and proceeds inward from the droplet surface, so a gelled shell forms first and the center gels last, with hardening over 15–30 min. Stage 3: each Ca^{2+} ion is chelated between two aligned G-blocks (poly-α-L-guluronate) of adjacent chains, forming the egg-box junction; M-blocks (mannuronate) do not participate in crosslinking. Intact alginate beads shield mineral ions from colorimetric reagents, and complete acid digestion of the bead matrix is required before iron estimation [4,5].

A critical but frequently underreported limitation concerns the selective retention of different core materials. The calcium-alginate gel network acquires a net negative surface charge at neutral pH and retains large, hydrophobic, or negatively associated macromolecules effectively. However, small, positively charged, or freely diffusing ionic species- including ferrous and ferric iron ions- exhibit higher diffusivity through the gel network and tend to leach during bead formation. This selective retention behavior is a consequence of the gel mesh structure, the average pore size of which has been estimated at 5–200 nm depending on alginate concentration and M/G ratio [6].

3.2 Pectin

Pectin is a structural polysaccharide found in the primary walls of cell structures in plants. The commercial source of pectin is mainly citrus peel and apple pomace, by-products of juice processing. The apple pomace available from cold-press juice industry in Himachal Pradesh, India is an easily available and economically viable raw material for pectin extraction with less dependence on imports. Its backbone consists of 1,4-linked alpha-D-galacturonic acid units, a proportion of which are methyl-esterified. High methoxyl pectin (HM pectin, DE > 50%) forms gels through hydrophobic interactions and

hydrogen bonding at low pH and high sugar concentrations, whereas low methoxyl pectin (LM pectin, DE < 50%) gels via Ca2+ crosslinking analogous to alginate but at lower cation concentrations [7].Composite pectin-alginate beads consistently outperform single-hydrocolloid systems in encapsulation efficiency for both polyphenolic and hydrophilic core materials, apparently because the two polymers form a denser interpenetrating network [8]. The slower gelation kinetics of LM pectin relative to alginate in CaCl2 can reduce EE% for small diffusible molecules unless the crosslinking step is carefully timed.

3.3 Chitosan

Among the common natural hydrocolloids, chitosan is special by being positively charged on the surface under acidic condition. It is produced by deacetylating chitin from the exoskeletons of crustaceans. The cationic charge density is regulated by its degree of deacetylation (DD). Chitosan is soluble in dilute organic acids and gels in the presence of polyanions like tripolyphosphate [9]. Chitosan is particularly useful in microencapsulation as a secondary coating on negatively charged alginate or pectin beads. This electrostatic bilayer coating reduces the surface porosity and postpones the leaking in acidic conditions, significantly enhancing the probiotic viability during gastrointestinal transit [10]. Its intrinsic antibacterial activity may be advantageous in food preservation applications. The direct application of chitosan in encapsulated food ingredients is limited for the Indian market as FSSAI does not specifically list it as a food additive in India.

3.4 Gum Arabic

Gum Arabic (GA) is an of *Acacia senegal* and *Acacia seyal* trees and is the food encapsulation hydrocolloid with the longest known commercial history. Its complex arabinogalactan structure with protein containing units makes it extraordinary surface active and emulsifying properties and it is outstanding for encapsulation of lipophilic compounds [11]. Gum Arabic gels by film formation and emulsification, not ionic crosslinking, and is stable over a wide pH range (3–10) and to heat and salt. In addition to its basic film forming properties, gum arabic can also form Maillard conjugates with proteins under controlled dry heating conditions. Heating with whey protein isolate or casein leads to the generation of glycoprotein conjugates with substantially improved emulsification capacity and oxidative stability due to the formation of covalent bonds between the reducing sugars of the arabinogalactan part and the free amino groups of the protein [12]. These conjugates are particularly useful as wall materials for encapsulation of highly unsaturated lipids such as fish oil and evening primrose oil where protection from lipid oxidation is crucial. The most popular industrial encapsulation method is spray drying, with gum Arabic being the preferred material for the majority of commercial flavor encapsulation. The protection mechanism is ascribed to the formation of a continuous glassy carbohydrate matrix around the core in the fast-drying process. The low moisture content and high glass transition temperature effectively immobilize the encapsulated compound and limit the diffusion of oxygen. The primary commercial constraint is the uncertainty in supply chains due to dependence on sub-Saharan African Acacia species. Indian Acacia species with functional equivalence could be potential domestic substitutes and need to be investigated.

3.5 Carrageenan

Carrageenan is extracted from red seaweeds and exists in three principal forms that differ in sulfation pattern and functional behavior. Kappa-carrageenan (one sulfate per disaccharide) forms firm, brittle gels with potassium ions and strong gels with proteins. Iota-carrageenan (two sulfate groups) forms softer, elastic gels with calcium ions that resist syneresis-making it preferable where freeze-thaw stability of the encapsulation matrix is required. Lambda-carrageenan (three sulfate groups) does not gel under normal conditions but acts as a thickening agent [13].

The synergistic interaction between kappa-carrageenan and Locust Bean Gum (LBG) is well characterized: LBG reduces gel brittleness and eliminates syneresis, producing a stable elastic composite gel with superior mechanical integrity for encapsulated bead applications [14] Table 1

Table 1: Comparison of physicochemical and functional properties of the three commercial carrageenan types

Property	κ (Kappa)	ι (Iota)	λ (Lambda)
Sulfate groups per disaccharide	1	2	3 [15]
Gelling cation	K^+	Ca^{2+}	Non-gelling [16]
Gel texture	Firm, brittle	Soft, elastic	Thickener only [17]
Syneresis tendency	High	Low	None [18]
Freeze–thaw stability	Poor	Good	N/A [18]

3.6 Guar Gum

Guar gum is derived from the endosperm of *Cyamopsis tetragonoloba*, a drought-tolerant legume primarily cultivated in Rajasthan and Gujarat, India. India accounts for approximately 80-85% of global guar gum production, making it the most cost-accessible hydrocolloid for Indian functional food applications. Guar gum is approved by FSSAI as a thickening agent (INS 412) without quantity restrictions in several food categories.

In encapsulation, guar gum alone provides viscosity control but limited structural integrity. However, blending with xanthan gum produces synergistic interactions analogous to the locust bean gum-carrageenan pair, generating highly viscous shear-thinning gels suitable for extrusion-based encapsulation systems. Guar gum has been investigated as a spray-drying wall material in combination with maltodextrin, and as a matrix for oral drug delivery systems exploiting its susceptibility to enzymatic hydrolysis by colonic microflora.

3.7 Emerging and Underutilized Hydrocolloids

Besides the six established hydrocolloids, several other natural polymers have been increasingly studied due to their unique functional properties, local availability in South and Southeast Asia, or sustainability credentials. Xanthan gum is obtained by fermentation of *Xanthomonas campestris* and has excellent thermal stability, acid tolerance and salt compatibility over a wide concentration range, which makes

it suitable for encapsulation systems in acidic food matrices such as dairy beverages and carbonated drinks. Its potential to form gels on its own is limited but it is important in combination with galactomannans such as guar gum. Xanthan is approved by the FSSAI (INS 415). Locust bean gum (LBG) is obtained from the endosperm of *Ceratonia siliqua.* It has a ratio of mannose to galactose of about 4:1 (compared with 2:1 in guar gum). This gives it lower solubility but stronger synergistic gel formation with xanthan and kappa-carrageenan. Encapsulation of iron and probiotics using LBG-kappa-carrageenan composite matrices showed reduced syneresis and enhanced bead integrity [20].Gellan gum is a linear anionic polysaccharide produced by *Sphingomonas elodea* which forms thermally reversible gels at concentrations as low as 0.1% w/v . Low-acyl gellan forms a firm, brittle gel like agar, whereas high-acyl gellan forms a soft, elastic gel. Its thermal stability and transparency make it attractive for functional beverage applications and for encapsulation of heat processed ingredients [21]. Konjac glucomannan (KGM) is a high molecular weight polysaccharide (200,000–2,000,000 Da) with excellent water-holding capacity and viscosity extracted from the tubers of Amorphophallus konjac. The KGM-alginate composite matrices have been reported to be superior wall systems for probiotic encapsulation due to the dense network formed by interchain hydrogen bonding between the two polysaccharides [22].

Tamarind seed polysaccharide (TSP), a xyloglucan obtained from the seed kernels of Tamarindus indica, is of particular interest to India as the tamarind is widely cultivated in southern and central India and the extraction of TSP from seed kernels is both a sustainable raw material source and a value addition route from an abundantly wasted agricultural by-product. The encapsulation of polyphenols from Indian spices and traditional herbal preparations using TSP has been investigated with promising stability data . This is of particular interest to Indian functional food manufacturers due to its availability, low cost and utilization in traditional foods Table 2.

Table 2: Comparative overview of principal natural hydrocolloids used as wall materials in food-grade microencapsulation systems

Hydrocolloid	Source	Gelation Mechanism	Surface charge	Typical EE%	Primary Application
Sodium Alginate	Brown seaweed	Ionic (Ca^{2+})	Negative	60–90%	Polyphenols, probiotics, minerals [23]
Pectin (LM)	Citrus peel, apple pomace	Ionic (Ca^{2+}) / H-bonding	Negative	55–85%	Polyphenols, anthocyanins [24]
Chitosan	Crustacean chitin	Ionotropic / pH	Positive (acidic pH)	70–95% (coating)	Probiotics, bilayer coating [25]
Gum Arabic	Acacia exudate	Film formation	Slightly negative	80–95%	Flavors, lipophilic compounds [26]
Carrageenan (κ)	Red seaweed	Ionic (K^+)	Negative	65–88%	Probiotics, polyphenols []27]
Guar Gum	Cyamopsis seed	Matrix viscosity	Neutral	50–75%	Controlled-release matrices [28]
Xanthan gum	Xanthomonas fermentation	Synergistic (+ galactomannan)	Negative	55–80%	Acidic food matrices [29]
Konjac glucomannan	Amorphophallus tubers	H-bonding / alkaline	Neutral	65–88%	Composite matrices, probiotics [30]
Gellan gum	Sphingomonas fermentation	Ionic (Na^+/Ca^{2+})	Negative	60–85%	Beverages, heat-processed systems [31]
Tamarind seed PSP	Tamarindus indica seed	Hydrogen bonding	Slightly negative	55–78%	Indian bioactives, spice extracts [32]

4. Criteria for Selection of Wall Materials

The selection of an appropriate hydrocolloid wall material for a given microencapsulation application involves a simultaneous trade-off among a number of physicochemical, regulatory, economic, and functional criteria. There is no one material that is universally optimal, and the practical literature provides many examples where seemingly straightforward material choices have resulted in unexpected performance shortfalls due to incompatibility between properties of the wall material and those of the core compound. The wall solution processability is directly determined by molecular weight and viscosity. Very high molecular weight hydrocolloids can provide excellent encapsulation at low concentrations but may pose challenges in atomization (during spray drying or extrusion) due to the high viscosity of the solution. For successful atomization during spray drying, wall material solutions with viscosities of less than about 300 mPas at the feed temperature are required [33].

The hydrocolloid's surface charge is particularly important in ionic gelation systems. Polyanions with negative charge (alginate, pectin, carrageenan) gel with divalent cations and are compatible as primary beads with positively charged chitosan as a secondary coating. Electrostatic interaction between charge-complementary hydrocolloids in bilayer bead systems yields a wall that is denser and less permeable than that formed by either polymer alone. For core compounds that are also ionically charged, compatibility between core and wall charge needs to be assessed - positively charged core molecules may interact unfavourably with anionic wall matrices and experience excessive leakage. It is a mandatory requirement for food applications seeking regulatory approval status.

In India, the FSSAI has approved major hydrocolloids such as alginate (INS 401), pectin (INS 440), gum Arabic (INS 414), guar gum (INS 412), carrageenan (INS 407) and xanthan gum (INS 415) for use as food additives under the Food Safety and Standards (Food Products Standards and Food Additives) Regulations 2011 [34]. As FSSAI does not explicitly mention chitosan and konjac glucomannan as food additives, their direct use in encapsulated food products in Indian market is restricted. But both are utilized in the nutraceutical and supplement categories.

5. Microencapsulation Techniques

5.1 Ionic Gelation

Ionic gelation- also termed ionotropic gelation or extrusion encapsulation- is the most widely employed laboratory-scale technique for producing natural hydrocolloid beads, because it requires no organic solvent, involves no heating step, and is operationally simple. In external ionic gelation, sodium alginate solution (typically 1–3% w/v) containing the core material is extruded dropwise through a needle or nozzle into a CaCl2 bath (0.5–5% w/v). Droplets solidify immediately on contact as calcium ions diffuse inward, creating a progressively stiffening gel shell. In internal ionic gelation, an insoluble $CaCO_3$ is incorporated into the alginate solution and activated by pH reduction with glucono-delta-lactone, distributing gelation more uniformly [35].

Encapsulation efficiency by ionic gelation is highly variable. Santos et al. [35] reported EE% of 61.6–71.0% for annatto extract. Internal gelation led to a more compact internal structure. Singh et al., 2018 in their study on alginate-pectin beads for papaya leaf extract, showed EE% in the range of 32.5–85.6% among the trials of optimisation, with syringe pump flow rate being the most important factor.

A practically important limitation of external ionic gelation for mineral encapsulation deserves explicit attention. Small ionic compounds, such as ferrous sulphate (Fe2+, MW 152 Da), are positively charged and highly mobile in aqueous environments. Two problems arise simultaneously: first, small ions diffuse out of the forming bead into the aqueous CaCl2 bath before the gel network fully consolidates; second, when beads are subsequently analyzed by colorimetric methods without prior acid digestion to dissolve the alginate matrix, the encapsulated iron registers as absent or near-zero — an analytical artifact rather than a true reflection of iron loss [35] Fig 3. The correct analytical approach requires complete dissolution of the alginate matrix in a mineral acid (HCl or HNO3) at low pH before iron estimation.

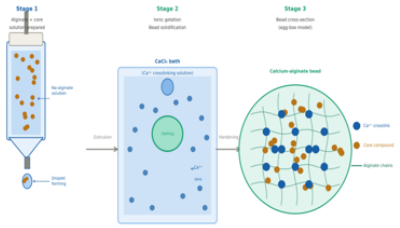

Figure 3. Schematic representation of the ionic gelation process for encapsulation of bioactive compounds using sodium alginate. Stage 1: Na-alginate solution containing core material is extruded via syringe nozzle. Stage 2: Droplet enters CaCl2 bath; Ca^{2+} ions crosslink GG alginate blocks via the egg-box mechanism. Stage 3: Formed bead cross-section showing Ca^{2+} crosslinks (blue), alginate chains (green), and encapsulated core molecules (orange). Key analytical note: intact alginate beads shield mineral ions from colorimetric reagents; full acid digestion is required before iron estimation Lee and Mooney (2012).

5.2 Spray Drying

Spray drying is the most widely used industrial technique for microencapsulation of bioactives because of its scalability, rapidity and continuous operation. The hydrocolloid-core solution is sprayed into a hot-air chamber, where water evaporates in seconds and the droplets become dry powder particles. The process is particularly suitable for hydrophilic wall materials such as gum Arabic, modified starch and maltodextrin which form glassy matrices after rapid drying. Spray-dried alginate or pectin particles are amorphous dry matrices without ionic cross-linked structure and their release behaviour is prone to rapid dissolution [37].

The encapsulation efficiency for lipophilic compounds is generally within 70-95% range using gum Arabic or whey protein isolate, but it decreases for hydrophilic compounds due to surface deposition. Inlet air temperature has been identified as the most important process parameter as heat-sensitive core materials degrade above 180°C. The presence of secondary wall materials like inulin, maltodextrin or whey protein considerably increases EE% due to the formation of a more continuous and less porous shell [38].

5.3 Lyophilization

If the retention of biological activity is more important than cost-effectiveness, the preferred method for thermally labile core materials such as heat-sensitive probiotics, volatile flavours or unstable vitamins is freeze drying (lyophilisation). The aqueous wall-core solution is frozen at -40 to -80 °C and water is removed by sublimation under vacuum. EE% values are generally higher than 85%. However, the dried matrix is porous and can be susceptible to oxidative degradation if not packaged under inert atmosphere [39]. Freeze drying uses about 5-10 times as much energy per unit mass of product as spray drying.

5.4 Coacervation

Complex coacervation is a liquid-liquid phase separation process in which oppositely charged polymers — most classically gelatin (cationic) and gum Arabic (anionic) — associate to form a polymer-rich coacervate phase that spontaneously deposits around core droplets. The shell is hardened by crosslinking with transglutaminase or glutaraldehyde. Complex coacervation yields the highest EE% of any encapsulation technique (often >90%) for lipophilic cores including essential oils, carotenoids, and omega-3 fatty acids [40]. The process is sensitive to pH, ionic strength, and biopolymer ratio, making optimisation demanding, and industrial scale-up has historically been challenging.

5.5 Extrusion

Extrusion encapsulation uses a co-extrusion nozzle to deliver wall material and core material concentrically, forming capsules with controlled shell thickness. It is suited to larger capsules (500–5000 µm) for confectionery and bakery applications. In extrusion, unlike spray drying, the cores are not exposed to heat and hence can be used for heat sensitive and volatile cores. It has higher geometric control but capital cost is higher than drip ionic gelation [41].

Table 3: Comparative assessment of principal microencapsulation techniques for food-grade bioactive delivery

Technique	Cost	Scale-up potential	Energy use	Typical EE%	Heat exposure	Best core type
Ionic gelation	Low	Medium	Low	32–90%	None	Probiotics, polyphenols, minerals [42]
Spray drying	Low	Excellent	Medium	70–95%	High (150–200°C)	Flavors, carotenoids, vitamins [43]
Freeze drying	High	Poor	Very high	80–96%	None	Probiotics, volatile actives [44]
Coacervation	Medium	Moderate	Medium	88–99%	Low	Essential oils, omega-3, lipophilics [45]
Extrusion	Medium	Good	Low	75–92%	None	Confectionery, bakery, volatiles [46]

6. Factors Governing Encapsulation Efficiency

The efficiency of encapsulation (EE%) is defined as the percentage of the total core material initially present that is retained within the microcapsule after encapsulation. Despite their central importance, EE% values are not always directly comparable across the literature because different methods measure them in different ways. Some groups report the ratio

of encapsulated core to total core added, while others report the ratio of core in the final dried product. Reporting of the EE% calculation method should be mandatory in publications.

6.1 Wall Material Concentration and Molecular Weight

For sodium alginate, concentrations between 1% and 3% w/v are most commonly used in ionic gelation. Below 1%, insufficient viscosity yields unstable beads; above 3%, solution viscosity impedes extrusion or atomization. In this range, the EE% tends to increase with increasing alginate concentration, for the formation of a denser gel network, but not in a linear manner [47]. For gum Arabic in spray drying, concentrations of 10–40% are typical due to its lower viscosity at equivalent concentrations.

6.2 Crosslinker Concentration

CaCl2 concentrations ranging from 0.3% to 5% w/v have been investigated in alginate systems. Santos et al. [48] found the optimal concentration for annatto extract was as low as 0.3%, because at very high CaCl2 concentrations the bead surface crosslinks so rapidly that a dense gel skin forms before Ca2+ can penetrate the interior, resulting in a hollow or heterogeneously gelled structure- confirmed by SEM in Ching et al. [49]. This rapid surface gelation effect explains why moderating crosslinking kinetics can improve bead structural uniformity.

6.3 Core-to-Wall Ratio

An excess of core relative to wall material leads to surface deposition and leakage; excess wall material increases protection but reduces loading capacity per unit encapsulant mass. A core loading of approximately 20% w/w of total solids has frequently been reported as close to optimal for alginate-based systems, though this varies substantially with core physicochemical properties.

6.4 Core Material Physicochemical Properties

The size, charge, and hydrophilicity of the core compound fundamentally constrain achievable EE%. Large macromolecules (MW > 5 kDa) are retained well within hydrogel matrices because their diffusion coefficients are very low relative to the gel mesh. Small molecules, particularly hydrophilic and uncharged or positively charged ones, diffuse more freely and leach during bead formation. Ions of mineral elements such as iron (Fe2+, Fe3+) and zinc (Zn2+) are extreme cases: they are small, highly mobile in aqueous media, and positively charged at physiological pH- the opposite of the gel surface charge. This combination creates systematically low EE% for mineral ionic species in alginate gels [33].

Composite wall systems substantially improve retention for these difficult core materials. Alginate-chitosan bilayer beads, where a positively charged chitosan outer shell is deposited by dip-coating, provide electrostatic retention for anionic core-wall complexes and reduce surface pore size below the diffusion threshold for small ions [10]. Alginate-pectin composites form denser interpenetrating networks that improve both mechanical strength and retention of small polar molecules [8, 50] Fig 3.

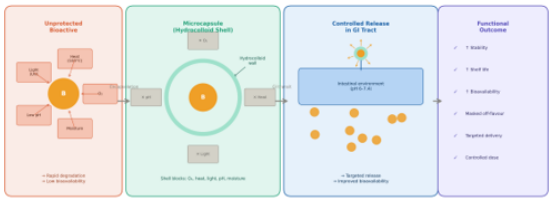

Figure 4. Conceptual overview of the role of microencapsulation using natural hydrocolloids in protecting bioactive compounds from environmental stressors, achieving controlled gastrointestinal release, and improving functional outcomes in food systems. B = bioactive compound Calderón-Oliver, M., & Ponce-Alquicira, E. (2022).

7. In Vitro Release Profile and Controlled Release Behavior

Protection is not the only functional reason for microencapsulation. The other main reason is to control and predict release from hydrocolloid matrices. The standard experimental approach is in vitro release studies, which are usually carried out in simulated gastric fluid (SGF, pH 1.2 with pepsin) and simulated intestinal fluid (SIF, pH 7.4 with pancreatin). It is worth noting that in vitro models (including the widely used static INFOGEST protocol) are major simplifications of the dynamic environment of the gastrointestinal tract, and the correlation between in vitro and in vivo bioavailability is not well established for hydrocolloid-encapsulated systems [41].

7.1 P^H-Triggered Release from Alginate Matrices

The calcium-alginate beads are swelling and dissolving in a pH dependent manner. At gastric pH (1.2-2.0) the protonation of the carboxyl groups causes contraction of the gel network and decrease of the diffusion. In the intestinal phase, increased electrostatic repulsion causes swelling and increased permeability as pH increases above 4.5 due to deprotonation. At pH > 6, the calcium alginate network begins to dissolve progressively due to calcium chelation by phosphates [34]. The release kinetics are usually modelled with the Korsmeyer-Peppas power law ($M_t/M_\infty = kt^n$), where the exponent $n < 0.5$ is indicative of Fickian diffusion; n between 0.5 and 1.0 indicates anomalous (non-Fickian) transport due to combined diffusion and matrix swelling. For most of the alginate-polyphenol systems reported, n values fall in the anomalous transport range [23].

7.2 Burst Release and Its Mitigation

Burst release- rapid uncontrolled release of a large fraction of encapsulated compound in the initial minutes- is common in hydrocolloid encapsulation, particularly for hydrophilic small molecules deposited near the bead surface during formation. Studies report initial burst releases of 20–45% within the first 30 minutes in SGF for alginate-polyphenol beads [23]. Burst release is minimised by chitosan secondary coating, which reduces surface porosity through electrostatic complexation [10].

8. Applications in Functional Food Products

8.1 Polyphenol Encapsulation

Polyphenols are the most extensively studied class of bioactive compounds in food encapsulation research. Curcumin, the primary bioactive of turmeric (*Curcuma longa*) - a spice of central importance in Indian cuisine and traditional medicine- has served as a model compound. Its combination of low aqueous solubility, susceptibility to alkaline degradation, and poor oral bioavailability (< 1% in free form) make it an ideal test case. Chitosan-alginate composites have been reported to achieve EE% of 75–92% for curcumin, with significant improvements in Bioaccessibility during in vitro digestion [24]. Anthocyanins from colored fruits- including Indian varieties such as jamun (*Syzygium cumini*) and mulberry- have been encapsulated in maltodextrin-gum Arabic matrices by spray drying with EE% of 80–94%, with markedly superior colour stability during storage [25].

8.2 Mineral Encapsulation

The encapsulation of mineral micronutrients- particularly iron- receives growing attention in the context of iron-deficiency anaemia, which affects an estimated 1.2 billion individuals globally, with adolescent females in South Asia among the most nutritionally vulnerable groups [6]. Iron encapsulation in sodium alginate via ionic gelation has been investigated to mask metallic off-flavours and reduce oxidative interactions. Reported EE% values are highly variable (ranging from < 30% to > 75%), depending on the iron salt form, alginate concentration, and the presence of a chitosan coating [27]. The highest EE% for mineral ions have been achieved with composite alginate-chitosan wall systems, where the positively charged chitosan layer electrostatically retains iron-alginate complexes.

Analysts working with iron-loaded alginate beads must use complete acid dissolution of the bead matrix (HCl or HNO3) prior to any iron estimation method. Standard colourimetric methods, such as o-phenanthroline or ferrozine assays, applied to intact alginate beads yield falsely low readings because the intact gel shields the iron from the chromogenic reagent- an analytical artifact that can mimic encapsulation failure [33].

8.3 Probiotics Encapsulation

Hydrocolloid microencapsulation is perhaps the most commercially important application of maintaining probiotic viability through processing, storage and gastric transit. Calcium alginate encapsulation of *Lactobacillus plantarum* has consistently shown better survival with reductions of only 1–2 log CFU/g for encapsulated bacteria compared to 3–5 log CFU/g for free bacteria in simulated gastric conditions [29]. Protection is further improved by chitosan coating of alginate beads. The dense hydrogen-bonded network of composite alginate-konjac glucomannan matrices has shown enhanced probiotic viability [39] Table 4.

8.4 Use in Functional Confectionery

Gummies, chewing gum and chocolate matrices are high growth segments for delivery of microencapsulated bioactives. Gummies are attractive because the semi-solid gel matrix is compatible with bead incorporation of sub-300 µm size without compromising the integrity of the capsule. The microencapsulated vitamin C in gum Arabic matrices showed significantly higher retention during storage at 25°C and 60% relative humidity compared to the free vitamin C [30]. Spray-dried or coacervated microcapsules (100-300 µm) are more suitable than ionic gelation beads (>500 µm) for transparent or smooth textured confectionery applications.

Table 4: Summary of selected recent studies on natural hydrocolloid microencapsulation of food bioactive compounds

Core material	Wall material	Technique	EE%	Year	Reference
Annatto extract (bixin)	Sodium alginate	External vs internal ionic gelation	61.6–71.0	2021	Naranjo-Durán et al., 2021
Papaya leaf extract	Alginate + Pectin	Ionic gelation (RSM)	32.5–85.6	2023	Wani KM et al., 2024
Lactobacillus plantarum	Alginate-chitosan-pectin	Ionic gelation + coating	1–2 log loss in SGF	2024	Liu et al., 2024
Gallic acid	Pectin + Alginate	Spray drying	78–88	2020	Fang, Z., & Bhandari, B. 2010
Beta-carotene	Sodium alginate	Ionic gelation	75–92	2016	Bakry et al., 2016
Curcumin	Chitosan-alginate	Ionic gelation	75–92	2022	Kumar et al., 2022
Multiple Bio actives	Alginate, chitosan, pectin, gum Arabic	Spray drying, freeze drying, coacervation	50–95	2022	Kumar et al., 2022

9. Characterisation Methods for Microencapsulated Systems

The characterisation of microcapsules is as important as their production, yet inconsistency in characterisation approaches across the literature limits cross-study comparability. A minimum characterization battery recommended for publication-quality work includes: encapsulation efficiency, particle size distribution, surface morphology by electron microscopy, FTIR spectroscopy, thermal analysis, and in vitro release under simulated digestive conditions.

Scanning electron microscopy (SEM) provides direct visualisation of bead surface morphology and is essential for identifying cracking, pore formation, or incomplete surface coverage. Smooth, spherical beads with no visible surface pores are associated with higher EE% and slower release. Confocal laser scanning microscopy (CLSM) with fluorescently labelled core and wall materials provides direct spatial visualisation of core distribution within the bead- distinguishing internal loading from surface deposition- an insight that bulk analytical methods cannot provide.

Laser diffraction particle size analysis provides the full volume-based particle size distribution (D10, D50, D90), more informative than a single mean diameter. Zeta potential measurement by electrophoretic light scattering quantifies surface charge: values more negative than -30 mV or more positive than +30 mV indicate colloidal stability via electrostatic repulsion, whereas values near zero suggest susceptibility to aggregation [41]. Zeta potential is particularly valuable for verifying bilayer coating deposition- successful chitosan coating over an alginate bead is confirmed by a shift from strongly negative to positive zeta potential.

FTIR spectroscopy confirms the presence of wall material on capsule surfaces. It identifies wall-core interactions- shifts in characteristic alginate carboxyl group absorption (1600–1650 cm-1 for COO- asymmetric stretch) are interpreted as evidence of crosslinking or wall-core interactions. X-ray diffraction (XRD) distinguishes crystalline from amorphous core states within the capsule: the loss of crystalline diffraction peaks of the core compound indicates amorphous dispersion within the wall matrix, which is generally associated with an improved dissolution rate. Differential scanning calorimetry (DSC) confirms encapsulation and characterises thermal stability of the capsule. Thermogravimetric analysis (TGA) quantifies moisture content and thermal degradation profiles, providing data on wall integrity under temperature stress.

Encapsulation efficiency is measured by either direct measurement of core in the wash solution versus total core, or by indirect calculation from the difference between total core added and core in the external medium. Neither approach is without limitations, and the calculation method must be explicitly reported. For mineral-loaded alginate beads specifically, complete acid digestion of the bead matrix is mandatory before any core quantification step [33].

10. Constraints and continuing difficulties

10.1 Preservation of Small Water-soluble Compounds

Hydrogel matrices of natural hydrocolloids show low retention of compounds with a molecular weight of <300 Da, particularly when they are hydrophilic and uncharged or positively charged at the encapsulation P^H. The average pore size of calcium-alginate gels has been estimated to be 5-200 nm depending on concentration and M/G ratio [33]. This is large compared to the hydrodynamic radii of small organic molecules and mineral ions. Composite wall systems are required to reduce the effective pore size, complexation of the core with a larger carrier molecule prior to encapsulation or a complete change to spray drying with a glassy wall matrix to enhance retention.

10.2 Discrepancy between in vitro and in vivo data

The vast majority of the studies dealing with microencapsulation use in vitro digestion models to assess the release and state of improved bioavailability . For studies that have done paired in vitro and in vivo evaluations of the same encapsulated compound, poor quantitative correlation is often seen [41], although the qualitative trend of improvement (encapsulated better than free) is usually maintained. The food science community should be wary of bioavailability claims based solely on in vitro data.

10.3 Scalability and Industrial Translation

Much of the literature on microencapsulation consists of experiments on a laboratory scale, with equipment and conditions that are not directly scalable to industrial volumes without re-optimization. For example, laboratory-scale ionic gelation using an extrusion nozzle produces beads at rates of grams per hour; industrial bead production requires multi-nozzle or vibrating nozzle systems producing kilograms per hour, and the changed hydrodynamic conditions at scale affect bead size distribution and EE% in ways rarely discussed in primary papers [20]. This difference between laboratory EE% values and production-scale values is a key reason why the commercial food industry continues to use established spray-drying techniques. One of the often-neglected metrics in the academic literature, from the economics perspective, is the cost of the encapsulation per kg of the product. The cost difference between freeze drying (around INR 800–1200/kg of finished product) and spray drying (INR 100–200/kg) is very significant for commodity functional food applications, 5-10fold difference, even though freeze drying offers superior EE%.

10.4 Regulatory Aspects in India

Most of the major hydrocolloids are approved as food additives under the FSSAI. However, chitosan and konjac glucomannan are not registered as specific food additives that would limit their use as encapsulated food ingredients in the Indian market. With the Indian nutraceutical market expected to grow to over USD 18 billion by 2030, harmonization of regulations with Codex Alimentarius and EU standards for encapsulation wall materials will become increasingly important for export oriented functional food manufacturers.

11. Future Prospects and Research Priorities

The main structural trend in the field is the development of composite multilayered wall systems exploiting the complementary properties of two or more hydrocolloids. Alginate-chitosan bilayer beads, alginate-pectin composites and alginate-konjac glucomannan matrices overcome specific drawbacks of single-component systems improving retention of small molecules, mechanical strength or probiotic viability respectively. India's unique botanical diversity is an unexplored source for novel hydrocolloid wall materials. The indigenous seed gums of *Sesbania bispinosa, Cassia tora, Moringa oleifera* and *Tamarindus indica* have similar galactomannan or xyloglucan composition as the commercially established materials. These sources, available domestically, could offer sustainable, lowcost alternatives to imported hydrocolloids and are a unique research direction for Indian food technologists. Artificial Intelligence and Machine Learning have great potential for formulation optimisation. Encapsulation efficiency is a non-linear response with multiple variables and is not easy to optimize using traditional one-factor-at-a-time experimental approaches or even response surface methodology. Published experimental datasets can be used to train supervised machine-learning models capable of predicting the EE% for new formulation conditions with reasonable accuracy, potentially lessening the experimental workload. Neural network models have been used for spray-drying conditions for polyphenol encapsulation with promising early results [42]. Stimuli-responsive smart delivery systems are the next generation of encapsulation by hydrocolloids. Enzyme-responsive systems are being developed to release their payload in response to the enzymatic activity of the colonic microbiome (beta-glucuronidase, nitro reductase), in addition to the intrinsic pH responsiveness of alginate and pectin, for colon-targeted nutraceutical delivery [43]. These systems provide the opportunity to deliver prebiotic or anti-inflammatory

compounds intact to the colon - a functionality that significantly enhances the efficacy of encapsulated actives. There is now academic interest in life-cycle sustainability assessment of hydrocolloid wall materials. Materials derived from agricultural by-products (citrus peel pectin, guar bean meal guar gum, tamarind seed TSP) have a lower environmental footprint than materials derived from marine harvesting (alginate, carrageenan) or fermentation (xanthan, gellan) [41]. As sustainability is being increasingly used as a criterion in the development of functional foods, the environmental profile of encapsulation wall materials will be a competitive differentiator. Regulatory advancement is needed for wider adoption of composite encapsulation systems in the Indian market, specifically clarification on FSSAI stance on chitosan, konjac glucomannan and gellan gum as food additives. Indian food technologists and food scientists must come forward to work with the FSSAI Scientific Committee so that Codex Alimentarius standards for these materials are incorporated in our domestic regulations.

12. Conclusion

Natural hydrocolloids constitute a diverse and scientifically validated class of wall materials for microencapsulation of bioactive food compounds. Their appeal rests on food-safe status, biodegradability, functional versatility, and a capacity for pH-responsive or triggered release that aligns with gastrointestinal physiology. Sodium alginate, by virtue of its simple ionic gelation chemistry, remains the most extensively investigated single-component hydrocolloid and has demonstrated clear performance advantages for probiotics, large polyphenols, carotenoids, and heat-labile vitamins.

Emerging hydrocolloids- particularly tamarind seed polysaccharide, konjac glucomannan, and xanthan gum- offer distinctive advantages in terms of domestic availability in India, sustainability, or functional complementarity with established materials, and merit prioritised investigation for Indian functional food applications.

The limitations of hydrocolloid encapsulation for small, ionic, positively charged core compounds- including mineral ions critical for addressing iron-deficiency anemia in South Asian adolescent populations- are real, mechanistically understood, and must be explicitly acknowledged in experimental design. Complete acid digestion of the gel matrix prior to mineral quantification is a non-negotiable analytical requirement.

Future progress in this field will be driven by composite wall systems, machine learning-assisted formulation design, stimuli-responsive delivery architectures, and a sustainability-conscious approach to wall material selection. Indian food science research is well positioned to contribute distinctively to this field through access to unique botanical raw materials and the clinical relevance of mineral and traditional bioactive encapsulation for domestic public health priorities.

Conflict of Interest

The authors declare that there are no financial, personal, or professional conflicts of interest that could have influenced the work reported in this manuscript.

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