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REVIEW ARTICLE

# Trends and applications of hot-melt extrusion in the encapsulation of bioactive compounds for nutraceutical products

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## Abstract

The encapsulation of plant-derived bioactive compounds, such as polyphenols and flavonoids, constitutes an essential strategy to mitigate their physico-chemical instability against oxidative factors, light and pH variations in the gastrointestinal tract. While conventional techniques like spray drying are common, they often present limitations related to the use of organic solvents and discontinuous processing. Hot-melt extrusion (HME) has emerged as a sustainable, continuous and solvent-free technology; however, its viability for processing thermosensitive molecules has historically been questioned due to the thermal stress involved. Unlike previous reviews, this study integrates a bibliometric analysis with a critical technical evaluation specifically focused on strategies to preserve bioactivity under shear conditions. A systematic review of 148 studies published between 2014 and 2024, retrieved from Scopus, Web of Science (WoS) and PubMed, was conducted, evaluating the interaction between process parameters and polymeric matrices. The results reveal that HME facilitates the formation of stable amorphous solid dispersions through intermolecular interactions that prevent active recrystallisation. It was demonstrated that rigorous control of barrel temperature, screw speed and residence time allows for the processing of thermolabile compounds with minimal degradation, achieving significant improvements in solubility and bioavailability compared to pure crystalline forms. In conclusion, HME consolidates itself as a robust and efficient industrial alternative for the development of nutraceuticals. The future perspective of this technology lies in the research of new biopolymers with generally recognised as safe (GRAS) status and advanced plasticisers that optimise bioactive loading and allow for customised release profiles.

**Keywords:** antioxidant activity; bioactive compounds; encapsulation; hot-melt extrusion; solid dispersions

## Introduction

Microencapsulation of bioactive compounds is a fundamental technological strategy that consists of isolating an active substance (core) within a polymeric matrix or membrane to form particles of micrometric size (1). This technique emerged from the need to protect sensitive ingredients against adverse environmental factors and to control their release at specific sites within the organism (2, 3). Currently, its application is critical in sectors such as the pharmaceutical, food and nutraceutical industries, especially for the management of phytochemicals such as flavonoids and polyphenols. Although these compounds possess recognised antioxidant, anti-inflammatory and anticancer properties, they present marked instability against light, heat and oxygen. Furthermore, their bioavailability is drastically reduced in the gastrointestinal tract due to acidic pH and the presence of enzymes and bile salts (4–6).

To overcome these biological and physico-chemical limitations, diverse encapsulation systems have been developed to maximise loading efficiency and protection (7, 8). A crucial aspect of this design is the selection of coating materials, which must be biocompatible, biodegradable and, in the case of food applications, comply with safety regulations such as the generally recognised as safe (GRAS) status of the FDA. Different studies have highlighted that the choice between natural biopolymers (proteins, starches) and synthetic ones (Eudragit, PVP) determines mechanical stability, responsiveness to pH stimuli and the controlled release of the active compound (7, 9–12).

Among the various existing encapsulation technologies (spray drying, electrospraying, coacervation, fluidised bed), hot-melt extrusion (HME) has recently emerged as a robust and scalable alternative (13, 14). Unlike traditional methods, HME enables the formation of amorphous solid dispersions without the use of organic solvents, making it a green and continuous technology (15). However, the implementation of HME in nutraceuticals faces significant technical challenges, mainly related to thermal degradation. Given that the process involves melting and mechanical shear, there is a risk of degrading thermolabile compounds such as phenols. Therefore, precise control of parameters such as barrel temperature, screw speed and the use of plasticisers is vital to balance polymer processability with the preservation of bioactivity (16–18).

Unlike previous reviews that focus broadly on the technique, this article provides a comprehensive and up-to-date analysis of the specific application of HME for plant-derived bioactive compounds. This review examines recent advances up to 2024, correlating critical

processing conditions (temperature and shear) with encapsulation efficiency. In addition, the most effective polymers for nutraceutical matrices are discussed and relevant characterisation methodologies are consolidated, offering a clear perspective on the industrial viability and future trends of this technology.

## Materials and Methods

This systematic review was conducted following methodological guidelines for bibliographic reviews, focusing on the application of HME for bioactive compounds. The literature search was performed across the indexed databases Scopus, Web of Science (WoS) and PubMed, covering the period from January 2014 to May 2024 to ensure the currency of technological information.

### Search strategy and study selection

The systematic literature search was designed to identify relevant studies on the encapsulation of bioactive compounds via HME. Specific search equations were constructed using Boolean operators (AND, OR), adapted to the syntax of each database (Scopus, WoS and PubMed). The detailed search strings used for data retrieval are presented in Table 1.

Following the initial retrieval, a screening process was conducted based on predefined eligibility criteria to ensure the quality and relevance of the selected studies. The specific inclusion and exclusion parameters regarding time frame, document type, language and research focus are summarised in Table 2.

Inclusion criteria comprised original research focusing on the application of HME for the processing, encapsulation or stabilisation of natural bioactive compounds, plant extracts and nutraceutical ingredients.

Conversely, exclusion criteria were applied to studies exclusively utilising synthetic active pharmaceutical ingredients (APIs). While HME is well established for synthetic drugs, this exclusion was necessary to address the unique challenges associated with natural products, such as their multicomponent matrix complexity and high thermal sensitivity, which require distinct processing strategies compared to the well-documented behaviour of thermostable synthetic molecules.

### Bibliometric analysis and cluster construction

Trend analysis and network visualisation were conducted using VOSviewer software (version 1.6.19). A text mining technique based on the co-occurrence analysis of Author Keywords was applied. To ensure the semantic relevance of the clusters, a minimum occurrence threshold of four times per term was established, excluding generic words with no technical contribution.

The construction of the clusters was based on the association strength normalisation method. In the resulting maps, each node represents a keyword, with its size proportional to the frequency of occurrence and the thickness of the connecting lines indicating the intensity of the intellectual relationship between concepts. Items were grouped into distinct clusters differentiated by colours, allowing for the identification of main research lines: (i) process conditions, (ii) polymer types and (iii) applications in specific bioactives. Finally, manual extraction of quantitative data was performed on the articles included in each cluster to populate the results tables.

## Results

### Research trends and bibliometric network analysis

The quantitative analysis of the selected literature allowed for the identification of the intellectual structure of the field through keyword co-occurrence network maps. As observed in the general overview (Fig. 1), the network is segmented into six distinct thematic clusters, evidencing the multidisciplinarity of HME.

The first three clusters (red, green and dark blue) group historical and foundational research on the extrusion of conventional foods:

- Cluster 1 (Red): Focuses on active packaging technologies. Predominant nodes relate extrusion to the incorporation of antioxidants into plastic films to control antimicrobial activity and extend the shelf life of packaged products.
- Cluster 2 (Green): Addresses general functional properties. Terms converge here regarding the thermal effect of extrusion on antioxidant activity and the general health benefits of processed foods, without necessarily focusing on strict encapsulation.
- Cluster 3 (Dark blue): Is linked to the processing of starchy matrices (cereals, snacks and pasta). This cluster reflects studies on how enrichment with anthocyanin-rich sources affects the physico-chemical characteristics of mass-consumption products.

However, for the objectives of this review, critical relevance lies in the emerging clusters (5 and 6), which denote the technological leap towards advanced nutraceutical applications:

- Cluster 5 (Violet): Groups strategies for microencapsulation and biopolymers. This cluster reveals a strong correlation

**Table 1.** Search strategy and equations used in bibliographic databases

| Database | Keyword and term combinations for the search | Generated documents |
|---|---|---|
| Scopus | (ALL(“Hot melt Extrusion” OR extrusion AND processes OR hme AND “fruit powders” OR fruit AND “Antioxidant activity” AND solid AND dispersions OR “solid dispersions by hot-melt extrusion” AND nutraceutical AND compounds) AND ALL(Antioxidant)) AND PUBYEAR > 2013 AND PUBYEAR < 2025 | 164 |
| WoS | “Hot melt Extrusion” OR extrusion AND processes OR hme AND “fruit powders” OR fruit AND “Antioxidant activity” AND “solid dispersions by hot-melt extrusion” AND nutraceutical AND compounds AND Antioxidant Author co.citations AND Author Keywords: Antioxidant | 218 |
| PubMed | “Hot melt Extrusion” AND Antioxidants | 42 |

**Table 2.** Inclusion and exclusion criteria for the systematic review

| Parameter | Inclusion criteria | Exclusion criteria |
|---|---|---|
| Time frame | Articles published between January 2014 and May 2024 | Articles published before 2014 |
| Document type | Original research articles and technical reviews | Conference proceedings, book chapters, editorials and theses |
| Language | Full text available in English | Articles in languages other than English |
| Research focus | Studies reporting HME process conditions (temperature, screw speed) applied to natural bioactive compounds | Studies focused exclusively on synthetic plastics or pharmaceutical drugs without natural bioactive components |

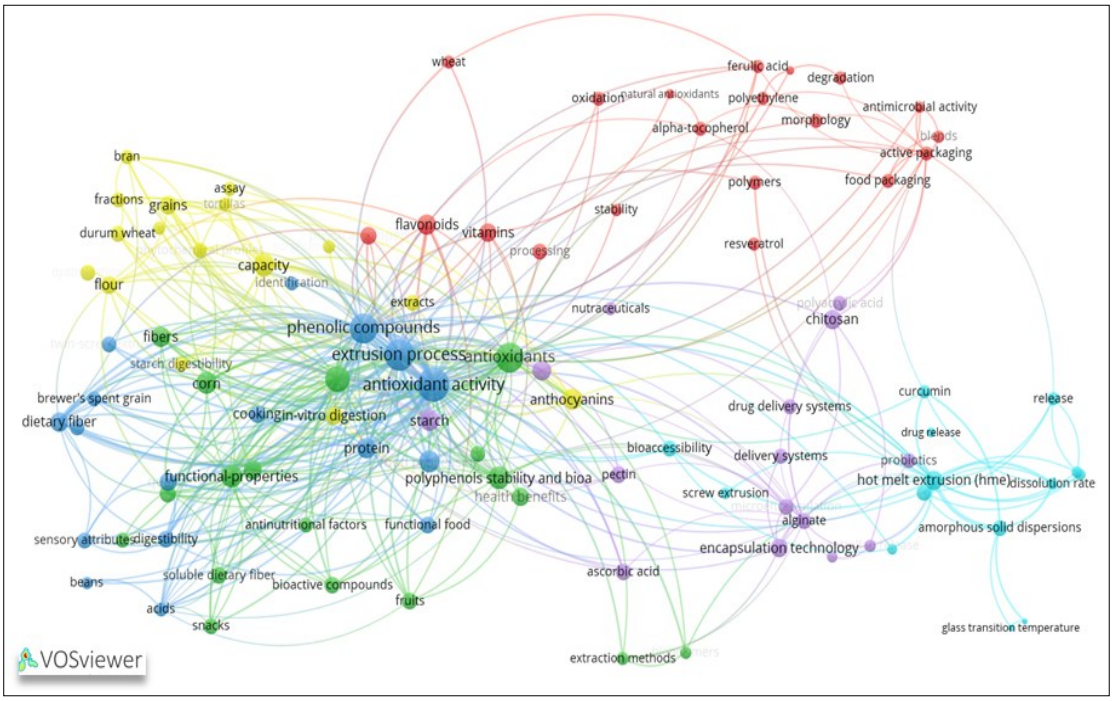


**Fig. 1.** Bibliometric network visualization of author keywords based on co-occurrence analysis. The colours represent distinct thematic clusters within the hot-melt extrusion research landscape.

between HME technology and the use of functional polymeric matrices such as alginate, pectin, chitosan and proteins. The literature within this group focuses on the design of controlled release systems, validating the use of biocompatible materials to protect the active load.

- Finally, cluster 6 (Light blue), detailed in Fig. 2, represents the core of innovation in HME for nutraceuticals. This cluster explicitly connects the term "hot-melt extrusion" with the formation of amorphous solid dispersions and improvements in biopharmaceutical parameters such as bioaccessibility and bioavailability. The appearance of specific nodes related to complex plant sources (e.g., blackberry, phenols) is noteworthy, confirming that current research has surpassed the stage of simple plastic extrusion to address the challenge of stabilising thermosensitive phytochemicals.

## Temporal evolution of the subject

The analysis of temporal progression (Fig. 3) confirms this technological transition. While research up to 2017 focused on conventional food production (cereals and feed), a strong emergence of terms associated with bioactive encapsulation is observed from 2019 onward. This trend coincides with the adoption of pharmaceutical extrusion techniques (fusion and molecular mixing), adapted to the food sector to resolve issues of low solubility and crystallinity in natural compounds.

Based on this bibliometric finding, the subsequent analysis of this review focuses primarily on studies belonging to clusters 5 and 6, whose technical data and operating conditions are consolidated and discussed in Table 3.

## Impact of critical processing parameters (temperature and shear) on bioactive behaviour

Hot-melt extrusion is fundamentally defined as a continuous manufacturing process capable of transforming a heterogeneous mixture of raw materials into a product of uniform density and geometry by forcing it through a die under controlled conditions. The feasibility of this operation relies intrinsically on the system's rheology; specifically, the melt viscosity must be modulated to ensure constant flow through the equipment (19) (Fig. 4).

Operationally, the procedure begins with the homogeneous mixing of the bioactive compound with functional excipients such as polymeric carriers, plasticisers, bulking agents, antioxidants and thermal lubricants either in a preliminary stage or within the feeder itself (20). Subsequently, through the application of thermal and mechanical energy in screw extruders (single or twin) or ram extruders, the components are melted and homogenised. Although processing below the glass transition temperature (Tg) is theoretically possible, the operation is conventionally conducted above the melting temperature (Tm) of the mixture to optimise rheological properties and facilitate the extrusion of the plastic mass, which is finally cooled and solidified for downstream processing (19).

In the reviewed studies, processing temperatures ranged widely between 70 °C and 200 °C, depending on the polymer's Tg and the thermal sensitivity of the active compound.

- Low-temperature processing (< 100 °C): Some studies employed mild conditions (80–100 °C) to preserve anthocyanins and whey proteins, which rapidly denature or degrade under excessive thermal stress (16, 21, 22).

- High-temperature processing (> 140 °C): In contrast, some studies processed quercetin and naringenin at temperatures of 150–200 °C (15, 23). Remarkably, chemical stability was maintained due to the solubilisation of the active compound within the molten matrix (e.g., Soluplus, PVP), which protected it from atmospheric oxygen (Table 3).

- Thermal degradation remains a primary concern for flavonoids; however, recent protocols have successfully mitigated this issue by controlling the operational window. Recent studies demonstrated that it is not necessary to reach the Tm of the bioactive compound to achieve a solid dispersion (24, 25). By maintaining extrusion temperatures substantially below the melting point of epicatechin, these studies preserved its chemical integrity while achieving a high loading capacity (50 %

**Table 3.** Comprehensive summary of process parameters, polymeric matrices and performance outcomes in the HME encapsulation of plant-derived bioactives (2014–2024)

| Plant Source / bioactive compound | Polymer matrix (Blend) | Conditions (Temp / speed) | Key findings and performance (quantitative improvement) | References |
|---|---|---|---|---|
| **I. Polyphenols, flavonoids and anthocyanins** | | | | |
| Quercetin/ pure quercetin | HPMC, Poloxamer 188, Soluplus, PEG 600 | 150–170 °C/ 100 rpm | Improved dissolution rate and oral bioavailability compared to pure crystalline quercetin. | (15) |
| Mulberry leaf (*Morus alba*) / isoquercetin, rutin | Whey protein isolate, soy lecithin, Vitamins C and E | 100 °C/ 50 rpm | Phenolic content increased from 22,12 to 31,14 mg GAE/g. Improved solubility and functional bioactivity. | (16) |
| Mulberry leaves (varieties)/ rutin, isoquercetin | Whey protein isolate, lecithin, vitamin C | 100 °C/ 50 rpm | Significant increase in biological activity. Optimized variety reached 31,14 mg GAE/g total phenolics. | (21) |
| Mulberry (*Morus alba*) / anthocyanins | Protein isolate, Sodium alginate, Poloxamer 188 | 80–100 °C/ 150 rpm | Hydrolysis of bound phenolics increased free content: Total phenolics reached 1109 mg/100g; Anthocyanins 247 mg/100g. | (22) |
| Citrus (orange/grapefruit)/ naringenin | PVP, polyglycerol O-50D | 180–200 °C/ 100 rpm | Aqueous solubility increased to ~15 mg/mL (200-fold higher than crystalline naringenin). | (23) |
| Epicatechin | Ethylcellulose, Eudragit L100, povidone | 160 °C/ 70–100 rpm | Enhanced solubility and effective taste masking suitable for chocolate matrices. | (25) |
| *Angelica gigas* Nakai / nanocomposites | HPMC (HP55, CN40H), sodium alginate | 70–100 °C/ 150 rpm | Extruded nanocomposites showed improved solubility. Phenolics: 2187 mg/100 g; Flavonoids: 165 mg/100 g. | (26) |
| Kenaf seeds / polyphenols | Seed flour, lecithin, whey concentrate | 80–120 °C/ 200 rpm | Enhanced extraction via shear: Phenolics increased from 1370 to 3342 mg/100g. | (27) |
| Scutellaria root/ baicalin | Chitosan, HPMC (25:75) | 150 °C/ 150 rpm | Tablets demonstrated controlled release of baicalin with good mucoadhesive properties. | (29) |
| *Angelica gigas* Nakai / phenols, flavonoids | HPMC (5 %), acetic acid (plasticizer) | 80–120 °C/ 220 rpm | Solubility improved by 65,5 %. Total phenolics reached 2832 mg/100g with enhanced bioaccessibility. | (30) |
| Mulberry (*Morus alba*)/ anthocyanins | Citric acid, sodium alginate | 80–100 °C/ 150 rpm | Enhanced water solubility and release rate. Anthocyanin content preserved at 331 mg/100g. | (31) |
| Acacia honey and pollen/ rutin, gallic acid | Honey/pollen blend (25:75 / 50:50) | 85–100 °C/ 180 rpm | Higher antioxidant activity. Combined phenols + flavonoids reached 25,05 mg QE/ g. | (33) |
| Quercetin (in carnauba wax) | Carnauba wax, Shellac, Zein (70/30) | 90 °C/ 100 rpm | Effective masking of bitter taste and achieved slow-release characteristics. | (39) |
| *Angelica gigas* Nakai polyphenols | Polymeric matrix | (Not reported) | Powdered products exhibited anti-inflammatory and antioxidant properties (65 % inhibition). | (47) |
| *Angelica gigas* Nakai solid extract (AGN) | Ascorbyl palmitate, lecithin | 100 °C/ 150 rpm | Successful development of solid dispersions viable for oral administration with medicinal potential. | (48) |
| *Sesbania aculeate* seeds | Self-derived food matrix | 150 °C/ 150 rpm | Improvement in techno-functional properties. Phenolics increased to 28.48 mg GAE/g. | (49) |
| Soy (*Glycine max*)/ isoflavones (genistein) | Soy (95 %), HPMC (5 %) | 80–130 °C/ 80 rpm | Increased solubility and total phenolics (from 4.27 to 9.82 mg/g) and antioxidant capacity. | (50) |
| *Polygonum cuspidatum*/ resveratrol (50 %) | HPMCAS, Eudragit EPO, Soluplus | 140 °C/ 40 rpm | Solid dispersions significantly improved solubility and dissolution rate of resveratrol. | (38) |
| **II. Curcuminoids (Turmeric)** | | | | |
| Turmeric (*Curcuma longa*) /curcumin | Soluplus® | 130–140 °C/ 75 rpm | Maintained formulation integrity, prevented recrystallization and improved release profile. | (17) |
| Turmeric (*Curcuma longa*)/ curcumin | Eudragit RSPO, Eudragit RLPO | 145 °C/ 80 rpm | Prolonged half-life, stable blood concentrations and improved bioavailability. | (51) |
| Turmeric (*Curcuma longa*)/ curcumin | Eudragit, compritol, ethylcellulose | 145 °C/ 80 rpm | Orthogonal experimental design allowed optimization of sustained-release solid dispersion. | (52) |
| Turmeric and black pepper/ curcumin, piperine | Kollidon VA64 | 150 °C/ 100 rpm | Significant increase in water absorption, flavonoid content and antioxidant potential. | (17, 27, 53) |
| **III. Others (alkaloids, cannabinoids, vitamins)** | | | | |
| Cocoa (*Theobroma cacao*) / theobromine | Soluplus, Plasdone S-630, Eudragit | 150–185 °C/ 50–100 rpm | Dissolution efficiency of 87 %, flowability of 88 % and contact angle of 47 ° (better wettability). | (10) |
| Ascorbic acid / vitamin C | Maltodextrin, Gum arabic, trehalose | 105–120 °C/ 60 rpm | Effective stabilization of vitamin C; matrices suitable for other sensitive bioactives. | (54) |
| Hemp leaves (*Cannabis sativa*)/ cannabidiol (CBD) | Ascorbyl palmitate, vitamin C | 80–120 °C/ 100 rpm | Massive increase in available CBD: from 736 to 2800 µg/g. Efficient decarboxylation and THC reduction. | (55) |

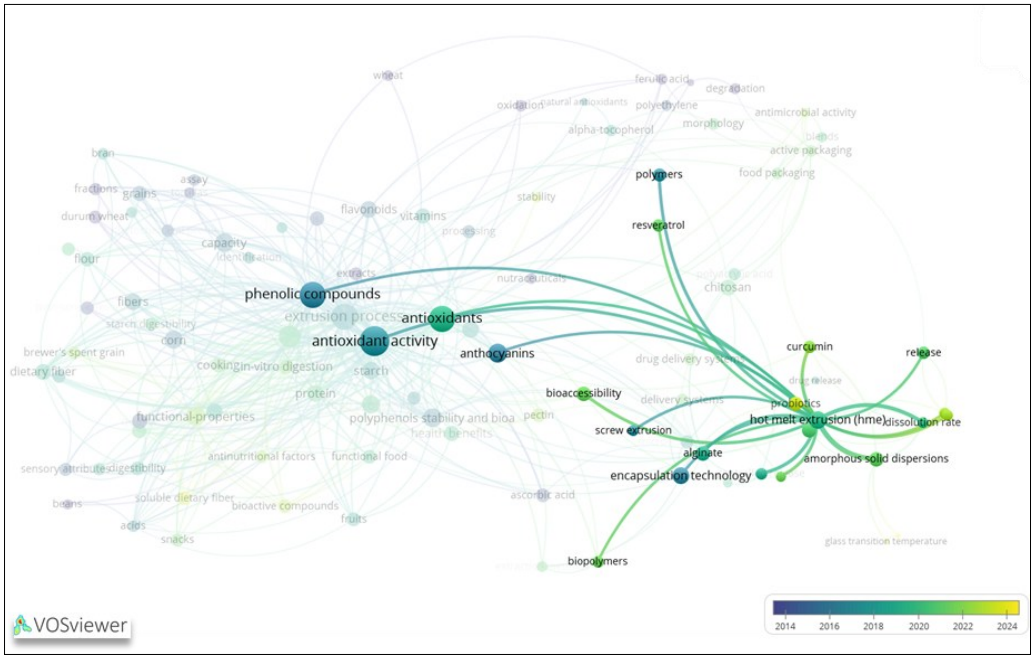


**Fig. 2.** Detailed visualisation of cluster 6 (blue), highlighting the HME node and its strong interconnections with bioactive compounds, solubility and bioavailability parameters.

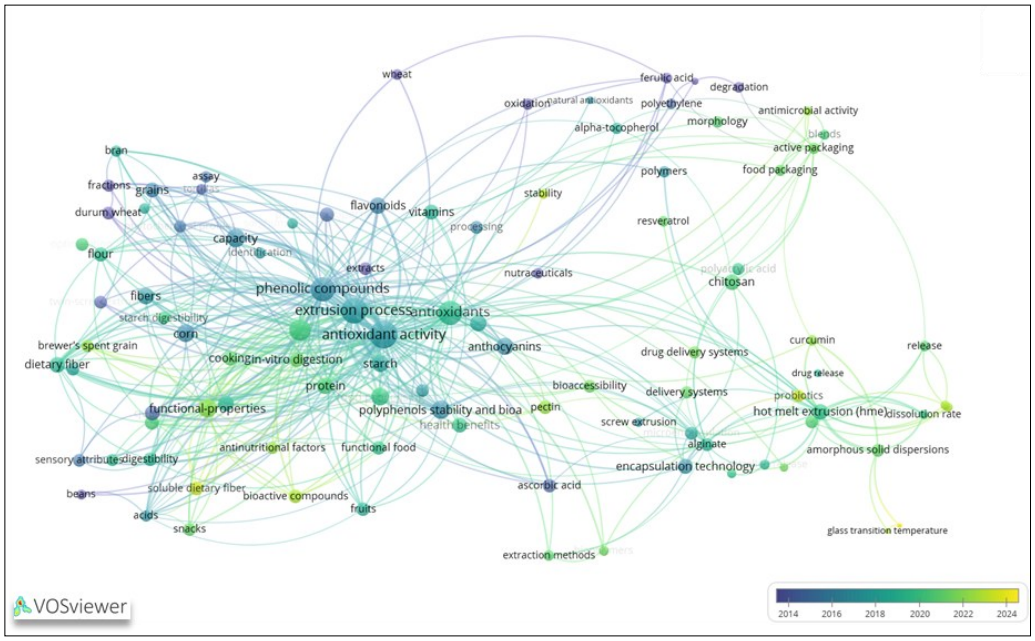


**Fig. 3.** Overlay visualisation of the keyword network illustrating the temporal evolution of research topics (2014–2024). Yellow nodes indicate emerging trends associated with encapsulation and nutraceuticals.

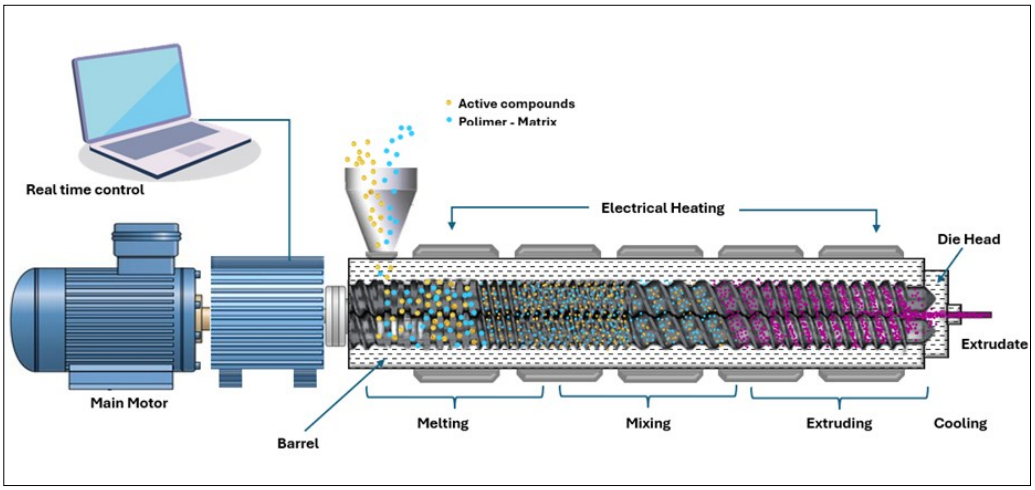


**Fig. 4.** Schematic representation of the HME process for encapsulating active compounds. The diagram illustrates key zones of melting, mixing and extrusion, highlighting the interaction between the polymer matrix and active compounds, driven by the main motor and controlled in real-time.

w/w), proving that the polymer plasticisation is sufficient to solubilise the crystal lattice of the bioactive.

- A counterintuitive finding in the extrusion of plant matrices is the apparent increase in bioactive concentration post-processing. Other studies demonstrated this phenomenon during the development of *Angelica gigas* Nakai (AGN) nanocomposites via HME (26). Their results indicated a significant increase in detectable total phenolic and flavonoid content (up to fivefold in HPMC-optimised formulations) compared to the raw, non-extruded powder. The authors attributed this enhancement to two simultaneous mechanisms induced by high shear stress: particle nanonisation (size reduction to 323 nm) and disruption of plant cell walls, thereby releasing phenolic compounds previously bound to the insoluble matrix and inaccessible via conventional extraction methods.

Screw speed (rpm) dictates residence time and specific mechanical energy. With the aim of optimising bioactive recovery, various studies have evaluated the impact of screw speed. On one hand, one study applied high shear stress (200 rpm) to kenaf seeds, demonstrating that this mechanical energy is indispensable for disrupting cell walls and releasing bound polyphenols (27). In contrast, another study opted for a moderate speed of 75 rpm to process curcumin, achieving a balance between efficient distributive mixing and the prevention of degradation caused by excessive friction (17). Likewise, analyses based on rheological behaviour indicate that the use of high-viscosity polymers (such as chitosan/ HPMC) demands precise torque control to minimise residence time and prevent material blockage within the barrel, underscoring the importance of characterising the material's thermal profile (28, 29).

The incorporation of specific plasticisers is a critical strategy to overcome the high melt viscosity of cellulosic polymers such as HPMC and improve their processability. For example, study demonstrated that the addition of acetic acid (0.5–1.0 M) as a plasticiser significantly reduced the Tg of the *Angelica gigas* Nakai (AGN)/ HPMC blend (30). By disrupting the intermolecular hydrogen bonds within the polymer chains, the plasticiser lowered the Tg from 68.5 °C (raw powder) to approximately 43 °C in the extrudates. This thermal depression is vital, as it expands the processing window, allowing the extrusion of heat-sensitive nutraceuticals at temperatures well below their degradation point.

## Bioactive behaviour and encapsulation efficiency

The physico-chemical nature of the compound dictates the formulation strategy. The review identified two main enhancement mechanisms via HME:

- The formation of amorphous solid dispersions is critical for enhancing the bioavailability of lipophilic crystalline compounds such as curcumin and quercetin. This process relies on amorphisation, which involves the transition from an ordered crystalline structure to a high-energy disordered state, thereby facilitating solubility. However, the main thermodynamic challenge lies in preventing recrystallisation, a phenomenon in which molecules tend to spontaneously reorganise back into their stable lattice form, negating solubility gains. In this context, studies demonstrated that molecular dispersion of curcumin within a Soluplus matrix effectively inhibits reversion to the crystalline state, maintaining stability for three months at 40 °C and resulting in a drastic improvement in its dissolution rate (17). In the case of epicatechin, one study reported a complete reduction in crystallinity, confirmed by DSC and XRD, after extrusion with GRAS polymers (24). This amorphisation resulted in a controlled release profile and significantly improved the water solubility of the flavonoid compared to its pure crystalline form.
- Extrusion-assisted extraction: For complex matrices such as plant extracts, HME acts as an intensified extraction process. Several recent studies reported an increase in mulberry polyphenol content from 615 to 1109 mg/100 g after extrusion (31). Similarly, a massive increase in kenaf polyphenols was observed rising from 1370 to 3342 mg/100 g (27). This phenomenon is attributed to the rupture of covalent bonds between phenolic compounds and dietary fibre, induced by the combined effects of temperature and shear.
- Beyond stability and solubility, HME also serves as an effective technique for organoleptic improvement in functional foods. It has been reported that dispersing epicatechin within a polymeric matrix effectively masks its inherent bitterness, making the formulation more palatable for oral consumption without the need for additional sweeteners (24).
- The ultimate goal of HME processing is to enhance the functional performance of the encapsulated bioactive compounds. Studies reported that the extrusion of AGN with acidified HPMC not only preserved the active ingredients but also significantly enhanced their extraction yield (30). The bioaccessibility of key coumarins, such as decursin and decursinol angelate, increased by up to 65.5 % compared to the control. Furthermore, the extrudates exhibited superior antioxidant activity (DPPH assay) and functionality, attributed to the effective amorphisation of the crystalline drug and the increased specific surface area resulting from the nanonisation process.
- Finally, in the case of thermolabile antioxidants, formulations incorporating protective matrices (such as the honey-pollen blends or whey proteins) have allowed for the retention of over 80 % of antioxidant capacity (DPPH/FRAP) after processing, validating HME as a viable technology for sensitive functional ingredients (32, 33).

## Comparison and performance

Polymer selection implies a technical trade-off. Synthetic polymers (PVP, Soluplus) offer greater control over release and physical stability due to their high Tg, but often require process temperatures > 140 °C. Conversely, natural polymers are biocompatible but require plasticisers (such as vitamin C, PEG, or citric acid, as observed in mulberry and hemp studies) to reduce viscosity and allow processing at < 100 °C, thus protecting the thermolabile payload. The current trend points towards hybrid systems, such as combinations of chitosan and HPMC, to leverage the best of both worlds, including processability and bio adhesion (28).

Optimising the interplay between barrel temperature and screw configuration is essential for achieving effective particle size reduction. In one study, a twin-screw extruder was operated with a specific temperature profile ranging from 100–140 °C across the zones, combined with a screw speed of 150 rpm (30). Under these conditions, the high shear stress exerted by the screws, facilitated by the softening effect of the HPMC/acetic acid matrix, successfully converted the coarse plant material into nanocomposites with an

average particle size of 341 nm. This process demonstrates that controlled thermal energy, when coupled with adequate shear, promotes the formation of amorphous solid dispersions without compromising matrix integrity.

Studies have shown that the use of hydrophobic polymers, such as Soluplus, improves the retention of active compounds by reducing recrystallisation and maintaining formulation integrity. For example, a previous study achieved improved encapsulation efficiency of curcumin by optimising the extrusion temperature and screw speed (17). Other studies have reported an increase in phenolic content in leaves processed by HME, suggesting that high shear forces and thermal conditions can release active compounds and improve their solubility (21). These factors, combined with a suitable polymer matrix design, are essential for maximising encapsulation efficiency (Table 3).

## Polymeric matrices and stabilisation mechanisms

The polymers utilised in the reviewed studies are primarily classified into synthetic and natural categories. The selection of the matrix is not trivial, as it determines the molecular interaction (e.g., hydrogen bonding) with the bioactive compound, directly affecting encapsulation efficiency, the Tg of the system and final bioaccessibility.

Beyond the physical dispersion of bioactive compounds, the stability of amorphous solid dispersions generated by HME is fundamentally governed by specific intermolecular interactions and thermodynamic properties. Previous research has demonstrated that hydrogen bonding between the hydroxyl groups of quercetin/resveratrol and the carbonyl groups of polymeric carriers (such as Soluplus) is a key factor in inhibiting recrystallisation (34).

Furthermore, the physical stability of these systems can be quantitatively predicted using the Gordon-Taylor equation, which estimates the theoretical Tg of the binary mixture. As thoroughly analysed, small bioactive molecules often act as plasticisers, reducing the overall Tg of the polymer matrix (35). Consequently, calculating the specific Tg mix is critical to ensure that storage conditions remain significantly below this value (typically Tg 50 °C), thereby restricting molecular mobility and preventing phase separation and devitrification (36, 37).

## Synthetic polymers and amorphous solid dispersions

Among the most widely used materials are Eudragit (RSPO, RLPO, L100-55), HPMC, PVP, Soluplus and poloxamers. These polymers exhibit high thermal stability and a superior capacity to inhibit crystal nucleation, thereby maintaining bioactive compounds in a high-energy amorphous state.

For instance, a significant improvement in the dissolution rate of quercetin was reported by employing a complex matrix of HPMC, poloxamer 188, Soluplus and PEG 600 (150–170 °C) (15). The underlying mechanism involves the formation of polymeric micelles that solubilise the hydrophobic drug. Similarly, researchers achieved a notable increase in resveratrol solubility using HPMCAS and Eudragit EPO (38). A standout case is the study on naringenin in citrus, where the use of PVP and polyglycerol at high temperatures (180–200 °C) increased aqueous solubility up to 200-fold compared with the pure crystalline form (23) (Table 3).

The amphiphilic copolymer Soluplus has proven particularly effective for curcuminoids and alkaloids. For example, researchers developed a solid dispersion at 130–140 °C that prevented recrystallisation for up to 3 months (17). Likewise, Soluplus-based formulations applied to cocoa extract achieved not only an 87 % dissolution efficiency for theobromine but also improved powder wettability (contact angle reduced to 47 °), which is critical for rehydration in beverages (10).

## Natural polymers and lipid matrices

Although historically limited by thermal degradation, biopolymers (proteins and polysaccharides) and lipids are gaining traction due to increasing demand for "clean labels" formulations. Several recent studies have employed whey protein isolate, lecithin and alginate to encapsulate anthocyanins from *Morus alba* (16, 22). Similarly, it was demonstrated that, in kenaf seeds, shear at 200 rpm using flour and whey matrices increased polyphenol content from 1370 to 3342 mg/100 g (27) (Table 3). In addition to improving solubility, natural polymers are also employed for taste masking. A relevant study utilised carnauba wax and zein at 90 °C to encapsulate quercetin, successfully masking its characteristic bitter taste through the formation of a hydrophobic lipid matrix, thereby enabling the development of more palatable functional ingredients (39).

Regarding natural matrices, several studies have provided compelling evidence for the suitability of starch as a GRAS carrier in extrusion processes (40). A study on chestnut starch demonstrated a synergistic effect, whereby the high shear forces during extrusion facilitate strong hydrogen bonding interactions between polyphenols (such as catechins) and starch chains. Interestingly, this interaction not only stabilises the bioactive compounds but also significantly inhibits starch retrogradation (recrystallisation) during long-term storage, thereby addressing one of the main technological limitations associated with the use of biopolymers in HME (41).

## Regulatory considerations: GRAS status and safety profile of polymers

The selection of the polymeric matrix for the development of nutraceuticals via HME should not be based solely on thermal processability and amorphisation capacity, but must also prioritise compliance with food safety regulations, specifically FDA guidelines. According to the Code of Federal Regulations (CFR) (Title 21, Chapter I, Subchapter B, Part 170), the eligibility of a substance as GRAS is determined either through scientific procedures or, for substances used in food prior to 1958, through experience based on common use (42).

In this context, natural biopolymers (starches, pectins and proteins) present a significant regulatory advantage, as most possess inherent GRAS status due to their long history of safe human consumption. This facilitates their incorporation into "clean label" formulations and reduces the regulatory burden associated with FDA notification. Conversely, the use of synthetic polymers (e.g., methacrylate derivatives or PVP) in nutraceuticals requires more rigorous evaluation. Although these materials offer superior rheological properties for extrusion, their inclusion is contingent upon being specifically listed as approved food additives or having a GRAS notification supported by robust scientific evidence of toxicological safety (43). As analysed in reviews of the GRAS notification pilot program, the current industry trend leans towards voluntary submission of notifications to the FDA to ensure transparency and safety, particularly when employing novel excipients or chemically modified polymers to enhance bioactive release (44). Therefore, for nutraceutical applications, it is critical to verify that the selected synthetic polymer grade is suitable for oral consumption (food grade) and not intended solely for pharmaceutical use (45, 46).

## Conclusion and Future Perspectives

This systematic review and bibliometric analysis confirm that HME has evolved from a conventional food-processing technique to a sophisticated platform for the encapsulation of bioactive compounds, successfully overcoming the poor water solubility of phytochemicals, with encapsulation efficiencies often exceeding 90 %. A comprehensive analysis of recent studies (2014–2024) provides conclusive evidence that the formation of stable amorphous solid dispersions relies heavily on the correct selection of polymeric matrices, particularly amphiphilic copolymers such as Soluplus and hydrophilic carriers (HPMC, PVP), which prevent recrystallisation through specific intermolecular interactions. Furthermore, contrary to historical concerns regarding thermal degradation, this review highlights that optimisation of residence time and shear stress is more critical than temperature reduction alone, thereby enabling the effective processing of thermolabile flavonoids and anthocyanins.

Looking ahead, the trajectory of HME in the nutraceutical industry is shifting towards "green extrusion" and advanced manufacturing. Future research trends are increasingly focused on identifying natural GRAS excipients such as modified starches and proteins to replace synthetic polymers and meet "clean label" consumer demands. Additionally, the technological integration of HME with 3D printing (fused deposition modeling) and co-extrusion represents a frontier for personalised nutrition, enabling the design of multi-layered dosage forms with tailored release kinetics. In summary, HME stands as a mature, solvent-free and scalable technology ready to drive the next generation of high-performance functional foods.

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## Authors' contributions

VHRB conceived the overall research idea, designed the study framework, conducted the bibliometric analysis and interpreted the results. VHRB, JFSD and JLHC jointly coordinated the preparation of the manuscript. JFSD designed the study framework and contributed to the refinement of the methodology. The review and editing process was carried out by VHRB, JFSD and JLHC. All authors read and approved the final version of the manuscript.

## Compliance with ethical standards

**Conflict of interest:** Authors do not have any conflict of interest to declare.

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