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REVIEW PAPER

Starches in the encapsulation of plant active ingredients: state of the art and research trends

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Abstract

As a natural polymer, starches and their derivatives have received widespread attention in the cosmetic and pharmaceutical industries, particularly for their use as a coating material. In this sense, as an encapsulating agent, starches stand out, considering the number of compounds that they can trap. Additionally, they provide a nutritional contribution and may improve acceptance by patients. As such, this type of material may serve as an alternative to overcome gaps such as loss of activity of the active principles, low assimilation, or deterioration under environmental and physiological conditions. In this paper, we aim to present the state of the art and research trends on the use of starch as a wall material for the encapsulation of active principles of plant origin. It was found that the most-encapsulated active principles are essential oils and polyphenols; native or modified starches are typically used, either as the sole wall material or in combination with other polymers; and the most widely used methodology is spray drying. The reviewed studies indicate the potential of starches for their use in active ingredient encapsulation processes, improving their viability and expanding their range of applications in different industries, as well as showing a clearly increasing publication trend over the last 10 years.

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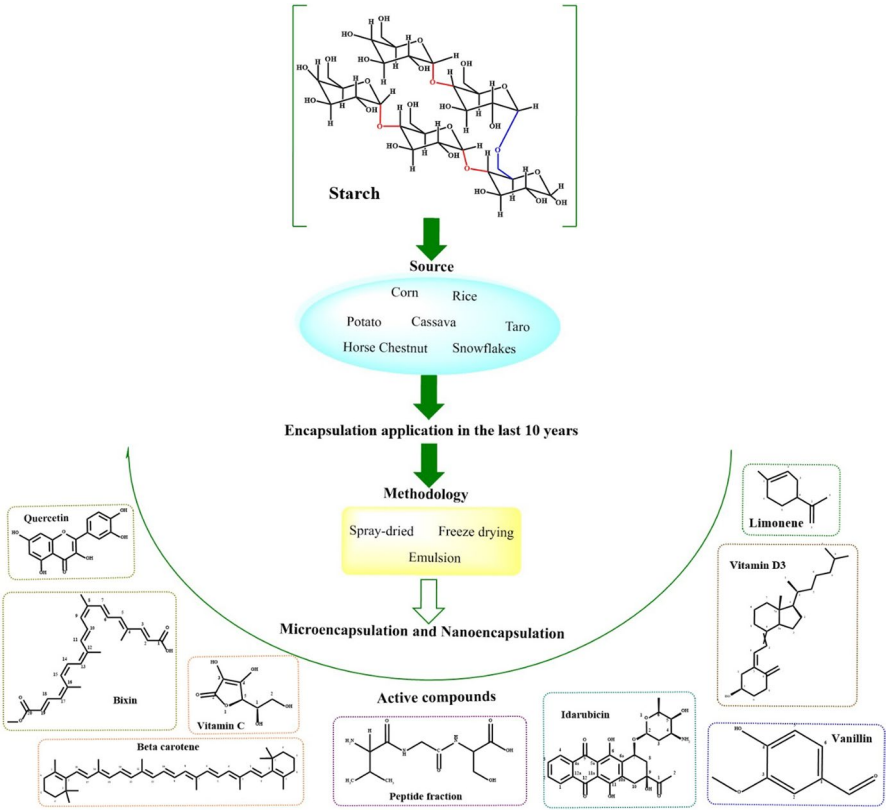
Graphical abstract



Keywords Starch · Encapsulations · Microencapsulation · Nanoencapsulation · Active compounds

Introduction

In recent decades, the high characterization of natural products of plant origin and their potential applications in different health areas—both in prevention and disease control—has led to a gradual increase in their research for pharmaceutical purposes; however, the lability of compounds, their low assimilation, and the difficulty of establishing efficient doses, due to their characteristic tendency of attacking different target sites, have generated challenges for the scientific community over the past few decades, in terms of generating new alternative products to those already established [1–5].

Gaps in the literature include the simultaneous administration of active principles, targeting, and controlled release, as well as extension of the shelf life of

compounds; researchers are currently seeking to overcome these issues through the implementation of various technologies [6, 7]. Encapsulation technology is an alternative that stands out for its ease, the number of compounds that can be encapsulated, and the variety of wall materials that are currently available (both natural and synthetic); however, the requirement of a particular design for each administration route is a key challenge for this methodology, as it is necessary to regulate the size, zeta potential, morphology, toxicity, and release characteristics for each treatment [8–10]. Considering this, extracts and new compounds with physical, chemical, and biological properties of interest are discovered frequently, which represent determinant factors for the increased characterization of new natural polymers to be used as wall materials and/or in new encapsulation methodologies [11].

In encapsulation process development, it is generally important to know the solubility of the compound (e.g., water or fat soluble); for example, a common material used for fat-soluble compounds is Arabic gum, while maltodextrin is used for water-soluble compounds [12]. Wall materials for encapsulation must have high solubility, film formation, drying properties, low oxygen permeability, non-toxicity, biocompatibility, biodegradability, and low viscosity in emulsion [13–16].

Natural polymers, such as pectin, cellulose, alginate, proteins, and starch, have received widespread attention, due to their bioavailability, low cost, and non-toxicity [2, 4, 17–19]. Among these materials, starch has become the focus of significant scientific research. It has various advantages, including its biocompatibility and hypoallergenic character, in addition to its acceptability, as it is a common component in the human diet [18, 20, 21]. Furthermore, through modification processes, it is possible to improve some of its disadvantageous physical and chemistry properties, allowing for degradation rate regulation and improved solubility index, which has placed it as a polymer of interest when developing encapsulation processes for the successful administration of active principles (e.g., secondary metabolites, vitamins, and even some synthetic drugs) [10, 20–23].

Once encapsulated, the literature has reported certain good characteristics, such as high entrapment efficiency, yield, oxidation stability, release parameters, cellular absorption, pharmacokinetics, and morphological and physicochemical characteristics [14, 24–26]; for these reasons, an increasing trend in the use of starches for encapsulation can be observed over the last few decades. This review aims to demonstrate how scientific gaps regarding the administration of active plant ingredients are being addressed, through an analysis of the literature on techniques and applications related to the native and modified starches of various species; in particular, as a wall material for the encapsulation of natural compounds. This was achieved by searching the SCOPUS and PubMed databases for publications over the last 10 years. We focus on highlighting the importance of one of the most abundant natural polymers worldwide—that is, starch—to overcome knowledge gaps associated to the protection, administration, and release of active ingredients, indicating it as a material with significant potential for biomedical applications in both the short and long term. This article offers a complete starting point for future research on the application and use of native and modified starches.

Gaps in the administration of active ingredients of plant origin

Multiple works have been carried out in the past few decades which have sought to bring natural compounds into advanced tests, and even as far as into the development commercial products [1, 6, 7, 27, 28]. However, there is some difficulty in evaluating them over time, due to the rapid deterioration they tend to present when interacting with oxygen, humidity, and UV radiation during their storage. Furthermore, their irregular assimilation, caused by their shape and structural variability and the genetic/condition differences of patients, has generated a need for careful design and choice of administration route, in order to establish effective times and doses. Additionally, all administration routes for drugs present some or all of the following difficulties: easy degradation or denaturation, enzymatic attacks, pH variations, or low permeability of membranes (Fig. 1) [8, 9, 29, 30]. This makes it difficult to predict the correct dosage to ensure an active and efficient pharmacological concentration [7, 29, 31, 32], especially when considering that principles commonly have multiple target sites [7, 29, 31, 32].

In this sense, investigations into administration systems and controlled release through encapsulation technologies using natural polymers (e.g., starches, gums, cellulose, dextran, proteins) for protection of the active compound have indicated the economic advantage of such biocompatible alternatives, along with high acceptance by patients; furthermore, according to the advances made over the past decade, these may effectively address current knowledge gaps [2–4, 6, 8, 9, 17–19, 22, 32]. On the other hand, unlike the chemical industry, in food and pharmaceutical applications, the use of materials classified as GRAS (Generally Recognized

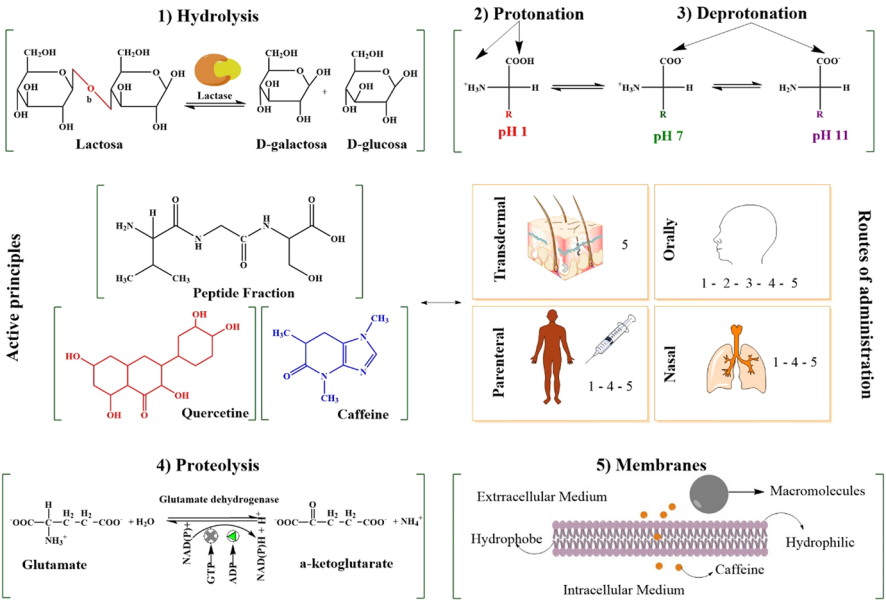


Fig. 1 Main problems of active principles in physiological systems

As Safe) for encapsulation has become important [13, 33], along with the need for new products that promote health benefits in addition to nutrition and energy. This has been reflected in growing interest in the development of functional ingredients [13, 34–39]. Therefore, the synthetic additives currently applied must be replaced by natural compounds obtained through green technologies; that is, biotechnological processes that yield materials that are safe for human consumption [36].

There are a variety of natural materials that have been researched for the encapsulation of food, pharmaceutical, and cosmetic products/ingredients, including hydrogenated oils, waxes, maltodextrins, starches, starch derivatives, pectin, glucans, cellulose, and protein materials (e.g., polypeptides, soy proteins, milk proteins, gelatin derivatives, and gums) [10, 14, 15, 24, 37, 40–47]. These materials must have the following characteristics [48, 49]:

- Good rheological properties at high concentrations;
- Disperse or emulsify the active material and stabilize the emulsion;
- Do not react chemically with the active ingredients to be encapsulated;
- Keep the active material within its structure during processing or storage;
- Completely release solvents and other materials used during the encapsulation process;
- Protect the active ingredient against environmental conditions;
- Solubility in solvents must be acceptable for the food and pharmaceutical industries.

In this sense, gum Arabic is the most used in encapsulation processes, applied by spray drying and lyophilization [37, 44, 50, 51]. In these systems, carbohydrates are generally used as a secondary wall material to improve the dry properties of the capsules [16]; nevertheless, the high cost, low availability, and impurities present in gum Arabic have promoted the search for new wall materials [52]. Therefore, carbohydrates such as native or modified starch have attracted attention, in terms of their use in the encapsulation of bioactive compounds, on account of their high availability, low cost, degradability, and hypoallergenic characteristics [12, 24, 25].

Starch: sources, characteristics, advantages, and disadvantages

Starch is an abundant material, with low cost, nontoxic, naturally renewable, and biodegradable properties. Many plants produce starches as energy storage molecules, and it also serves as a nutritional source and is a basic product in alimentary industries. It consists of two kinds of α-glucan (amylose and amylopectin), which present molecular structural differences [21, 53–57]. Amylose is a linear molecule that consists of D-glucopyranose linked by α-1,4-glycosidic bonds and slightly branched by α-1,6-glycosidic bonds. Meanwhile, amylopectin is a highly branched polymer, containing 5–6% α-1,6-glycosidic bonds at the branch points, which can be classified into A, B, or C chains, according to their multiplicity in the branch [58–60]. The intricate organization of these two molecules produces relatively

water-insoluble granules that possess alternating amorphous and semi-crystalline regions between 100 and 400 nm thick [55, 61–64].

Common sources of starch are corn, potato, rice, wheat, and cassava, while less-common sources include barley, quinoa, oats, sago, sorghum, and yam [65]. The botanical source offers different structural and physicochemical properties, such as variation in the composition of amylose and minor components, the amylopectin structure, granular architecture, and morphology, which have important impacts on the application [10, 23, 65, 66]. These polymers and their derivatives have received attention in the plastics and pharmaceutical industries, due to their gelling ability, film formation, and biodegradable properties [22, 59, 61, 62, 65, 67–71]. For example, amylose and long-branched chains of amylopectin have the unique characteristic of forming helical inclusion complexes with various hydrophobic compounds, such as iodine, lipids, and flavoring compounds (Fig. 2) [68, 72, 73]. For these reasons, in the pharmaceutical industry, research has been carried out in which starch has found applications in solid-oral dosage forms, as a binder, diluent, and/or disintegrant [10, 23, 65, 74].

The systems that can be obtained (as will be detailed throughout the document) have the ability to catch, protect, and release (in a controlled way, in some cases) a wide range of active compounds, which places starch as a biopolymer with significant potential for the development of new food and pharmaceutical products, with acceptable sensory, nutritional, and flavor properties, as well as facilitating a longer shelf life [23, 73, 75, 76].

However, some problems have been reported; for example, the industrial application of native starch is limited due to properties such as high hot paste viscosity, instability under thermal and shear stresses, and tendency to rapidly retrograde, leading to low stability. Poor cold storage and poor resistance to acid, shear, and high temperatures can lead to deterioration of the quality of the product [77, 78].

Native starch has strong hydrophilicity, due to the presence of large amounts of hydrophilic hydroxyl groups, causing starch molecules to exhibit strong inter- and

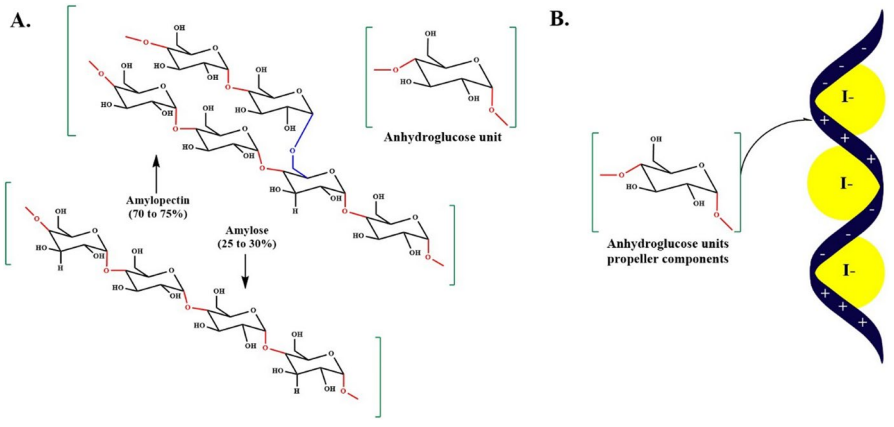


Fig. 2 General structure of starch, **A** Amylose and amylopectin; **B** Helical complex

intramolecular hydrogen bonds, making it a poorly soluble material in water at room temperature and easily self-aggregating in organic media [73, 79]. These properties limit the application of starch in food, cosmetics, and medicines, due to the difficulties associated with its industrialization. This deficiency can be overcome by combining it with other polymers with different water activities [79–81], or by making structural modifications, as the hydrophobic groups of OSA-modified starch, for example, can decrease the hydrophilicity and water dispersibility of the resulting particles [81].

On the other hand, foods that use native starch are often easy for the human body to digest, leading to excess energy and a host of health subproblems [82], a problem that has been addressed through treatment for the generation of resistant starches, which have been reported to have human health-related benefits, as they can improve blood glucose and insulin response, reduce blood glucose and cholesterol levels, control lipid metabolism, and regulate intestinal microorganisms [83].

In this sense, starches have the clear advantage of being able to modify their physical and chemical characteristics through different methodologies, which has allowed for the adjustment of certain properties required for their application in encapsulation; in addition to mixing with other polymers, this allows for significant expansion of the range of compounds that can be caught.

Encapsulation with native starches

Native starch is one of the natural materials commonly used in encapsulation processes, due to its high trapping efficiency. It has been used as a wall material in encapsulation to contain and protect volatile compounds, scents, as well as to replace fats (Fig. 3) and stabilize emulsions, where biodegradability, alimentary grade use, and pharmaceutical use are relevant aspects [13, 28, 33, 46, 61, 84–90]. The increase in research in this area over the last 10 years has indicated the tendency to evaluate multiple starch sources and its derivatives for the encapsulation process, achieving encapsulations at micro- and nanoscale, both in mixture with other polymers and individually, depending on the methodology and the intended use.

Microencapsulation is a technique in which solid or liquid particles are trapped by a wall material, providing a physical barrier between the central compound and its environment, in which the active agent forms a core surrounded by an inert diffusion barrier called the microcapsule, which is no larger than 1000 μm in diameter [15, 91]. The key components for successful encapsulation are the process itself, the intrinsic physicochemical characteristics of the matrix (generally natural polymers), and the active compound [37, 41, 92–95]. This technique has been used to overcome current gaps, such as low absorption in the intestine (due to intestinal and hepatic metabolism and rapid elimination) and, therefore, the low bioavailability of the active ingredients [92, 96–99]; therefore, it has been widely used in the pharmacology, food, and cosmetics sectors [40, 92, 100–104].

Different methods for microencapsulation have been reported, where their main difference is the means of combination of the wall material and the core, encompassing chemical (e.g., suspension polymerization, emulsion polymerization,

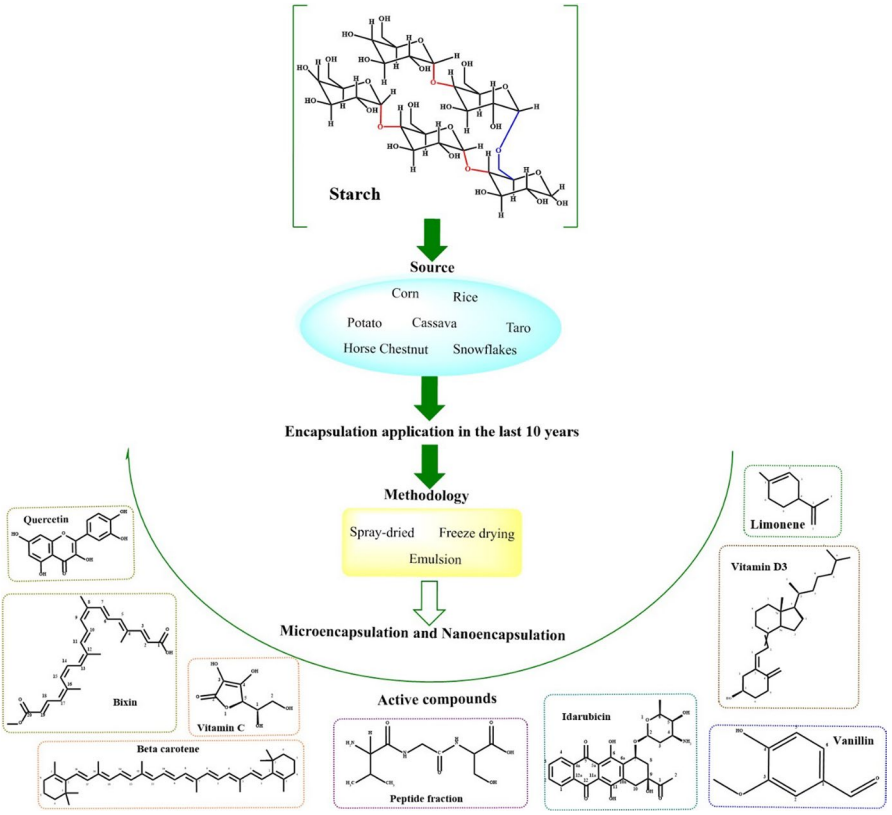


Fig. 3 Compound classes, methodologies, and native starch sources used in encapsulation

dispersion, interfacial) and physical methods (e.g., suspension cross-linking, evaporation solvent, coacervation, spray drying) [14, 101, 105, 106]. The evaluation of microcapsules must take into account the content of the compound and its time-dependent stability, maintenance of biological activity, and the structural changes of the matrices and their release processes, as controlled release of the active ingredient(s) is typically sought [61, 101, 107]. Spray drying the most-used microencapsulation technique, due to its efficiency, continuous production, and ease of industrialization [15, 16, 40, 52, 99, 105, 106, 108, 109]; however, a related disadvantage is the use of high temperatures for drying, which leads to greater oxidation of some active ingredients. Therefore, drying at lower temperatures (lyophilization) is an alternative that has drawn the attention of researchers, through which heat-sensitive and aromatic materials (e.g., oils) are dehydrated essentially [106, 110, 111].

On the other hand, nanoparticles—solid or colloidal particles that consist of macromolecular substances that vary in size from 10 to 1000 nm [92, 112, 113]—have obtained growing interest, in terms of their use instead of microparticles in pharmaceutical and food applications. This is based on their ability to increase

the absorption rate, improve bioavailability, and the high stability of the compounds, as well as their bioactivity, long residence time, passive accumulation in tissues, high encapsulation of principles, slow release, shelf-life optimization, and allowing for the administration of active principles for the therapy of different diseases, by intravenous, oral, and/or nasal routes, to various target sites in the body [24, 45, 67, 112, 114–118].

Nanoparticles with different physicochemical, technical, or mechanical properties can be obtained, as well as modulated release characteristics for bioactive or therapeutic agents [65]; for example, its size can affect the interaction with other molecules, cells, and tissues, or its ability to be absorbed by cells through paracellular penetration, endocytosis, or lymphoid tissues associated with mucous [118]. Nanoencapsulation of poorly water-soluble compounds can improve their transparency, stability, and solubility, consequently increasing the gastrointestinal absorption rate [116, 119]. Furthermore, the formulation of cytotoxic agents in polymeric nanoparticles provides a significant reduction in side effects by increasing the therapeutic index of antitumor drugs.

Multiple active ingredients have been encapsulated with native starches in microencapsulation and nanoencapsulation contexts, as reported in the literature. Starches with type B crystallinity, such as potato starch, have adequate ability to embed, protect, and administer molecules of interest. Such starches have been evaluated as trapping agents for three drugs (ibuprofen, benzocaine, and sulfapyridine), a nutraceutical (curcumin), a flavoring compound (thymol), and a vitamin (ascorbic acid). Using this type of starch, the authors observed a delay in the degradation time and improved bioavailability of the principles, overcoming the limited half-life of some of the compounds evaluated [62]. In the encapsulation of phenolic compounds, systems using potato starch allow for the generation of microcapsules that are homogeneous, when compared with wall materials such as maltodextrins, when trapping tannins [95].

Corn starch has been used successfully in the encapsulation of yerba mate extracts, which are rich in phenolic compounds, regulating its release *in vitro* systems and maintaining its antioxidant activity without statistically significant changes, diminishing the loss gap of activity and promoting its evaluation in *in vivo* systems [22].

Other unconventional starch sources have begun to be evaluated, with the increase in demand and the advances made with traditional polymers; for example, horse chestnut, water chestnut, and lotus stem starch have been evaluated for the encapsulation of catechin, establishing entrapment yields greater than 50%, data, which the authors attributed to the particle size diameter (approximately 322 nm), as small particles provide better encapsulation efficiency, due to their ability to form a better film of material around the core, thus improving retention of the encapsulated molecules [45]. These systems initially protected the gastric environment *in vitro* and maintained the biological activity of the compound after its exposure to digestion conditions, thus establishing new sources of starch with suitable characteristics for this type of compound [45]. Table 1 details the works carried out over the last 10 years using native starches as wall materials. We further denote the research that has been carried out to fill existing knowledge gaps concerning the need for

Table 1 Encapsulation of active principles using native starches

Starch source	Active compounds	Encapsulation methodology	Entrapment efficiency	Size	Ref
Rice	Idarubicin	Emulsion and diffusion	85%	214 ± 5 nm	[24]
	Glycosylated triterpenes	Crystallization and sonication	≥ 57%	234–894 nm	[118]
	Vitamin D3	Freeze drying	22.34%	32.04 nm	[120]
	Essential oil	Freeze drying	86.6%	93–113 nm	[68]
	Vitamin C	Spray dried	57.7%	7.2 µm	[121]
	Orange essential oil	Spray dried	≥ 57%	*NR	[33]
Corn	Tea extract	Spray dried	80%	10.26 µm	[122]
	Annatto oil	Emulsion and freeze drying	94 ± 3%	127–142 µm	[36]
	Bixin	Emulsion and lyophilization	23.30–82.91%	*NR	[103]
	Tea polyphenols	Emulsion and freeze drying	10.0%	15–18 µm	[123]
Potato	Vitamin D3	Freeze drying	94.8%	99.2 nm	[120]
	Tannins	Emulsion and freeze drying	27.7–48.8%	20–65 µm	[95]
Cassava	Cinnamon essential oil	Spray dried	98,89 ± 0,50%	*NR	[42]
Taro	Polyphenols	Emulsion	61.5–67.3%	460 nm	[69]
Snowflakes	Achiote oil	Emulsion and freeze drying/spray dried	94 ± 3%	124–142 µm	[36]
Horse chestnut	Catechin	Freeze drying	59.09%	322.7 nm	[45]

**NR* no report

materials that meet the physical and chemical requirements of the different active ingredients characterized.

Despite the encapsulating benefits of starches, those considered to be native have certain limitations, such as their insolubility in water and organic solvents, propensity to retrograde, high viscosity, and the inability to resist high temperatures, thus limiting their application in systems for the release of some active principles and constituting frontiers in research [58, 59, 67, 68]. For this reason, it is necessary to test structural modifications aimed at a specific encapsulation application or desirable characteristic, such as solubility, texture, adhesion capacity, dispersion, or thermal tolerance [124–131]. It has been reported that the physicochemical properties of starches, such as swelling capacity, solubility, degradability, and light transmission, can be significantly affected by chemical modification [65, 67, 69].

Modified starches as wall materials

Modified starches are those that undergo a structural change to improve certain physicochemical characteristics, typically depending on the end use. These modifications can be carried out in different ways and are generally achieved through physical methods that use high temperatures and pressures, chemical methods (e.g., etherification, esterification, cross-linking, oxidation), or through the implementation of biotechnological processes [21, 127, 132–134]. These modifications are directly related to reactions with the hydroxyl groups of the polymer (see Fig. 4).

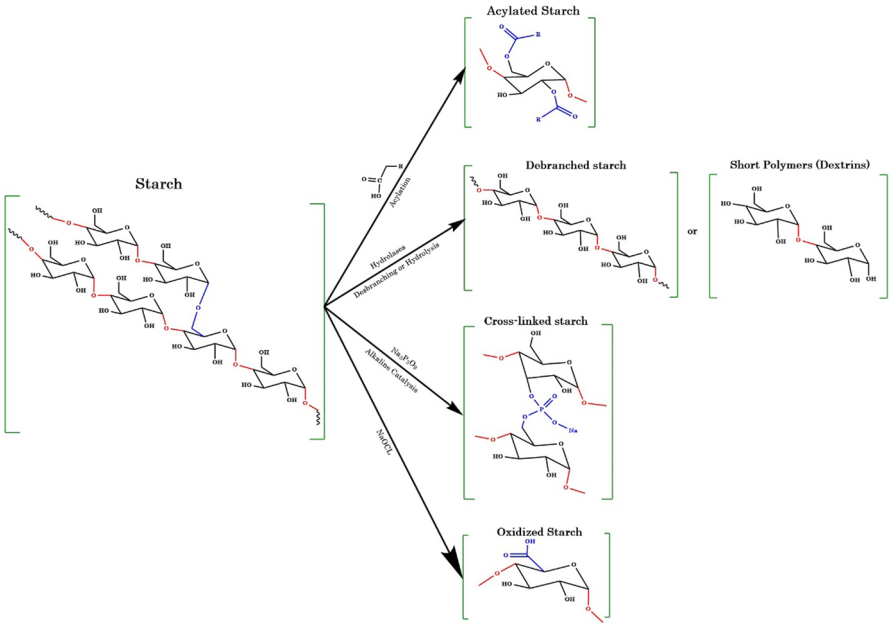


Fig. 4 Main chemical modifications made to starch

Chemical modifications such as acetylation and hydroxypropylation increase the swelling ability, solubility, and light transmission, while cross-linking has been observed to decrease—depending on the type of cross-linking agent and the degree of cross-linking—the power swelling and solubility of starches from various sources. The introduction of acetyl groups into starch molecules leads to structural reorganization caused by steric hindrance, which results in repulsion between the starch molecules, thus facilitating an increase in water percolation within the amorphous regions of granules and a consequent increase in swelling capacity [135, 136].

Modification methods that use physical and chemical processes present marked disadvantages, in terms of their industrial application in important sectors such as pharmacology and cosmetics. The use of aggressive, alkaline, or acidic reaction conditions and toxic and/or environmentally unfriendly chemical products, in addition to unspecified production, limits their suitability, given the high amounts of enantiomeric by-products that can be teratogenic, and reducing their biocompatibility by allowing reactions causing adverse health effects, such as intoxication, allergic responses, cytotoxicity, and/or bioaccumulation [126, 137].

In general, the acetylation and/or acylation of polysaccharides with organic acids and acid derivatives is the most versatile transformations [125, 135]. These modify the hydrophilic nature of the polymer and generate significant changes in the mechanical and thermal properties [69, 138]; in particular, starches esterified with long-chain fatty acids (where the fatty acid chain is greater than eight carbons) have good thermoplastic, hydrophobic, and biodegradability properties [136].

An example of this is hydrophobic starches, such as acetylated starch, which can spontaneously self-associate, forming hydrophobic nuclei. This self-association makes it difficult for fluids to penetrate the particles, protecting the encapsulated active principles of proteolytic enzymes at the physiological level [15, 67, 97], thus enhancing their use in the administration of drugs, genes, and other biomedical applications, especially considering the hydrophilic–hydrophobic balance, low biodegradability, and having been approved by the Food and Drug Administration (FDA) as a food additive [67, 69, 138]. On the other hand, their slow degradation at the intestinal level makes these starches an optimal vehicle for applications of principles that control glycemic dysregulation by not contributing significantly to increases in blood glucose [62]. In addition, they are colorless and tasteless, and present high stability, long residence time, high drug encapsulation, capacity as a stabilizer not dependent on pH and ionic strength of the medium, better storage life, and the ability to translocate through the intestinal barrier, making these the currently most widely used for encapsulation processes [67, 97, 139, 140].

Another case reported as successful is starches modified with succinic anhydride n-octenyl (OSA), derived from waxy corn, which has been reported as suitable for the encapsulation of flavors, vitamins, and spices [67, 97], due to its high viscosity when solubilized in water and its resistance to oxidation, making it adequate for the protection of labile substances and establishing a longer shelf life, which are currently some of the main problems associated with natural products [13, 15, 51].

On the other hand, there has been growing interest in using debranched starch (short glucan chains) as a source for the production of resistant starch, starch-based nanoparticles, and drug carriers [62, 68]. Zhang et al. [141] reported that

debranching could improve the formation of starch and lauric acid complexes, and that this process, in addition to treatment with β-amylase, significantly increased the complexing of stearic acid in potato and corn starches. These materials can also act as dietary fiber, which is fermented in the colon without being assimilated in the upper gastrointestinal tract. The above results confirm such starches as suitable vehicles for drugs that control blood glucose levels [62]. Table 2 details the investigations carried out since 2009 in the area of encapsulation of natural products of plant origin using starches with adjusted characteristics, in order to improve their performance as wall materials.

Even though the range of natural products encapsulated with native and modified starches is wide, as mentioned above, mixtures with other polymers have also been reported in the literature, in order to better adapt the characteristics of the capsules to certain types of compounds and administration systems.

Starches mixed with other polymers as wall materials in encapsulation

Given that the biological actions vary between different bioactives, the joint administration of a mixture of compounds can provide several advantages over the intake of individual formulation. For this reason, such combinations have attracted significant attention, as they can facilitate action against various pharmacological targets, which currently constitutes a key gap in the multifactorial disease treatment field [96]. In this sense, the design of biopolymeric matrices for the simultaneous transport of two or more natural compounds constitutes a new approach to improve the functionality of conventional encapsulation systems [34, 41]. In some cases, mixtures of gums, proteins, and carbohydrates are applied together to improve the encapsulation efficiency and the release of nuclei [94, 97, 145].

Systems using corn starch and alginate, for example, have been used to encapsulate a yerba mate extract and zinc, used as a food additive, both masking the flavors of the two active ingredients and protecting them from deterioration caused by storage and gastric conditions. Once consumed, this system exhibits strong inhibitory activity toward free radicals. The proposed methodology could be used for the simultaneous transport of other active compounds when a possible interaction must be avoided, as expressed by the authors [34].

In this sense, hydrolyzed starch is commonly used as an auxiliary wall material, due to its relative benefits, such as its low cost, neutral taste, low viscosity, and protection against oxidation when used as a wall material in the microencapsulation of food components; however, the main problem associated with this wall material is its low emulsifying capacity. Therefore, it is typically used in combination with other surfactant biopolymers, modified starches, and proteins (e.g., sodium caseinate and whey protein concentrate) to obtain adequate microencapsulation of fish oils [110]. Besides this, for the encapsulation of virgin olive oil, better performance of microcapsules, efficiency, and proportion of internal and external fat were achieved when a combination of proteins and starch was used as a component of the wall. The oils that had the highest amounts of total phenolic content, OSI index, and ratio

Table 2 Encapsulation of active ingredients of vegetable origin using modified starches in the last 10 years

Starch source	Active principle	Encapsulation methodology	Entrapment efficiency	Size	Reference
Commercial modified starch (no modification specified)	Curcumin	Emulsion and spray dried	56.2%	*NR	[92]
	Fish oil	Spray dried	74.19%	18.6 µm	[107]
	Linseed oil	Emulsion and spray dried	40%	2.14–5.2 µm	[52]
	Pequi extract	Spray dried	24–49%	90 µm	[140]
	Lycopene	Spray dried	494.4 µg/g	*NR	[105]
	Vitamin E	Spray dried	94.5%	457–617 nm	[142]
Starch derivatized with n-octenyl succinate	Vitamin C	Emulsion and spray dried	71.5%	12.5 µm	[101]
	Vitamin A	Emulsion and spray dried	96.38 ± 0.71%	66–153 µm	[40]
	B-Carotene	Solvent emulsion and evaporation	65–90%	300–600 nm	[143]
	Lycopene	Emulsion	65–79%	125–250 nm	[144]
	Vitamin E	Emulsion and spray drying	71–79%	208–235 nm	[115]
	Tocopherol	Emulsion	2.5% w/w	150 nm	[139]
	Vitamin E	Emulsion and spray dried	53–63%	1–12 µm	[15]
	Phenolic compounds	Spray dried	70%	5–8 µm	[104]

**NR* no report

between C18:1/C18:2 fatty acids were those that performed best in the microencapsulation process. Furthermore, the fatty acid profile did not change after the process, regardless of the value of the other parameters studied, thus maintaining their biological characteristics [41].

On the other hand, mixtures of cassava starch with gum Arabic and whey protein made it possible to capture limonene in proportions greater than 40% when employing spray drying. This process generated homogeneous capsules without cracks, indicating that the encapsulation system and the used matrices can offer adequate protection to the asset [61]. The presence of starch helps to retain the crystalline phase of gum Arabic and to stabilize the whey protein concentrate system, providing a greater retention capacity for the encapsulated principle. The gum Arabic blend showed higher encapsulation efficiency than whey protein, but its storage stability under accelerated aging conditions was markedly reduced, compared to the whey protein concentrate and cassava starch blends, once again managing to increase shelf life [61].

Finally, protein derivatives, such as oyster peptides, have also been encapsulated using an alginate/chitosan/starch mixture through an (external or internal) emulsion gelling process, in which the swelling power of the microcapsules increased with the presence of starch, which triggered an increase in water absorption. This effect was correlated with the release rates of the peptides, showing a controlled release. The authors demonstrated that the release behavior of these microcapsules was affected by various material properties, including concentration and pH, as well as swelling and erosion of the gel matrix of the system. This study also reported that alginate/chitosan/starch microcapsules may serve as a potential system for the delivery and release of actives in the colon [146]. Other works focused on the use of starches and other polymers in combination are listed in Table 3.

Capsule characteristics

As can be seen from Tables 1, 2, and 3, the developed capsules present varied characteristics. In particular, starch-based encapsulation systems can adopt a range of different morphologies, zeta potential, crystallinity, sizes, and release parameters, depending on the encapsulated active principles, the methodologies used, the starch sources, and the materials with which they are mixed. These properties are relevant, as they open the possibility for their application in the different systems under various administration routes.

Morphology and particle size

Particle size plays an important role in the stability of systems, where a decrease in particle diameter has been reported to increase the bioavailability of the encapsulated compound(s) [116]. The particle size of the capsules and their homogeneous or heterogeneous distribution are closely related to the release properties [18], which is why they are relevant when considering the administration of

Table 3 Starches mixed with other natural polymers as wall materials in encapsulation processes

Starch source	Second material	Ratio	Active principle	Methodology	Entrapment efficiency	Size	Reference
Commercial	Gum Arabic/Maltodextrins	0.17/0.64/0.17	Ubiquinone 10	Emulsion and spray drying	71.53%	17.46 µm	[147]
	Whey Protein/Glucose	5.3%/5.3%/5.3%	Resveratrol	Emulsion	*NR	7–9 µm	[96]
	Gum Arabic/Maltodextrins	45%/25%/10%	Oil	Emulsion and spray drying	75.59%	17.6 µm	[25]
	Alginate	2%/3% in 100 mL of water	Caffeine	Emulsion and freeze drying	78.86%	52.43 µm	[102]
	Gum Arabic/Whey Protein	33%/33%/33%	Anthocyanins	Spray drying	99.2%	*NR	[51]
	Soy protein	17%/30%	Anthocyanins	Spray drying	83%	*NR	[148]
	Chitosan/Alginate	20%/2%/10%	Peptides	External gelling	90%	1328 µm	[146]
Cassava	Gum Arabic	50%/50%	Limonene	Spray drying	89%	1.5–14 µm	[61]
	Protein	2.6/1	Rice oil	Emulsion and spray drying	76.97%	*NR	[149]
	Gum Arabic	20%/80%	Palm oil	Emulsion and spray drying	96.53%	12–32 µm	[37]
Citron	Gum Arabic	75%/25%	Vitamin C	Spray drying	67–85%	9.8–10.9 µm	[58]
Corn	Calcium Alginate	2%/2%	Yerba Mate extract	Ionic gelation	9.2 mg/g	0.15–0.25 cm	[34]
Corn resistant starch	Whey Protein/Maltodextrin	3%/2%/25%	Vitamin D3	Spray drying	96.4%	140 nm	[112]
	Xanthan gum	9.5%/2.3%	Polyphenols	Spray drying	80%	10 µm	[18]
	Whey Protein	20% (W/V)	Folic acid	Electrospraying	52%	0.5 µm	[109]
	Maltodextrins	6.41%/23.08%	Olive oil	Emulsion and spray drying	33.44%	*NR	[41]
Hydrolysable Starch	Alginate	2%/3%	Caffeine	Emulsion and spray drying	78.86%	52.43 µm	[97]

Table 3 (continued)

Starch source	Second material	Ratio	Active principle	Methodology	Entrapment efficiency	Size	Reference
Starch modified with octenyl succinate anhydride	Soy Protein/Chitosan	NR	Vanillin	Emulsion and spray drying	51.91%	4.3 µm	[108]
Starch modified with octenyl succinate anhydride	Maltodextrins	20%	Linseed oil	Emulsion and spray drying	95.7%	2.11 µm	[94]
	Inulin	5 mg/5 mg	Tiger nut milk	Lyophilization	92.2%	388 nm	[106]
	Protein	4:1 w/w	Linseed oil	Emulsion and spray drying	81.86%	4.33 µm	[14]
	Gum Arabic	25:75	Nutmeg Oleoresin	Spray drying	95.38%	6–20 µm	[98]

**NR* no report

compounds that require specific target sites. This effect has been described in the context of cancer treatment drugs, as it offers the possibility to more easily penetrate through cell barriers and maintain retention at the site of action, in the case of nanocapsules [24]. Furthermore, as the bioavailability of a compound that is poorly soluble in water is limited by the dissolution rate, the use of nano-sized particles can improve the dissolution rate and, therefore, improve the bioavailability; especially for compounds that are adsorbed in a defined region of the gastrointestinal tract, considering oral administration [116]. These characteristics enable the production of microcapsules, nanocapsules, and capsules of millimeter size, as can be seen from Tables 1, 2, and 3.

Montoya et al. [150] encapsulated phenolic compounds using rice starch modified by enzymatic acylation as a wall material and reported the morphologies of the native and modified starches, as obtained by SEM. First, they showed that the granules presented a dodecahedral structure, typical of this type of starch; however, most of the granules had an irregular shape. In addition, in this research, the authors reported the maintenance of morphology of the granules after the encapsulation process, highlighting the tendency of starch to generate agglomeration—a characteristic of interest when assessing it for the oral administration of active ingredients. Encapsulation was achieved by spraying and emulsion. In addition, there was no evidence of any change in size or deformation of the granules, which was maintained in the range below 8 microns.

On the other hand, in 2019, Ahmad et al. [90] used starch from horse chestnut (HSC), water chestnut (WSC), and lotus stem (LSC) to prepare catechin nanocapsules. It was reported that all of the obtained nanocapsules presented different morphologies. In the HSC capsules, highly porous systems were achieved, with empty or full channels, in addition to showing an irregularity in the morphology of the encapsulates. In the WSC and LSC encapsulates, a possible gelatinization process and few granular structures were observed. The authors, when comparing the morphological characteristics of catechin hydrates, concluded that HSC, LSC, and WSC were successfully embedded with catechin particles; however, it was not clear to them, as the size and shape of the encapsulated catechin did not appear similar to that of native catechin after the nanocapsule preparation process.

It has been evidenced that the morphology of the capsules may be variable. Ramakrishnan, Adzahan, and Muhammad [12] observed that the morphology clearly indicated the type of wall material used to prepare tamarillo capsules, considering the significant differences in their microstructures. Spray-dried tamarillo powders with OSA had smoother and less-dented surfaces when compared with spray-dried powders produced using maltodextrins. Ordoñez and Herrera [61] observed spherical shapes after a spray-drying process, with sizes from 2 to 13 µm, when using cassava starch. A noticeable decrease in the encapsulated size and smooth uninterrupted surfaces was observed. In this sense, Gonçalves et al. [35] observed that particles obtained using OSA starch and lecithin showed a higher number of agglomerates composed of partially fused spheres; however, none of the dry particles obtained presented surface cracks. In this context, SEM and optical microscopy are the most-used methodologies to establish such characteristics [89, 151–154].

X-ray diffraction analysis

X-ray diffraction is useful in determining physicochemical features, as it can provide the crystalline formation for the nutrients or drugs encapsulated in nanoparticles or microparticles [3, 155]. After the coating process, due to intermolecular interactions, the nanoparticles prepared using starch and chitosan for entrapment polyphenols typically present amorphous structural characteristics [3]. This property is important when the release parameters are evaluated, as the liberation of volatile compounds encapsulated in amorphous matrices can occur when the amorphous matrix transforms from the vitreous state to the gum state, either through an increase in moisture content or at elevated temperatures, leading to a collapse of the system; for example, spray drying may induce changes in the crystallinity of the capsule, leading to amorphous states [61]. The presence of cassava starch could help to recover the crystalline structure of the system, as the presence of active compounds was not observed in the surface of the samples analyzed by scanning electron microscopy when limonene was encapsulated with starch and gum Arabic, in comparison with the gum Arabic samples [61].

In another investigation, Borrmann et al. [101] stored n-octenylsuccinate-derivatized starch-encapsulated passion fruit (*Passiflora*) juice systems for 25 days. The tests showed that both the starch used as wall material and the encapsulated systems had an amorphous structure. According to the authors, n-OSA starch shows a small tendency to crystallize, as represented by the peaks in the graph, which was reduced when the encapsulation process was carried out. This is noteworthy, as it has been reported that amorphous samples have a tendency to increase their hygroscopicity and absorb water during storage periods. The authors emphasized that this characteristic can be a point against the application of this system, as storage is impaired, given that the absorption of water increases the weight and degradation of vitamin C, thus collapsing the microstructure and triggering microbiological instability.

Glass transition temperature (T_g)

The T_g of a starch-containing material can be used to predict the product storage stability [121]. The glass transition temperature is the transition of an amorphous material from a hard and relatively brittle state into a molten or rubber-like state; this is important as, due to changes in the heat capacity at the phase transition temperature, a stepwise change in the heat flow generally occurs during the glass transition of amorphous materials [138]. The use of starch can cause a broad glass transition, due to its amorphous structure, and may be due to the overlapping of endothermic events at the gelation point of amylopectin [116]. In this sense, high-amylose corn starch has been shown to have lower transition temperatures, which may be caused by the presence of a lower crystalline region as well as gelatinization enthalpy. Furthermore, starches with long-branched chain length present higher gelatinization enthalpy, indicating that more energy is required to gelatinize long-chain length amylopectin [116, 120]. On the other hand, the presence of a maximum of the T_g suggests a glassy transition to rubbery states, indicating critical water activity for

which molecules cannot be stabilized within the starch structure. Such a structural transition probably leads, in turn, to changes in the effective diffusivity of water molecules within the starch matrix, as has been reported by Palma-Rodriguez et al. [121].

López-Córdoba et al. [34], by encapsulating yerba mate extract using a mixture of starch and alginate as the wall material, managed to establish a T_g of 55.9 °C a result they attributed to the effect of plasticization of the low molecular weight compounds present in the capsules, such as polyphenols, and the low water content of the matrix once the process was developed (aw = 0.53). In this sense, the authors argued that this T_g (higher than room temperature) is indicative of good stability, as the oxygen diffusion phenomenon is reduced in a glassy state, thus protecting the active ingredients from denaturation. In this sense, Xie et al. [40] had already reported a similar result when they encapsulated vitamin A using starch octenylsuccinate, showing a T_g of 56.355 °C; this T_g value is greater than the normal storage temperature (25 °C), indicating that, when stored at room temperature in a glassy state, the storage stability of vitamin A microcapsules is good. The authors related this T_g to the amorphous structure of the material used.

Zeta potential

Another important parameter is the zeta potential. High zeta potential values are responsible for electrostatic repulsion, which is desirable for the stability of the particles; in particular, through decreasing the van der Waals forces, which are responsible for their agglomeration, eventually resulting in larger particles [45]. If the zeta potential value of a colloidal solution is between + 18 mV and − 18 mV, it may not be stable and aggregation can occur [89, 102, 116, 156]. Electrostatically stabilized particle systems have a zeta potential above + 25 mV or below − 25 mV; between these values, they are not electrostatically stable, which would make it difficult to apply the encapsulates in intravenous administration. In addition, the negative charge of a biopolymer also promotes endocytosis-mediated cellular uptake by interaction with anionic sites in the cell membrane, thus enhancing intracellular distribution [28].

A negative zeta potential could be due to the interaction of starch and active compounds; for example, the interactions with catechin (phenolic compounds) imparted a negative charge when encapsulated using horse chestnut starch [45]. Similarly, after ultrasound-assisted rutin encapsulation using quinoa and maize starches, an increase in the number of hydroxyl groups on the surface of starch capsules has been observed. The ionization of hydroxyl groups on the surface increases the electronegative population of starch nanoparticles. Systems having zeta potential within these limits (i.e., above + 25 mV or below − 25 mV) are considered to be relatively stable [155].

On the other hand, Hasanvand et al. [116] observed an encapsulation efficiency value higher than 71%, due to the numerous interactions between vitamin D and the starch used as wall material leading to a lower zeta potential. The interaction of starch and vitamin D was likely related to hydrogen bonds formed between

vitamin D and the high-amylose corn starch, causing a decrease in the negative surface charge [116]. This interaction and the altered glycosidic bonds of starch reduced the effect of alpha-amylase, causing the formulation to have higher loading efficiency and enhancing slow-release parameters [116, 120]. In this sense, experiments have shown that sonication time had significant effects on size, zeta potential, particle distribution index, and loading efficiency; in particular, the results showed that decreasing temperature led to a reduction of particle size and increased zeta potential when using potato starch with higher amylopectin percentage as a wall material for vitamin D_3 encapsulation [120].

Ahmad et al. [45] observed that the zeta potential of all nano-encapsulated catechin samples took negative values, where the capsules using starch as wall material presented significantly higher negative zeta potential (− 20.1 mV) than capsules with other polymers, which had zeta potential around − 18.05 mV. In the same way, Hasanvand et al. [116] reported that the zeta potential values of all encapsulated samples were negative. The maximum negative value of the zeta potential was − 46.05 mV. High absolute values of zeta potentially lead to higher repulsion force of the particles and emulsion stability.

Conclusion

We conducted a literature review including a wide range of studies, which allowed us to conclude that the use of starches as a wall material has been widely considered in the context of the pharmaceutical, cosmetic, and food industries over the last decade. This rise in popularity of starches can be attributed to the diversity of compounds that they can trap, their low toxicity, and their nutritional contribution. Starches can be obtained from different sources, providing various characteristics that differentiate them and, thus, determining their affinity for the encapsulation of different pharmacological groups. Furthermore, starch modifications provide a new horizon for research, given the possibility of generating materials with specific properties relating to the compounds to be encapsulated.

The generation of encapsulates has focused on obtaining nanoparticles to provide increased absorption rates, improved bioavailability, passive accumulation in tissues, and slow release of the encapsulated principles, making this type of polymer a viable alternative to overcome the knowledge gaps that currently exist regarding the administration of active ingredients of natural origin.

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Declarations

Conflict of interest The authors report there are no competing interests to declare.

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