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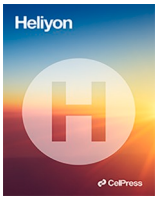

Review article

Emulsification and stabilisation technologies used for the inclusion of lipophilic functional ingredients in food systems

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ABSTRACT

Food industry is increasingly using functional ingredients to improve the food product quality. Lipid-containing functional ingredients are important sources of nutrients. This review examines the current state of emulsification and stabilisation technologies for incorporating lipophilic functional ingredients into food systems. Lipophilic functional ingredients, such as omega-3 fatty acids, carotenoids, and fat-soluble vitamins, offer numerous health benefits but present challenges due to their limited solubility in water-based food matrices. Emulsification techniques enable the dispersion of these ingredients in aqueous environments, facilitating their inclusion in a variety of food products. This review highlights recent advances in food emulsion formulation, emulsification methods and stabilisation techniques which, together, improve the stability and bioavailability of lipophilic compounds. The role of various emulsifiers, stabilizers, and encapsulation materials in enhancing the functionality of these ingredients is also explored. Furthermore, the review discusses different stabilisation techniques which can yield in emulsion in a solid or liquid state. By providing a comprehensive overview of current technologies, this review aims to guide future research and application in the development of functional foods enriched with lipophilic ingredients.

1. Introduction

Global population is estimated to reach approximately 8.5 billion by 2030, according to the [1]. However, the expansion of agricultural land may not keep pace with such rapid growth. As daily food requirements increase, meeting the rising demand has become a challenge. A potential solution to this dilemma is the development of healthy and more nutritionally dense food products. This approach allows individuals to fulfil their nutritional needs while consuming smaller portions of food, as proposed by Santos-Buelga et al., in 2019 [2]. These foods are commonly referred to as functional foods. Functional foods represent a category of food that has the potential to enhance various physiological aspects of human well-being. They are designed not only to provide essential nutrients but also to positively impact specific functions within the human body. Functional foods play a crucial role in improving human vitality by promoting the overall health and preventing or managing diverse metabolic disorders and illnesses [3,4].

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Functional ingredients that confer specific benefits to foods are distinct from conventional food additives. These ingredients, which are extracted from bioresources, retain their inherent properties after extraction [5]. Lipophilic functional ingredients such as vitamins, essential oils, and fatty acids play a crucial role in human health. For instance, provitamin A carotenoids like beta-carotene convert in the body to retinal and retinoic acid, supporting vision, immune function, and growth [6,7]. Consuming polyunsaturated fatty acids (PUFA) offers antiarrhythmic effects, prevents platelet aggregation, relaxes arterial smooth muscle, and reduces plasma triglycerides and blood pressure. Increased intake of omega-3 fatty acids may also protect against chronic and inflammatory disorders like rheumatoid arthritis and asthma [8–10]. Vitamin E is a fat-soluble antioxidant that prevents the production of reactive oxygen species during fat oxidation, aiding muscle preservation and brain health. It protects fatty acids in lipoproteins and cell membranes, reducing oxidative damage to DNA and RNA, and boosts the immune system, potentially lowering the risk of cancer and arthritis [11–15].

Incorporating these compounds directly into food systems is challenging because of various factors, such as limited water solubility, susceptibility to chemical or enzymatic degradation, and incompatibility with the surrounding food matrix, which collectively hinder their optimal oral bioavailability [16]. Consequently, protection of lipophilic functional ingredients is essential. Protecting of lipophilic functional ingredients via emulsification plays a vital role in increasing the oral bioavailability and providing stability and protection to these compounds [16–18].

Emulsions are colloidal systems made up of at least two immiscible liquids in which one of them if finely dispersed in the other with the help of surfactants and homogenization technologies. The dispersed liquid is known as the dispersed phase, and the other liquid is known as the continuous phase [19]. Oil and water are the most common liquids used to prepare food-grade emulsions; therefore, there are two basic types of emulsions: oil-in-water emulsions (O/W), where oil is the dispersed phase, and water-in-oil emulsions (W/O), where water is the dispersed phase. O/W and W/O emulsions are the most common types of colloidal dispersions used for the encapsulation and controlled release of lipid components such as lipophilic vitamins and essential fatty acids [20,21].

Such systems are thermodynamically unstable because the contact between water and oil is unfavourable; therefore, they break out over time, affecting the shelf-life and protective capacity of the emulsions [18,21]. Several mechanisms can cause the destabilization of an emulsion, among which creaming, flocculation, and coalescence are the most common [22]. Different stabilisation techniques such as drying, bead encapsulation, or mixtures of different emulsifiers can be used to guarantee the stability of emulsions and prevent their separation over time. Spray drying is one of the most commonly used techniques for the stabilisation of emulsions [23–25] however, several emerging and traditional technologies are being studied as alternatives to stabilise these emulsions and confer additional protection against adverse conditions in the gastrointestinal tract.

The study of the fate of emulsions in the gastrointestinal tract is important because food digestion is a complex and multi-stage process that has a direct link between the food ingested and the health or disease of a person [6,18,19]. During human digestion, two main processes occur simultaneously: (i) mechanical transformations that reduce the size of food particles and (ii) enzymatic transformations, where macromolecules are hydrolysed into smaller constituents that have the potential to interact and be absorbed by the organism to reach the bloodstream and become bio-accessible [19,21,26,27]. The digestibility of the fundamental components of the emulsions was measured separately to observe the behaviour of the compounds and verify their digestibility. It is also important to evaluate the behaviour of emulsions after passage through the gastrointestinal tract to assess their protective effect on the compound of interest. A study on the behaviour of the stabilized emulsion is also required to determine whether the stabilisation process can provide an additional protective effect to the emulsions. Finally, analysis of the behaviour of the emulsion in the final matrix is essential to determine whether it will reach the final consumer or if there is some interaction between the functional ingredient and food.

Publications on the encapsulation and stabilisation of food-grade functional oils via emulsification or other technologies and the study of their functional activities have increased since 2015. A total of 157 research articles on this topic were published between

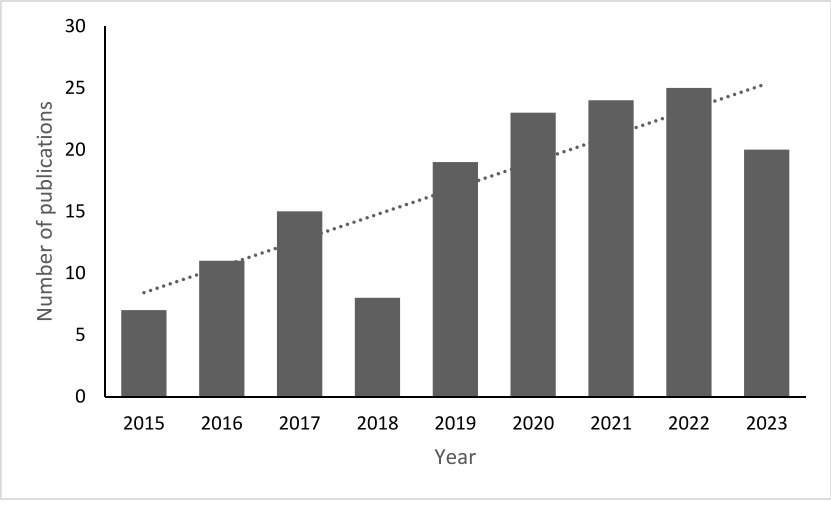

Fig. 1. Publications on encapsulation and emulsification of food grade functional lipophilic ingredients.

2015 and 2023, with the number increasing in recent years, showing the growing research interest in this area (Fig. 1).

The process of developing, stabilising, and protecting lipophilic functional compounds involves several steps Fig. 2. This figure also describes the organisation of topics presented in this review. The development of functional emulsions begins with knowledge of the food matrix in which the encapsulated functional ingredients are included. The type of emulsion to be prepared depends on the characteristics of the food system and the raw materials required for the preparation and stabilisation of the emulsions. Once the formulation is established, an emulsification process is performed in which various emulsification techniques are used to generate stable food systems. However, in many cases, these systems are not stable, and it is necessary to use stabilisation technologies to increase the shelf-life and achieve greater protection of lipophilic functional compounds. The selection of these technologies is important for product development. The functionally encapsulated and stable lipophilic compound is then included in the food system to reach the final consumer. Finally, evaluation of the behaviour of lipophilic functional ingredients during their movement through the gastrointestinal tract at each stage of the encapsulation process is important to ensure the bootability of functional ingredients susceptible to degradation. The aim of this review was to provide insights into the emulsification and stabilisation technologies used for the inclusion of lipophilic functional ingredients in food systems. This review also discusses various raw materials, formulations of emulsions, and emulsification and encapsulation technologies for the release of compounds during passage through the gastrointestinal tract.

For the preparation of this review, articles, including both original research and review papers, were sourced from the ScienceDirect database. The selected articles contained the keywords “emulsion,” “encapsulation,” “food,” “oil,” and “functional” in their titles, keywords, and abstracts and the time period ranged between 2015 and 2023.

2. Food emulsion formulation

2.1. Emulsions

An emulsion is a fine dispersion system in which a discontinuous phase is homogeneously distributed as small spherical droplets in a continuous phase, with the emulsion droplets stabilized by an amphiphilic surface-active surfactant [16,19]. Emulsions can be classified according to the spatial organisation of their phases. A system consisting of oil droplets dispersed in an aqueous phase is called an oil-in-water emulsion (O/W), and a system in which water is dispersed in an oil phase is called a water-in-oil emulsion (W/O) [17,21,28]. The choice of emulsion system for the protection of functional ingredients depends on the nature of the compound to be protected and the matrix in which the emulsion is to be incorporated.

The oil-in-water emulsion consisted of small oil droplets dispersed in an aqueous medium surrounded by a thin interfacial layer consisting of emulsifier molecules. The advantages of these systems include their relative ease of preparation and low cost [29]. Regarding the protection of lipophilic functional ingredients, oil-in-water (O/W) emulsions are very common; however, O/W

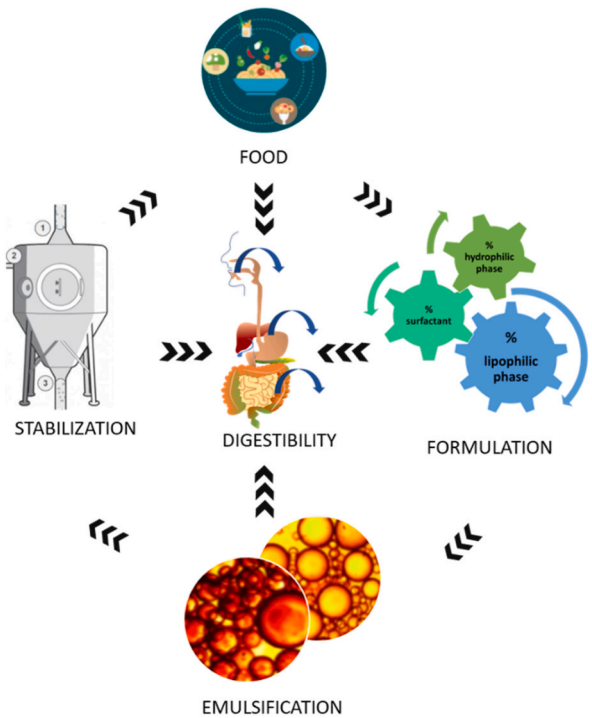

Fig. 2. Steps for the development and evaluation of stable functional emulsions.

Table 1
Main research articles on the use of functional lipophilic ingredients as active ingredients for the formulation of emulsions.

Lipophilic functional ingredient	Disperse phase	Continuous phase	Main findings	Emulsification technology	Reference
Curcumin	Rapeseed oil	Chitosan (CS) in acetic acid solution and gum Arabic (GA) in ultrapure water	Curcumin emulsions showed a half-life of 144 h, and approximately 50 % of curcumin was degraded. Pickering emulsion stabilized by CS-GA nanoparticles significantly improved the bioavailability of curcumin.	Pickering emulsion. High-speed homogenizer at 12,000 rpm for 8 min	(Han et al., 2020) [91]
	Medium chain triglyceride (Miglyol 812)	Octenyl succinic anhydride (OSA)-modified quinoa starch in phosphate buffer solution	Pickering emulsion preserved up to 95.3 % of curcumin after simulated oral digestion and 86.2 % after simulated gastric digestion, reaching the intestine, where most nutrient absorption occurs, ultimately retaining up to 86.8 % of encapsulated curcumin.	High sheer homogenizer at 22,000 rpm for 30 s	(Marefati et al., 2017) [171]
	Curcumin and lecithin diluted in warm absolute ethanol	Debranched starch dispersed in distilled water	Curcumin was successfully encapsulated using debranched starch but the stability of the emulsions was not good, consistent with the low zeta potential values. Results also showed that water bridges between the debranched starch and curcumin may contribute to the complexing processes for encapsulation.	High pressure homogenizer at 500 bar for 2 cycles	(Feng et al., 2020) [185]
	Medium chain triglycerides	Modified kaolinite with phosphatidylcholine dispersed in deionized		high-shear homogenizer at 8000 rpm for 30 s	
Carotenoids	Medium Chain Triglycerides oil	pullulan and whey protein isolate dissolved in deionized water	Concentration of beta carotene went from 0 %, after 7 d of storage without wall material, to 40 % after 7 d of storage with the wall material mixture showing an improvement in beta carotene stability due to emulsion formulation	Ultrasonication at 0, 120, 240, 360, and 480 W	(Niu et al., 2020) [63]
	Corn oil	Wheat gluten and Xantan gum dissolved in deionized water	Presence of Xantan gum in the formulation increased the stability of beta carotene by 39 % after storage at 60 °C for 14 d.	High-shear mixer at 13,500 rpm for 2.5 min	(Fu et al., 2019) [64]
	Soybean oil	Whey protein concentrate dissolved in distilled water and Zein from corn dissolved in a water-ethanol mixture (20:80 v/v)	Emulsion exhibited high beta carotene concentration, up to 74 %. Encapsulated beta carotene showed a higher bioaccessibility after in-vitro digestion, which was negligible in its free form.	High-shear mixer at 6000 rpm for 5 min and ultrasonication at an amplitude of 10 % and frequency of 20 kHz for 2 min in pulse mode.	(Gómez-Mascaraque et al., 2017) [51]
	Medium chain triglycerides	OSA-starch (Hi-cap 100®) dissolved in distilled water and chitosan dissolved in acetic acid solution (1 % v/v)	OSA-starch and chitosan interface in Layer-by-Layer emulsion showed a higher stability of beta carotene against heating than the single layer emulsion.	High-pressure homogenizer at 600 bar for 3 cycles for OSA-starch primary emulsion and 300 bar for 4 cycles for OSA-starch-chitosan emulsion	(S. Fang et al., 2019) [65]
	Corn oil	β-lactoglobulin and tween 20 dispersed in phosphate buffer	Vitamin E and CoEnzyme Q10 were used as antioxidant agents to prevent beta carotene oxidation during emulsion storage. Vitamin E, along with the wall materials, effectively retarded beta	Microfluidizer at 9000 psi for 3 cycles	(Qian et al., 2012a) [66]

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Table 1 (*continued*)

Lipophilic functional ingredient	Disperse phase	Continuous phase	Main findings	Emulsification technology	Reference
			carotene degradation in emulsion systems.		
	Vegetable oil	Maltodextrin and sodium caseinate	Lower temperature and feed rate conditions showed a higher beta carotene preservation than higher conditions. Storage temperature had influence on the degradation of beta carotene since higher loss was observed at 45 °C.	High pressure homogenizer at 68.95 MPa for 1 cycle	(Coronel-Aguilera and San Martín-González, 2015) [67]
Vitamin E	Sunflower oil	Maltodextrin, acacia gum, and β-glucans-rich extracts obtained from *P. ostreatus* powder in distilled water	Samples with 10 % oil and β-glucan extracts had an α-tocopherol retention up to 95.7 %, which was 68 % higher than that of the control with no β-glucan extracts, which have antioxidant properties.	Rotor-stator homogenization at 3900 rpm for 10 min	(Gallotti et al., 2020) [75]
	Medium chain triacylglycerol oil	OSA-modified starches (Capsul, Capsul TA, and HICAP100) dissolved in distilled water	Vitamin E retention ranged from 79.16 to 71.46 %. Dried emulsions containing Capsul were able to retain up to 50 % of the encapsulated vitamin E after 60 d storage at 4–35 °C. HICAP showed a reduced retention and shorter half-life (35 d).	Rotor-stator homogenizer at 14000 rpm for 5 min	(Hategekimana et al., 2015) [76]
	Canola and coconut oils	Ora-pro-nobis mucilage and whey protein isolate dispersed in distilled water	α-Tocopherol retention and degradation kinetics were greater affected when canola oil was used as a dispersed phase compared to coconut oil, where more than 85 % was retained. Wall materials improved the chemical stability and bioaccessibility by increasing its value by 5.8-times.	High-pressure homogenizer at 50 MPa and 10 cycles	(Neves et al., 2020) [78]
	Orange oil	Whey protein isolate, sucrose, stevia, and citric acid dissolved in distilled water.	Vitamin E retention reached levels up to 85 % for most emulsion formulations, which indicates that emulsions show great potential as delivery systems of lipophilic bioactive compounds.	Single stage valve homogenizer at 25 MPa for the first cycle and at 8 MPa for the second cycle	(Raikos, 2017) [77]
	Medium Chain triglycerides (Miglyol 812)	Citric Buffer	Surfactants present in the nanoemulsions led to the formation of colloidal structures in the mixed micelle phase that were able to encapsulate vitamin E acetate, thereby achieving a bioavailability value of 98 %.	Microfluidizer at 12,000 psi for 4 cycles	(Mayer et al., 2013) [69]
Omega 3	Sacha inchi oil	Xantan gum, pectin, and ovalbumin	Pectin had a greater efficiency to hold Sacha Inchi Oil than Xantan Gum. Procedure did not affect the omega 3 content in the emulsion. Microcapsules showed a very slow omega-3 release profile, which is good because it prevents omega-3 loss before reaching the intestine.	Sonication at a frequency of 30 KHz and amplitude of 100 % for 12 min	(Vicente et al., 2017) [84]
	Fish oil (extracted from Carp viscera)	Chitosan dissolved in acetic acid (1 % v/v)	Encapsulation process had no impact on the oil quality. They also showed a potential	Mechanical stirrer at 10,000 rpm for 10 and 20 min	(Esquerdo et al., 2015) [186]

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Table 1 (*continued*)

Lipophilic functional ingredient	Disperse phase	Continuous phase	Main findings	Emulsification technology	Reference
			to retard the primary oxidation of unsaturated fatty acid contained in the Carp viscera oil		
	Fish and garlic essential oils	Persian gum dissolved in distilled water and chitosan dissolved in glacial acetic acid (1 % v/v)	Increasing Persian gum concentration improved the stability of emulsions. Capsules were stable at temperatures under 250 °C, indicating that wall material may protect the encapsulated oil in food processing methods involving high temperatures	Rotor-stator homogenizer at 4000 and 10,000 rpm for 3 and 1 min, respectively, followed by sonification at an amplitude of 100 % for 4 min for the first layer; rotor-stator homogenizer at 3000 rpm for 0.5 min and 10,000 rpm for 0.5 min followed by sonication at an amplitude of 80 % for 0.5 min	(Raeisi et al., 2019) [83]
	Fish oil	Whey protein isolate (WPI) and fish protein hydrolysate (FPH) dissolved in distilled water	WPI exhibited a higher emulsifying property than FPH and led to more physically stable blend with smaller droplet size. Although most of the fish oil was encapsulated inside the fibers as small droplets, the fibers presented poor oxidative stability	Microfluidizer at 9000 psi for 3 cycles	(García-Moreno et al., 2016) [187]
Vitamin D	Corn oil, fish oil, mineral oil, orange oil, or Miglyol 812	Q-Naturale® diluted in 10 mM sodium phosphate buffer	Bioaccessibility was highly dependent on the type of carrier oil present in the nanoemulsions. It was higher for corn and fish oils due to the higher solubilization capacity for vitamin D3 of mixed micelles formed by long chain free fatty acids. Long chain triglyceride nanoemulsions were the most suitable for increasing the bioaccessibility of vitamin D3.	High-pressure homogenizer at 12,000 for 3 cycles	(Ozturk et al., 2015) [188]
	Soybean oil	Nanofibrillated cellulose (NFC) or WPI diluted in 10 mM sodium phosphate buffer	Residual vitamin D in emulsions after passage through simulated conditions of the gastrointestinal tract decreased when the NFC content increased because the cellulose fibres did not allow lipase to access the oil droplets present in the emulsion. The cellulose fibres did not have a major impact on vitamin bioaccessibility and stability, and led to higher values for whey protein when used in low concentrations (1:10 NFC-to-oil).	High pressure homogenizer at 12,000 psi for 3 cycles	(Winuprasith et al., 2018) [55]

emulsions are not only designed to protect lipid compounds, Essential oils [30–34] and microorganisms [35–37] can also be protected in O/W emulsions.

W/O emulsions are usually present in the solid or semi-solid state because they are very unstable in the liquid state owing to the high mobility of water droplets [38,39]. Water-in-oil emulsions can also be used as a tool to reduce the saturated and *trans*-fat content of processed foods [40]; Luo et al. (2019) fabricated a water-in-oil emulsion using tea polyphenol palmitate as a stabiliser to substitute for transfat-rich solid or semi-solid fats. They found that the palmitate particles produced stable, surfactant-free, gel-like W/O emulsions. The emulsion had a high slip melting point, comparable to that of margarine, providing a potential alternative to solid fats [41].

Oil-in-water or water-in-oil emulsions are not the only possibilities; it is also possible to create several types of emulsions, such as oil-in-water-in-oil (O/W/O) or water-in-oil-in-water (W/O/W) emulsions [42]. Water-in-oil-in-water (W/O/W) emulsions consist of small water droplets contained within larger oil droplets dispersed in an aqueous continuous phase, whereas oil-in-water-in-oil emulsions consist of small oil droplets contained within larger water droplets dispersed in an oil continuous phase. In the food industry, double emulsions are used in the preparation of low-calorie and reduced-fat-content products to encapsulate and protect bioactive ingredients (vitamins, minerals, enzymes, proteins, polysaccharides, etc.), antioxidants, and flavour compounds and to control the release of bioactive compounds during digestion [42–45]. Double emulsions are more accurately designated as W1/O/W2 or O1/W/O2 emulsions, where W1 or O1 is the inner phase and W2 or O2 is the outer phase, which may have different compositions. Chouaibi et al. (2019) used W/O/W emulsions to encapsulate quercetin using different oil phases (olive, red pepper seed and sunflower oils) finding that double emulsions were stable when stored at refrigerated (5 °C) and room temperature, they also concluded that the release behaviour was strongly influenced by the composition of the oil phase, being sunflower oil the one that showed a greater release rate and red pepper seed oil the one with the smallest release rate [46].

2.2. Lipophilic functional ingredients

There has been growing interest in the food industry to encapsulate, protect, and release various lipophilic food components [47]. Functional ingredients such as β-carotene [48–51], tocopherols [52–54], vitamine D_3 [55,56], etc. have been encapsulated successfully, improving the bioavailability, and stability of functional ingredients. Table 1 lists some of the most common lipophilic ingredients encapsulated using the emulsification technique.

Carotenoids: Carotenoids are a large and diverse group of yellow-to-red lipophilic compounds that consist of 3–13 conjugated double bonds and, in some cases 6 carbon ring structures at one or both ends of the molecule [57,58]. Carotenoids containing oxygen are known as xanthophylls while those without oxygen are known as carotenes, where β and α-carotene are found [59]. Carotenoids, particularly beta-carotene, can be metabolized within the body to form retinal and retinoic acid, compounds vital for vision, immune function, normal growth, and development. Retinoic acid has demonstrated the ability to inhibit the development of various cancers in both animal and in vitro studies. Additionally, the accumulation of lutein and zeaxanthin in the eye has been linked to a decreased risk of age-related macular degeneration, a major cause of blindness in older adults. These compounds help protect against light-induced damage, a key risk factor for macular degeneration, by filtering high-energy blue light and neutralizing reactive oxidants [60,61].

Owing to their importance in the food industry and their role in promoting human health and reducing the risk of chronic diseases due to vitamin A deficiency, carotenoids, specifically beta-carotene, are lipophilic ingredients that have drawn the attention of the food industry [58]. Carotenoids that are naturally present in foods are generally stable; however, when used as food additives, they are relatively unstable because they are sensitive to pH, light, oxygen, and temperature and are susceptible to oxidation, isomerisation, and photosensitization during production and storage, in addition to their low water solubility [16]. Therefore, the use of carotenoids as additives in food matrices may result in degradation and poor solubility [62]. To overcome these difficulties, several researchers (Table 1) have included beta carotene in emulsion systems. All results showed how beta-carotene stability improved [63–67] and how bioavailability increased as a result of water solubility improvement [51].

Vitamin E: Vitamin E is a yellow viscous oil that is readily oxidised when exposed to light and oxygen [13]. Vitamin E is made up of out of eight lipophilic compounds; four tocopherols and four tocotrienols designated as α, β, γ and δ, with α-tocopherol being the most abundant and bioactive form [68,69]. Tocopherols differ from tocotrienols in that they contain three double bonds at carbons 3, 7, and 11 [70]. Vitamin E plays a crucial role in human health, offering a range of benefits supported by substantial research. It helps to strengthen the immune system and prevent inflammation, which can reduce the risk of diseases such as cancer and arthritis. By inhibiting the production of reactive oxygen species during fat oxidation, vitamin E may also play a key role in preventing muscle degeneration associated with aging. Additionally, vitamin E contributes to brain health by combating oxidative stress, a major factor in neurodegenerative disorders such as Alzheimer's disease [68,71,72].

Vitamin E has low water solubility and poor chemical stability (to oxygen, light, and heat). Food processing may lead to substantial changes in the vitamin E content that is naturally present in food, leading to variable bioavailability [73,74]. Suitable delivery systems are required to incorporate Vitamin E into aqueous products to improve their chemical stability [75–77] and bioavailability [69,78].

Omega-3 fatty acids: Omega-3 fatty acids are unsaturated fatty acids with a double bond that is 3 carbon atoms from the methyl end of the molecule. The most common ω-3 fatty acids are α-linolenic acid, eicosapentaenoic acid, and docosahexaenoic acid [59]. The literature indicates that omega-3 fatty acids are essential for normal growth and development, as well as for their positive effects on the heart, brain, eyes, joints, skin, mood, and behaviour [79,80]. Had also shown to possess antiarrhythmic actions, prevention of platelet aggregation, relaxing of arterial smooth muscle, lowering of plasma triglycerides and blood pressure, and a modest antithrombotic effect [81]. Fatty acids are highly susceptible to lipid oxidation and have low water solubility. Therefore, omega-3 fatty acids are usually incorporated into colloidal delivery systems to protect them during processing, storage, and transport [82]. The main food source of Omega-3 fatty acids is fish oil [83], which is why most authors shown in Table 1 use fish oil as the dispersed phase in the formulation of emulsions. However, new sources of omega 3 fatty acids, such as Sacha Inchi oil, have been explored [84].

Vitamin D: Vitamin D is a liposoluble micronutrient found in two different chemical forms, vitamin D_2 (ergocalciferol) and vitamin D_3 (cholecalciferol). In humans, vitamin D_3 has greater biological potency than vitamin D_2 and is typically synthesised by the skin after exposure to light [55,85]. Vitamin D plays a crucial role in maintaining the health of older adults by preventing osteoporosis and reducing the risk of cancer. A deficiency in this nutrient has been linked to heart disease, including hypertension, type 1 and 2 heart failure, and ischemic heart disease. Long recognized for its role in bone health, insufficient vitamin D can lead to rickets, a condition characterized by soft bones and skeletal deformities. Adequate dietary intake of vitamin D is essential for supporting overall health and

mitigating these health risks [26,86,87].

Fortification with vitamin D has been a challenge to the food industry because of its instability under various conditions (temperature, iodine, acidic conditions, and oxidation) and heterogeneous distribution in food [87]; therefore, its incorporation into colloidal delivery systems is an alternative to overcome these difficulties. The results presented in Table 1 show that bioaccessibility increased when vitamin D was included in colloidal delivery systems. However, it is also important to optimise the encapsulation conditions because excessive use of stabilisers can prevent the release of the oil included within the emulsions, limiting the bioavailability and bioaccessibility of functional oils even more than when they are not included in the colloidal matrices.

Other lipophilic functional ingredients: Curcumin, a non-polar polyphenol and the main component of turmeric roots, is often used as a natural food pigment due to its bright yellow colour [88,89]. Its use in the food industry as a pigment is well known, but curcumin has also aroused great interest in the functional food industry owing to its health benefits, including antioxidant, anti-inflammatory, and anticancer activities might be found. Therefore, there has been great interest in including this ingredient in different food products. However, the poor physicochemical stability and bioavailability of curcumin severely limit its application, and therefore, its encapsulation has attracted great attention [90]. The emulsification technique improved the half-life and bioavailability of curcumin under storage conditions [91].

Another lipophilic functional ingredient of interest is resveratrol, a natural polyphenolic compound found in various plants, including grapes, red wine, and some berries. It is well-known for its potential health benefits, including antioxidant and anti-inflammatory properties [23]. The incorporation of resveratrol into functional food matrices can be challenging because of its poor water solubility, low bioavailability, and chemical instability [92]. Emulsions can provide a protective environment for resveratrol by enhancing its stability, solubility, and bioavailability [93]. Several studies have shown that the use of emulsions improves resveratrol bioavailability [17,18,92,94].

Coenzyme Q10 (CoQ10), another lipophilic functional ingredient of interest, is an endogenous compound found in human cells that plays a pivotal role in cellular energy production and functions as an antioxidant [95,96]. This molecule plays an important role in the electron transport chain and is responsible for generating adenosine triphosphate (ATP), the primary cellular energy source. Notably, CoQ10 contributes to cell well-being by supporting the maintenance of mitochondrial function. While the body has the capacity to synthesise CoQ10 naturally, its production can diminish with advancing age or specific medical conditions [97]. CoQ10, being fat-soluble, holds the potential to undergo degradation and oxidative stress. Thus, emulsions have emerged as valuable tools. Emulsions provide an ingenious means of encapsulating and protecting CoQ10 from the detrimental effects of oxidation, light, and heat. This encapsulation strategy enhanced the stability and bioavailability of CoQ10, thereby amplifying its effectiveness. Moreover, the oil-in-water (O/W) structure of emulsions promotes enhanced solubility and controlled release of CoQ10 in the digestive system, thereby optimising its absorption [98].

2.3. Solid particle stabilizers

Solid particles can adhere to the interface between immiscible phases, forming a tangible barrier that effectively halts droplet merging, a phenomenon known as coalescence. These particles, which can originate from natural sources such as clays or proteins, as well as synthetic sources such as polymers and nanoparticles, act as emulsion stabilisers. When these solid particles were introduced, they occupied the interfacial region, establishing a robust and intricate lattice-like structure. This framework exerts its influence by acting as a shield, shielding droplets from coalescence [99]. This stabilisation mechanism involving solid particles enables the creation of more stable emulsions. The same solid particles used to stabilise emulsions can also be employed to encapsulate lipophilic ingredients within an emulsion system. These particles serve a dual purpose: they provide stability to the emulsion, while also acting as carriers for the encapsulated lipophilic components [63,100].

Solid Particle selection is crucial during emulsion preparation as it influences the rheological properties of the emulsion, the mechanical stability, encapsulation efficiency and has an important effect on the shelf-life of the final product. Solid particles used in functional food-based emulsions should have good water solubility, film-forming properties, and emulsifying properties [99,101]. The

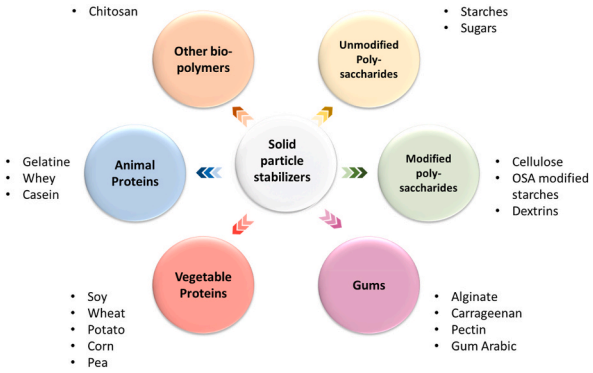

Fig. 3. Wall material classification. Adapted from: (Lekshmi et al., 2021a; Sobel et al., 2014).

solid particles are mainly natural polymers. These can be classified, as shown in Fig. 3. Their selection depends on the characteristics of the lipophilic ingredient to be encapsulated and the desired characteristics of the final product.

Proteins and polysaccharides are widely used as solid particle stabilisers [102]. Proteins can be divided into animal or vegetal proteins depending on their origin. Common animal proteins include milk-derived proteins (whey protein, milk protein concentrate, and different forms of caseinates, among others), ovalbumin, and gelatine. These proteins have unique properties, including biodegradability, biocompatibility, and a high degree of structural flexibility, making them attractive materials for encapsulation. Whey protein, a byproduct of cheese production, has been studied for the encapsulation of various oils, including pomegranate seed oil [103], sunflower oil [104,105], as well as functional ingredients such as iron [21], resveratrol [106], and tocopherol [107]. The encapsulation efficiency of whey protein was high, and the coated oils showed improved oxidative stability [108]. Gelatine, a protein obtained from collagen, has also been studied for encapsulation of edible oils and functional lipophilic ingredients [109–112]. Casein, a milk protein, forms stable emulsions with various oils [113,114].

Among vegetable-based proteins, legumes, nuts, tubers, and cereal proteins are used for the encapsulation of lipophilic active ingredients. These natural polymers have good emulsifying and amphiphilic properties, which provide the necessary physical, chemical, and functional properties for the encapsulation of these ingredients. Legume-based proteins are the group with the greatest variety and include soybeans, peanuts, peas, chickpeas, beans, and lentils. Among the legume groups, soy protein is the most commonly used protein in the emulsification of bioactive compounds [115]. Vegetable proteins are often combined with natural polymers to create stable emulsions. Yang et al. (2020) studied the interaction between soy protein isolate (SPI) and chitosan (CS) and found that SPI and CS produced very stable emulsions that could be stored for 20 days, showing little change in appearance or droplet size. Wu et al. (2022) prepared emulsions based on soy protein isolates and mixed them with tannic acid to improve the antioxidant properties of the emulsion and protect essential oil properties. The results showed that nanoparticles also maintain the aroma of beverages and lengthen their shelf-life.

There are several advantages of using vegetable-based proteins for emulsification over animal-based proteins, including Allergen-free: Vegetable-based proteins that are free from common allergens such as milk, egg, and wheat, making them suitable for consumers with food allergies or intolerances [118]; Vegan-friendly: Vegetable-based proteins are suitable for vegan diets, which are becoming increasingly popular owing to ethical and environmental concerns and Health benefits: Some vegetable-based proteins, such as soy protein, have been associated with health benefits such as improved cholesterol levels and reduced risk of heart disease [35,119].

Polysaccharides are commercially used in the food industry because of their low toxicity, high biocompatibility, and biodegradability. Polysaccharides can reduce the Brownian movement of the system, maintain the stability of the emulsified components for a longer time, and can be used as thickeners or stabilisers to form a network structure to increase the viscosity of the continuous phase. Finally, polysaccharides can modify the rheological properties and increase the stability of emulsions [120]. Among polysaccharides, gums, starches, and chitosan are the most commonly used.

Natural gums are widely used in emulsification processes owing to their ability to stabilise emulsions and improve their texture and stability. One commonly used natural gum in emulsification is xanthan gum, a polysaccharide produced by Xanthomonas campestris [121]. Guar gum, derived from the seeds of the guar plant, *Cyamopsis tetragonoloba*. It is a galactomannan gum that has excellent water-binding properties [120]. Pectin is a heteropolysaccharide composed mainly of galacturonic acid units found in the cell walls of plants [120], and Gum Arabic: A natural exudate obtained from the Acacia tree. It is a complex mixture of polysaccharides and glycoproteins [31]; Carrageenan: Extracted from red marine algae, is an anionic polysaccharide consisting of D-galactose chain bearing one sulphate group every two of units. Commonly used stabilisers and emulsifiers for emulsions [122].

Starch is an abundant natural polysaccharide found in plant cells that serves as an energy storage molecule. Traditionally, they are recognized for their thickening and gelling properties. Starches act as emulsifying agents through several mechanisms including steric stabilisation, electrostatic interactions, and film formation [123]. When starches are dispersed in water, they form a colloidal system that can coat oil droplets, preventing their aggregation and subsequent phase separation [124]. The use of starch in emulsification is of growing interest given its abundance, feasibility of extraction, and potential to be modified to increase its hydrophobicity through esterification, such as hydrophobisation with octenyl succinic anhydride (n-OSA) which confers amphiphilic properties to the starch molecule [124,125].

Moreover, chitosan is a polymer obtained from the alkaline deacetylation of chitin, which naturally occurs in the cell walls of some fungi but mainly in the insect cuticles and exoskeletons of crustaceans [126,127]. Owing to its film-forming properties and antimicrobial activity, chitosan has been used as a wall material to carry lycopene [128,129] and essential oils, such as clove [130], cinnamon [33], garlic [32] and allspice [131]. The results showed that chitosan nanocarriers loaded with different pro-vitamin A showed improved thermal stability, dispersibility, and controlled release, indicating that it is a good alternative for the delivery of several bioactive compounds [129]. Essential oils retain their antimicrobial and fungicidal properties and enhance food and plant preservation [32,33,130,131].

Proteins and polysaccharides are commonly used to produce O/W emulsions. Proteins can stabilise emulsions based on their isoelectric points (Ips) [132]. However, aggregation can occur at high pH, Ip, and ionic strength. Therefore, the addition of polysaccharides can increase the stability of aggregation because polysaccharides form a thick charged interface layer that increases steric and electrostatic repulsion between droplet surfaces [133]. Polysaccharides are hydrophilic molecules that can be adsorbed onto the surface of protein-coated oil droplets through electrostatic interactions. Therefore, the stability of emulsions can be improved by forming complexes between the proteins and polysaccharides [120]. Several authors have reported the use of protein-polysaccharide complexes to encapsulate lipophilic ingredients such as clove essential oil [31]soybean oil [123], medium-chain triglycerides [63,65,120], curcumin [91], fish oil [83], and Sacha inchi [84].

2.4. Surfactants

A surfactant, short for surface active agent, is a compound that has both hydrophilic and hydrophobic properties. This unique chemical structure allows surfactants to lower the surface tension between two immiscible phases by adsorption at the interface between them [134]. Therefore, surfactants are used in a wide range of applications, including emulsification, foaming, wetting, and dispersion. Surfactants contain polar head groups that can interact with water molecules via hydrogen bonding or electrostatic interactions. The head group can be a variety of chemical groups such as carboxylate, sulphate, amine, or quaternary ammonium. In addition, they have a nonpolar tail group, mainly composed of hydrocarbon chains of varying lengths and saturation levels, or fluorinated chains [135].

Surfactants produce more stable emulsions by reducing the interfacial tension between the two immiscible phases in the emulsion. This interfacial tension is the force that opposes the formation of an interface between the two phases and can lead to the formation of unstable emulsions, such as oil droplets coalescing or settling. Surfactants are commonly used in the food industry to emulsify oils and water-based ingredients and create stable matrices. Different types of surfactants, including natural and synthetic, can be used for food oil emulsification. An important consideration when selecting surfactants for food oil emulsification is their safety and potential impact on human health. Some surfactants may have negative effects on human health, such as allergic reactions or disruption of hormonal balance. Table 2 lists the most common surfactants used in the food industry and their accepted daily intake per kg of body weight. Another consideration when selecting surfactants for food-oil emulsification is their ability to form stable emulsions. Surfactants that are too weak may not be effective in creating stable emulsions, whereas surfactants that are too strong may cause emulsions to break

Table 2
Common surfactants used in the food industry, their description, and European Food Safety Authority (EFSA) accepted daily intake (ADI).

Surfactant	Description	International numbering system (INS)	EFSA	Reference
Lecithin	Lecithin is a naturally occurring phospholipid, a type of fat molecule, found in many plant and animal tissues. It is commonly extracted from sources such as soybeans, egg yolks, and sunflower seeds	E322	No safety concern for the general population from more than 1 year of age	(Mortensen et al., 2017b) [189]
Polysorbates	Polysorbates, also known as tweens, are non-ionic surfactants derived from polyethylene glycol (PEG) and sorbitan. The number in the name (20, 40, 60, 65, and 80) refers to the number of ethylene oxide units in the PEG chain	E432, E434, E435, E436, and E433	ADI of 25 mg/kg body weight/day. There are no concerns of genotoxicity, carcinogenicity, or developmental toxicity if the ADI dosage is followed.	(EFSA Panel of Food Additives and Nutrient Sources, 2015) [190]
Salts of fatty acids	Salts of fatty acids, also known as soap salts, are compounds formed from the reaction of a fatty acid with an alkali metal hydroxide or alkaline earth metal hydroxide. This reaction produces a salt, with fatty acid as the anion (negatively charged ion) and metal ion as the cation (positively charged ion). The most commonly used salts of fatty acids are sodium and potassium salts	E470	There were no data on subchronic toxicity, chronic toxicity, and reproductive and developmental toxicity of the salts of fatty acids. There was no concern for mutagenicity. No ADI was needed.	(Younes et al., 2018) [191]
Mono- and diglycerides	derived from natural sources such as vegetable oils and animal fats. Mono- and diglycerides are composed of one or two fatty acid chains attached to a glycerol backbone. They are soluble in both water and oil, which makes them ideal for stabilising emulsions	E471	No evidence for adverse effects was reported in short-term, subchronic, chronic, and reproductive and developmental toxicity studies. Neither carcinogenic potential nor a promotion effect in initiation/promotion was reported. No ADI was needed.	(Younes et al., 2017) [192]
Sorbitan fatty acid esters	Sorbitan monostearate (span 60), sorbitan tristearate (Span 65), sorbitan monolaurate (Span 20), sorbitan monooleate (Span 80), and sorbitan monopalmitate (Span 40) are non-ionic surfactants often used as food additives	E491, E492, E493, E494, and E495	ADI of 25 mg/kg body weight/day for E491, E492, and E495 and ADI of 5 mg/kg body weight/day for E493 and E494. Span may have side effects on the kidneys. No indication of carcinogenicity was observed if ADI was respected.	(Mortensen et al., 2017a) [193]
Ammonium phosphatides	They are derived from a mixture of phospholipids, primarily derived from soybean oil, and are composed of phosphatidylcholine, phosphatidylethanolamine, and phosphatidylinositol among others.	E442	ADI of 30 mg/kg body weight/day. Usage as a food additive at the permitted ADI dosage is not of safety concern.	(Mortensen et al., 2016) [194]
Sucrose acetate isobutyrate	It is a complex mixture of esters formed by the reaction of sucrose with acetic and isobutyric acids	E444	ADI of 20 mg/kg body weight/day. High dosage may cause hepatic effects. Usage as a food additive at the permitted ADI dosage is not of safety concern.	(EFSA ANS Panel (EFSA Panel on Food Additives and Nutrient Sources added to Food, 2016) [195]

Table 3
Description of main emulsification technologies.

Emulsification technologies	Description	Equipment	Example	Reference
Mechanical agitation	Mechanical agitation is a common method used to create emulsions by applying mechanical energy to the mixture. This method involves the use of an external device, such as a mixer, blender, or homogenizer. The process involves subjecting the mixture to high shear forces, which help to break up the droplets and disperse them throughout the continuous phase	Stirring or mixing: a mixing device, such as a paddle, propeller, or anchor, is rotated in a vessel or tank. Rotational movement creates turbulence and shear forces. High-Speed Homogenization: rotor-stator mixers or high-shear mixers generate high shear forces by rapidly rotating a rotor within a stationary stator. Colloid Mills: They consist of a rotor and a stator with a narrow gap between them. The product is forced through the gap, where high shear forces and friction occur.	Fish oil loaded gelatine/surfactant-stabilized emulsions were obtained by homogenizing the disperse phase into the continuous phase using an ultra-turrax homogenizer at a speed of 8000 rpm and homogenizing time of 2 min. Emulsions with droplet sizes ranging from 4.02 to 15.51 μm were obtained.	(Zhang et al., 2020) [138]
High pressure homogenization	It is a mechanical process used to reduce the particle size and create a stable dispersion of components in a fluid, typically in the form of an emulsion. It involves subjecting the sample to high pressure and forcing it through a narrow gap or valve, resulting in intense shear forces and turbulence. This process facilitates the breakup of larger particles or droplets, leading to a reduction in size and improved homogenization	Piston-gap homogenizer: It consists of a reciprocating piston that forces the sample through a narrow gap or valve, generating high shear forces and turbulence. Valve homogenizer: The sample is pumped into the homogenizer and forced through a small orifice or series of valves. The sudden reduction in cross-sectional area at the valve creates high shear forces.	Clove essential oil-loaded sodium caseinate and gum Arabic emulsions were obtained via high pressure homogenization. Coarse emulsion was subjected to homogenization under different pressures (200, 400, 600, and 800 bar) and homogenization times (2.5, 5, 7.5, and 10 min). Droplet size ranged between 200 and 260 nm. Average droplet size decreased as homogenization pressure increased, reaching a maximum stability of 60 d.	(Yin et al., 2023) [31]
Microfluidization	In this method, the emulsion is forced through microfluidic channels under high pressure, which causes the droplets to break up and disperse into the continuous phase. The microfluidic channels are designed with narrow and precise dimensions to provide a controlled and uniform distribution of shear forces, resulting in the formation of droplets with a narrow size distribution. The emulsion is usually passed through the microfluidic channels multiple times to further reduce the droplet size and improve the stability of the emulsion.	Microfluidizer: It uses microchannels or chambers with micron-sized dimensions to exert intense shear and impact forces on the sample.	Most favourable conditions for the microfluidization of high oleic palm oil emulsion were studied. Pressure varied from 10000 psi to 20000 psi for 1, 2 and 3 cycles. Results showed that 20000 psi for 2 cycles avoided the coalescence effect, producing stable nanoemulsions. Average droplet size ranged between 163 and 2268 nm	(Ricaurte et al., 2016) [54]
Ultrasonication	Ultrasonication is a technology that involves the use of sound waves in a specific frequency range between 20 kHz and 10 MHz (above the range of human hearing) to produce a shear force and break up the droplets in the emulsion. Microbubbles will form, expand, and eventually collapse in the medium during this process, which is caused by alternating pressure changes, known as acoustic cavitation.	Ultrasonic bath: consists of a tank or bath filled with a liquid sample. It contains ultrasonic transducers that emit high-frequency sound waves into the liquid. The sample is immersed in the bath, and the ultrasonic waves cause cavitation and acoustic streaming. Ultrasonic Probes/Horns: They are handheld or mountable devices that are directly inserted into the liquid sample. When the generator is activated, the probe vibrates at a high frequency, producing ultrasonic waves that propagate through the liquid. The intense cavitation and shear forces generated by the probe result in particle size reduction.	Different homogenization techniques were evaluated in the production of high oleic palm oil nanoliposomes. Coarse emulsion was homogenized using ultrasound (20 kHz), microfluidizer (500–1500 bar), and rotor stator (16000–26000 rpm). Results showed that ultrasound and microfluidization nanoliposomes presented the highest stability when loaded oil was maximized.	(Beltrán et al., 2020) [167]
Membrane emulsification	Membrane emulsification is a specialized emulsification technique that involves the use of porous membranes to create small and uniform droplets in the emulsion. It offers advantages, such as precise control over droplet size, high production efficiency, ability to work	A suitable membrane is chosen based on factors such as membrane material, pore size, and surface properties. The membrane should have a pore size smaller than the desired droplet size to ensure efficient droplet formation (Konovalova et al., 2023)	Influence of the emulsification techniques (membrane and ultrasound) on microparticles properties was evaluated. Droplet size obtained by membranes ranged between 154 and 52 μm, while ultrasound produced 0.16 μm size droplets. Even though ultrasound particles were larger than membrane	(Consoli et al., 2023) [23]

(*continued on next page*)

Table 3 (*continued*)

Emulsification technologies	Description	Equipment	Example	Reference
	with a wide range of materials, and protection of heat and shear sensitive ingredients (Konovalova et al., 2023)		emulsion particles, membrane technique had the highest encapsulation efficiency and resveratrol retention.	

down over time. The properties of surfactants can be adjusted by varying their concentration and type.

2.4.1. Natural surfactants

Natural surfactants are compounds derived from natural sources, such as plant extracts, proteins, and lipids, and are commonly used as emulsifiers in the food industry. They are preferred to synthetic surfactants because of their perceived safety, natural origin, and environmental sustainability [136].

Lecithin, a phospholipid found in egg yolk and soybeans, is one of the most commonly used natural surfactants in the food industry. Lecithin is primarily used to stabilise emulsions, particularly oil-in-water emulsions, by reducing the interfacial tension between the oil and water phases, showing good results compared to synthetic surfactants such as Tween 80 and Span 80 [137,138]. Several studies have used lecithin as a surfactant to stabilise emulsions [54,139–141].

Protein-based surfactants, such as casein and whey protein, are also widely used as emulsifiers in the food industry. Casein, a milk protein, is commonly used in dairy products, such as cheese and yoghurt. Both proteins exhibit excellent emulsifying properties and are often used to improve the texture and stability of oil-loaded emulsions [108,128,142–144].

Plant-based extracts, such as saponins, which are found in plants such as quinoa and spinach, are also used as natural surfactants in the food industry. Other plant-based surfactants include gum arabic (GA) [105,145,146] and pectin [120,147].

2.4.2. Synthetic surfactants

Synthetic surfactants are widely used as emulsifiers in the food industry owing to their high efficiency, versatility, and cost-effectiveness. Synthetic surfactants are often produced from petroleum-based sources via chemical synthesis and can be tailored for specific applications and performance requirements [148]. Examples of the synthetic surfactants commonly used in the food industry are polysorbates, sorbitan esters, and polyglycerol esters [149]. Polysorbates, such as polysorbate 80, and sorbitan esters, such as Span 80 are widely used as emulsifiers [19,149–151]. Although synthetic surfactants are highly effective emulsifiers, their use in the food industry has raised concerns regarding their safety and environmental impact. Some synthetic surfactants exert adverse health effects, such as disruption of the endocrine system and allergic reactions. Additionally, synthetic surfactants are not biodegradable and can accumulate in the environment, posing a threat to ecosystems.

2.4.3. Recent advances in surfactants

New surfactants are constantly being developed and introduced into various industries. Recent examples include the following:

Biosurfactants: Surfactants are produced by microorganisms, such as bacteria, fungi, and yeasts. Biosurfactants have attracted considerable attention because of their biodegradability, low toxicity, and unique properties. Biosurfactants are widely seen as a favourable replacement for their synthetically produced counterparts; as such, their applications and the proportion of the overall surfactant market they occupy have been increasing over the past 15 years [152]. Similar to their synthetic counterparts, biosurfactants have various chemical structures. They are produced by microorganisms grown on insoluble (oils, fossils, and hydrocarbons) and soluble (carbohydrates) substrates [153,154].

Gemini surfactants contain two hydrophilic head groups and two hydrophobic tail groups covalently attached by spacer groups [155,156]. Gemini surfactants have been found to have superior emulsification and wetting properties compared to conventional surfactants. They are used for applications such as enhanced oil recovery, detergency, and drug delivery. Different types of classifications for GS based on physicochemical aspects of the spacer and tail groups. One of the key features of gemini surfactants is their low critical micelle concentration (CMC), which is 100 times lower than that of single-chain surfactants. It is also more effective at reducing the interface/surface tension. GS was first used as a catalyst; however, new applications of this technology include the cosmetic industry and drug delivery systems [155].

Sustainable surfactants: With the increasing demand for sustainable products, there has been a focus on developing surfactants that are derived from renewable sources and have a low environmental impact. Examples include surfactants produced from vegetable oils, sugars, and proteins [31,123,125,132].

3. Emulsification technologies

3.1. Main emulsification technologies

Emulsification technologies play a crucial role in various industries by enabling the production of stable homogeneous emulsions with finely dispersed droplets. These technologies have advanced significantly and offer efficient and scalable methods for emulsion formation. Table 3 shows some of the most commonly used emulsification technologies. The emulsification technologies and their operational ranges are listed in Table 1.

Each emulsification technology has its advantages and limitations, and the choice depends on factors such as the desired droplet size, stability requirements, scalability, and properties of the materials involved. Understanding the principles and capabilities of these emulsification technologies allows the selection of the most suitable method for specific applications, leading to efficient and optimised emulsion production and the conservation of lipophilic functional ingredients.

3.2. Emulsion characterisation and stabilisation mechanisms

Emulsion characterisation involves the measurement and analysis of various physical and chemical properties such as droplet size, charge, stability, and composition. The techniques for emulsion characterisation included Microscopy, Particle size analysis, rheological analysis, electrical conductivity, chemical analysis, and ultrasonic attenuation spectroscopy (Table 4). These techniques can be used in combination to comprehensively characterise emulsions and are essential for understanding and optimising the performance of emulsions in various applications.

Multiple factors influence emulsion destabilization, such as temperature, pH, ionic strength, and emulsifier concentration, thorough emulsion characterization can provide valuable insights into potential stability issues and help guide adjustments to maintain emulsion integrity across different environments and conditions.

Emulsion destabilization is influenced by temperature, which plays a crucial role in reducing interfacial tension between phases and accelerating processes such as drop coalescence and flocculation. Increased temperature can lead to phase inversion, making the emulsion more sensitive to mechanical degradation and potentially causing the emulsion to transition to a microemulsion state. Additionally, pH level can impact interfacial tension and charge characteristics, affecting the stability of emulsifier films and increasing susceptibility to flocculation and coalescence. Ionic strength also plays a part in stabilising or destabilizing emulsions, depending on the types of surfactants used, while emulsifier concentration must be carefully managed to prevent excessive micelle formation and potential coalescence.

Finally, factors such as creaming, flocculation, Ostwald ripening, and phase separation may occur due to changes in these variables, affecting the stability of oil-in-water emulsions [120,122,157]. The first step in the separation of emulsions is the rising of the dispersed phase through the continuous phase. This is known as creaming and is a result of the density differential between the two phases. There are typically three mechanisms associated with creaming, but the process is usually governed by one. These mechanisms are gravitational drainage, packing and shrinkage of the droplet layer, and flocculation during the creaming process [158–160]. Flocculation is a process whereby droplets come together to form loose clumps or aggregates. These aggregates will then interact to form a more rigid, compact structure with a higher packing density than the primary droplet structure. The interaction between droplets is due to an attractive interaction potential, which is always a result of a free energy increase in the system [158–160]. Flocculation is driven by many mechanisms, including van der Waals forces, coalescence, bridging flocculation, and hydrophobic interaction. Ostwald ripening is a slow process that is driven by differences in droplet size. Due to Laplace's law, smaller droplets will have a higher solubility, resulting in the diffusion of material through the droplet surface into the continuous phase, and the diffusion of material from the continuous phase into the droplet. This leads to smaller droplets dissolving and the material precipitating in the region of the droplet

Table 4
Emulsion characterisation techniques.

Technique	Description
Microscopy	A technique used to observe the droplet size and morphology of emulsions under a microscope. The most used technologies include optical, electron, and atomic force microscopy. These techniques often provide information about structurally complex systems in a way that is easily understood by humans. Each microscopic technique operates on different physicochemical principles and can be used to examine different levels and types of structural organization [196].
Particle size analysis	This technique is used to measure the size distribution of the droplets in an emulsion. It can be performed using laser diffraction, dynamic light scattering, and nanoparticle tracking analysis. Laser diffraction is a fast and non-destructive technique to measure the particle size in emulsions, and it can be used for a wide range of particle sizes. In laser diffraction, the sample is first dispersed in a liquid medium and then introduced into the instrument. Intensity of the scattered light at each angle is then measured and used to calculate the particle size distribution and stability of the sample. Nanoparticle tracking analysis is also used to measure and analyse the nanoparticles in various liquid samples. It is particularly valuable for characterizing and quantifying nanoparticles in the size range of approximately 10 nm to 1 μm. It operates based on the principles of light scattering and Brownian motion similar to dynamic light scattering technique [197].
Rheological analysis	Rheology delves into the behaviours of material flow and deformation. This field explores viscosity, elasticity, and various attributes within emulsions. To maintain physical stability, interfacial rheology emerges as a crucial factor. Traditionally assessed through dilatational or shearing deformations, interfacial rheology wields significant influence. Dilatational measurements are more relevant for short-term stability, whereas interfacial shear rheology provides more valuable information on middle or long-term stability [198].
Electrical conductivity	Electrical conductivity measurements can be used to estimate the stability of an emulsion by monitoring the changes in conductivity due to changes in droplet size, concentration, and composition [54,167].
Chemical analysis	Chemical analysis can be used to determine the composition of emulsions, such as the concentration of oil, water, and surfactants, and to monitor changes in the composition of emulsions over time [196].
Ultrasonic attenuation spectroscopy	It is an interesting method to measure the distribution of droplet sizes in emulsions due to its ability to analyse viscous and optically transparent emulsions. This method depends on the ultrasound acoustic speed and viscosity coefficient in emulsion stability and changes in emulsion droplet size (Alshaafi, Prakash, and Mercer 2020) [199]. In ultrasonic spectrum analysis, frequency-dependent ultrasonic attenuation coefficients and acoustic velocities in colloidal dispersions are analysed to determine the concentration and size distribution of the colloidal particles [200].

with a higher curvature. This results in larger droplets growing and smaller droplets shrinking. The difference in the rate of material movement in and out of droplets is what separates this process from coalescence [158–160]. Coalescence is a dynamic process influenced by many factors. It is generally agreed that, before two droplets can coalesce, they must come into close contact with one another. The force that brings droplets together is the disjoining pressure [158–160].

4. Emulsion stabilisation technologies

Several emulsion stabilisation techniques have been used to prevent phase separation and maintain the stability of the dispersed phase within the continuous phase. Common stabilisation techniques include surfactants, emulsifiers, rheological modifications, Pickering emulsions, and encapsulation technologies. Surfactants and emulsifiers, both known as emulsifiers, form a protective interfacial layer that is adsorbed on the surface of the droplets (reviewed in previous sections), whereas stabilisers tend to maintain the physicochemical state of the emulsion by modifying its rheology. By modifying the viscosity and rheological properties of the continuous phase, emulsions can be stabilized [161]. Thickening agents such as gums, starches, or cellulose derivatives can be added to increase the viscosity and provide resistance to droplet movement and coalescence [157].

Pickering emulsion stabilisation involves the adsorption of fine insoluble solid particles at the oil–water interface [161]. The stabilisation of pickering emulsions occurs by reducing the interfacial energy between two immiscible phases through the adsorption of solid particles on the surface of the droplets, providing excellent stability to the emulsions [126]. Good Pickering stabilisation can be achieved when the average size of the adsorbed solid particles is at least one order of magnitude smaller than that of a dispersed-phase droplet [161]. The particle-based stabilisation of fluid-in-fluid dispersions has been actively studied as an alternative to surfactant-based stabilisation [112,114,116,124,162–166]. Matos et al. (2018) compared the use of OSA-modified quinoa starch as a solid particle stabiliser with that of Tween 20, a common non-ionic surfactant. The results showed that Pickering emulsions were more stable against creaming than Tween 20 emulsions. Additionally, the Pickering emulsions exhibited higher encapsulation efficiency, reaching values of up to 98 % [162]. More than one solid particle source can be utilised to obtain better results, H. Yang et al. (2020) used a mixture of soy protein isolate and chitosan to stabilise emulsions, obtaining stable emulsions up to 20 days, stored at 4 °C. emulsions showed good storage stability and anti-coalescence ability [116]. Pickering emulsions are promising candidates for overcoming the adverse effects of classical emulsifiers on environment and human health [117].

In emulsion stabilisation, encapsulation technologies can be classified into two distinct categories: those that yield liquid-state products, such as micelles and liposomes, and those that result in solid-state presentations, such as drying technologies. First, liposomes are spherical vesicles characterised by a lipid bilayer encapsulating an aqueous core and hold a significant position in colloidal

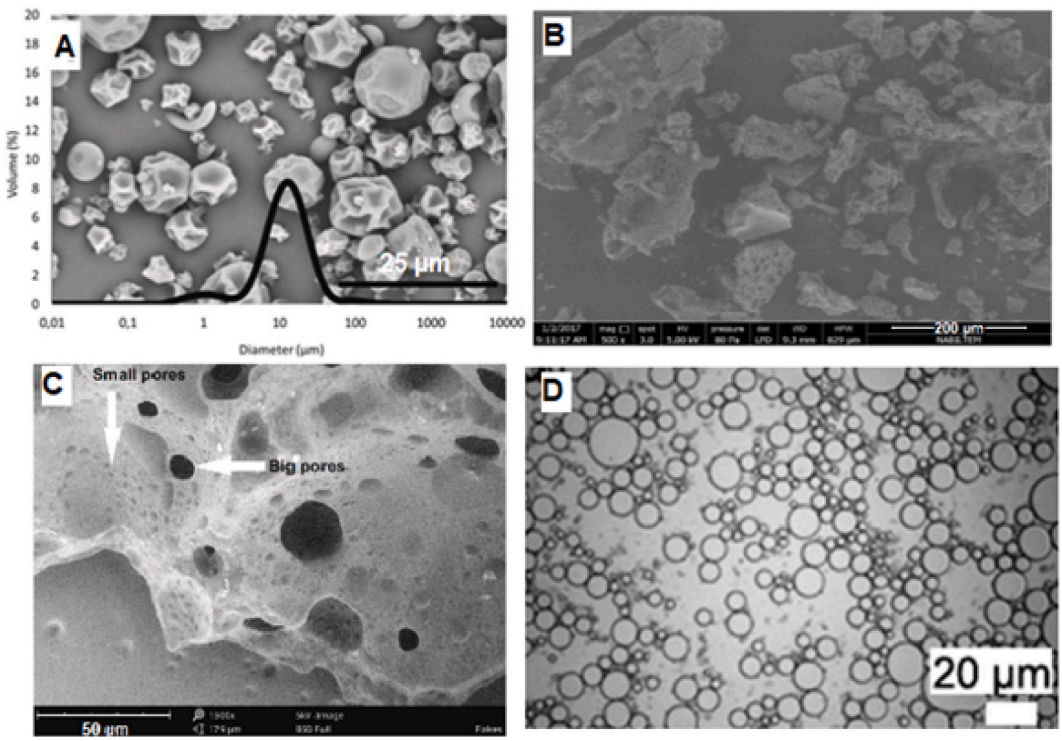

Fig. 4. Microphotography of emulsions stabilized via A) spray drying (Used as per permission from Elsevier [23], B) freeze drying (Used as per permission from Elsevier [80]) C) refractance window drying (Used as per permission from Elsevier [141]), **and D) pickering (Used as per permission from Springer Nature** [166]**).**

systems. Their unique structure offers remarkable versatility for various applications, with a particular focus on the encapsulation of water-soluble hydrophilic substances [167]. The encapsulation process is facilitated by an aqueous core that serves as an ideal environment for housing such substances. Simultaneously, the lipid bilayer enveloping the aqueous core acts as a protective barrier, effectively segregating aqueous content from the external environment. In this context, liposomes have emerged as valuable tools for stabilising oil-in-water (O/W) emulsions [168]. Because of their lipid bilayers, liposomes can interact with and envelop oil droplets dispersed in an aqueous medium. This enveloping action effectively hindered the coalescence and merging of oil droplets, thereby contributing to the stability of the O/W emulsions. Secondly, the micelles exhibit a spherical or cylindrical morphology, comprising amphiphilic molecules. These molecules manifest as a hydrophobic moiety that congeals at the micelle core and a hydrophilic segment that resides on its surface. Micelles demonstrate enhanced suitability for stabilising oil-in-water (O/W) emulsions, primarily because the hydrophilic region of amphiphilic molecules is oriented towards the aqueous phase, while the hydrophobic component resides within the micelles, protecting the dispersed water droplets within the oil phase [26].

Encapsulation technologies such as drying can be employed to stabilise emulsions by removing the liquid phase and converting the emulsion into a solid or powdered form. This can enhance the stability, shelf-life, and ease of handling of the emulsions. Common drying techniques used for emulsion stabilisation include spray drying, freeze drying, and refractance window drying.

Spray drying is a widely used technique for converting liquid emulsions into powder form. Spray drying offers advantages, such as functional lipophilic ingredient protection, shelf-life extension, ease of storage or transport, and easy reconstitution with water or other suitable solvents when needed [23]. Several researchers have explored the use of spray drying as a stabilisation technique [23,65,67,169]. When using spray drying to encapsulate and seal an emulsion, temperature is a very important factor to consider. Temperature might vary between 160 °C and 185 °C [65] and it will depend on the emulsion properties. X. H. Zhao & Tang. (2016) used a pea protein isolate, soy protein isolate, defatted milk powder, OSA starch, and spray drying to stabilise and encapsulate CoQ_{10}. Good encapsulation efficiency and redispersion behaviour were obtained [170].

Freeze-drying involves freezing the emulsion, followed by sublimation of the frozen water directly into vapour. This method preserves the structure of the emulsion while maintaining its original particle size and distribution. Freeze-dried emulsions exhibit improved stability, prolonged shelf-life, and the ability to be reconstituted by adding a suitable liquid. Marefati et al. (2015) [171] used a freeze-drying technique to dry multiple emulsions and obtained food-grade oil-filled powders from OSA-modified starch Pickering emulsions with an oil content of approximately 70 wt %. When working with multiple emulsions, heat might cause emulsion destabilization, which is why freeze-drying is a good alternative for drying these emulsions.

Although spray drying and freeze drying are the most commonly used technologies for stabilising emulsions through drying, in recent years, technologies that allow for cost reduction and drying time optimisation have emerged. Refractive window drying is based on the evaporation of moisture through the transfer of thermal energy from hot water to the material being dried, primarily through infrared radiation [172]. To date, few studies have been conducted on the use of refractance window technology to stabilise oil-in-water emulsions. In 2010, Cadwallader et al. [173], the use of a refractance window and spray drying were compared as technologies for the encapsulation of orange oil. The authors found that, for spray drying, the surface oil content increased with increasing inlet temperature, whereas for refractance window drying, the surface oil content decreased with increasing temperature. Overall, oil retention was better for refractance window drying. Research on the stabilisation of high-oleic palm oil nanoemulsions using the refractance window method has been conducted by Hernandez-Carrión et al. [174], and Henao-Ardila et al. [141], from the same research group. High-oleic palm oil was encapsulated in whey protein and starch (cornstarch and OSA modifies starch, respectively). The first work focused on the design of nanoemulsions suitable for refractance window drying, obtaining optimal whey protein and corn starch concentrations. The second work focused on the effect of the wall material on lipophilic functional compounds, finding the concentration that maximized their conservation.

Emulsion electrospinning is a novel and straightforward technique for creating multifunctional packaging materials capable of effectively protecting loaded active substances during the production process. This method allows the preparation of emulsion-based gelatine edible films that can encapsulate, protect, and release bioactive compounds for food packaging applications. The process demonstrates significant potential for stabilising and delivering lipophilic functional ingredients in emulsions, offering enhanced

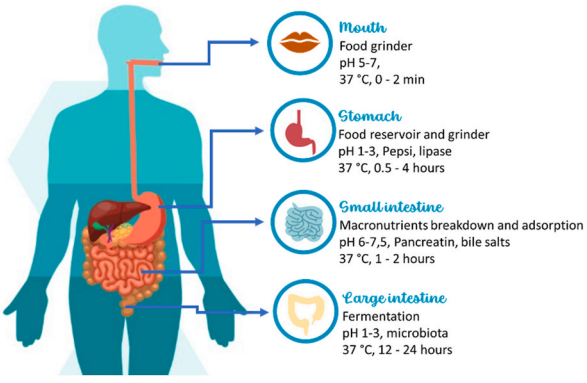

Fig. 5. Typical conditions for bioaccessibility evaluation of lipophilic functional ingredients.

Table 5
Main evaluation results of the digestibility of lipophilic functional ingredients.

Lipophilic functional ingredient	Main results	Reference
β-carotene	After in vitro digestion, the bioaccessibility of β-carotene increased from 3.1 to 35.6 % after nanoencapsulation. Moreover, the modified starch used as a wall material led to higher retention but lower bioaccessibility of β-carotene.	(Liang et al., 2013) [57]
Tocopherol and total phenolic content	Digested nanoemulsions showed high lipid digestion (85.25 %) and bioaccessibility of antioxidants and a lower rate of phytosterol degradation compared to the digested untreated oil.	(Cheong et al., 2016) [184]
β-carotene	Bioaccessibility of β-carotene decreased in the order of LCT > MCT > orange oil. Bioaccessibility of β-carotene was very low (0 %) in orange oil nanoemulsions because mixed micelles were not formed to solubilize β-carotene but high in LCT nanoemulsions (66 %).	(Qian et al., 2012b) [66]
Astaxanthin	Encapsulated astaxanthin was more stable than non-encapsulated astaxanthin.	(Acevedo et al., 2014). [183]
β -carotene	All encapsulation structures, except those obtained from high-speed homogenization of whey protein concentrate emulsions, increased the bioaccessibility of β-carotene after in vitro digestion, which was insignificant in its free form.	(Gómez-Mascaraque et al., 2017) [51].

performance and versatility in food packaging. The use of edible packaging materials filled with functional lipophilic ingredients have shown to effectively inhibit the growth of bacteria in packed food [175], produce packaging bio films with antioxidant activity [111] and inhibit the growth of microorganism and prolong the shelf lives of foods [176].

Drying technologies are often used for the encapsulation of sensitive ingredients, but drying may also be considered a stabilisation technique because it removes the liquid phase, which can be a potential source of instability, and converts the emulsion into a more stable solid or powdered form [23]. Drying can also enhance the long-term storage stability of emulsions by reducing the water activity and inhibiting microbial growth. However, it is important to note that the choice of drying technology should consider the specific properties of the emulsion, such as its composition, viscosity, and thermal sensitivity. Optimisation of the drying process parameters, including temperature, residence time, and drying medium, is crucial for maintaining emulsion quality and stability during the drying process.

The final particle size and shape depend on the stabilisation technique. Micrographs of the final stabilized and encapsulated emulsions obtained using the four different technologies are shown in Fig. 4. Fig. 4A shows the resveratrol emulsion powder obtained by spray drying. Rounded soft particles of several sizes were obtained. Fig. 4B shows the freeze-dried emulsions. Several additional porous structures were obtained. The size depended on the post-milling operation. Fig. 4C shows an emulsion of highly oleic palm oil that was dried using a refractance window. Flakes of several sizes with homogeneous structures were obtained using this technique. Fig. 4D shows an O/W Pickering emulsion stabilized with cellulose. This is a liquid with an emulsion stabilized by solid particles. The drop size depended on the emulsification technique used. These stabilisation techniques can be used individually or in combination, depending on the specific emulsion system and desired properties. The choice of technique depends on factors such as the nature of the components, application, and desired shelf-life of the emulsion.

5. Digestibility of lipophilic functional ingredients

Food digestion is a complex and multistage process that has recently attracted the interest of the food industry owing to the development of links between food and health or diseases [177]. During human digestion, two main processes occur simultaneously: (i) mechanical transformations that reduce the size of food particles, and (ii) enzymatic transformations where macromolecules are hydrolysed into smaller constituents that are released from the food matrix during digestion to become available for absorption (bioaccessibility) and are therefore absorbed from the digestive tract and utilised by the body (bioavailability) [178].

To evaluate the bioavailability of phytonutrients of interest, food digestion can be studied using in vitro or in vivo models. In vitro digestion assays simulate the physiological conditions of the human gut and are useful tools for studying and understanding the changes, interactions, and bioavailability of nutrients. In vivo models refer to the process of food digestion that occurs within a living organism. Ideally, the study of food digestion should be conducted in vivo, considering the complexity of the gastrointestinal tract. However, obtaining data on the human gut is challenging. The variations observed from one individual to another, invasive techniques required, and ethical and financial aspects involved make in vivo testing almost impossible [179]. In vitro digestion helps overcome these drawbacks. Human gastrointestinal tract can be seen as 4 in series reactors, each with specific conditions of pH, residence time, enzymes and fluids which can be replicated in vitro models [180], Fig. 5 shows the relevant regions and their conditions typically simulated *in vitro* models.

In vitro models can be static, semidynamic, or dynamic. Static models simulate the stages of digestion using different solutions that mimic the physiological conditions of the gastrointestinal tract. Specific digestive enzymes such as amylase, pepsin, and pancreatic enzymes are used, and parameters such as pH and temperature are controlled. Food was mixed with the digestive solutions and incubated for specific periods to simulate oral, gastric, and intestinal digestion [180,181]. is the most widely used approach for assessing the bioaccessibility of compounds. They have simplified the highly complex processes of human digestion. Consequently, they can be used in most laboratories as they do not require complex equipment or analyst training. Examples of the static in vitro digestion methods used to evaluate the bioaccessibility of different compounds are presented inTable 5.

Although the simplicity of the static approach is a major advantage, it fails to capture the dynamic nature of in vivo digestive

processes. Various efforts have been made to study digestion under physiologically relevant conditions. The goal of this study was to provide physiologically relevant kinetic information on nutrient partitioning patterns and structural changes in food [180]. Therefore, the semi-dynamic in vitro technique involves a gradual pH change in the digestive tract, especially in the gastric phase, secretion of enzymes and digestive fluids, and gastric emptying, making the semi-dynamic technique suitable for investigating the structural changes in food during digestion, such as the effect on the breakdown of the food matrix and absorption of nutrients [179].

Although static models are simple and easy to use, they are inaccurate. It is difficult to recreate the entire dynamic environment of the human body and the flow of food through the different compartments in a real digestive system. Several dynamic models, such as the dynamic gastric model (DGM), human gastric simulator (HGS), gastric digestion simulator (GDS), dynamic gastrointestinal simulator (SIMGI) dynamic gastrointestinal digester (DIDGI), TIM, and simulator of the human intestinal microbial ecosystem (SHIME), have been explored [182]. Such models are much more complex and expensive, require large amounts of enzymes and samples, and necessitate the use of specific equipment that is not normally available in the laboratory.

6. Final remarks

The use of emulsions offers an effective approach for incorporating functional lipophilic compounds into food matrices, addressing the challenges posed by their sensitivity to environmental factors, processing conditions, and gastrointestinal environments. By encapsulating these compounds in emulsions, their protection from external stressors is enhanced, and their passage through the gastrointestinal tract is improved. This method increases solubility, facilitates controlled release, and ultimately boosts the bioaccessibility and bioavailability of these beneficial compounds. Emulsions therefore represent a promising strategy for maximizing the health benefits of lipophilic functional ingredients in food products. Future trends in the field may involve the development of more advanced and sustainable emulsification technologies, such as the use of natural emulsifiers and innovative encapsulation materials. Additionally, the integration of emulsified lipophilic compounds into novel food formats and delivery systems may expand their applicability and impact on consumer health. Further research into the optimisation of emulsion-based delivery systems will likely lead to improved stability, targeted release, and enhanced functionality, opening new opportunities for the incorporation of lipophilic functional ingredients in a wider range of food products.

Formulations should be considered to achieve an efficient emulsification process. Regarding wall materials, plant-based proteins have gained increasing interest in recent years because of their advantages over animal-based proteins. Additionally, it is recommended to employ more than one natural polymer in emulsion formulations, as they enhance emulsion stability by forming complexes among themselves. Blends of proteins and polysaccharides have shown positive results when functional lipophilic ingredients were encapsulated in emulsions.

Inclusion of digestion studies for emulsions is essential for developing effective functional encapsulation systems. It is crucial that protection systems for functional lipophilic ingredients not only safeguard them during processing but also ensure their resilience against the stomach environment. This allows the ingredients to reach areas where they can be absorbed by the digestive system and become bioavailable for use in various metabolic pathways within the human body. Conducting in vitro digestion studies on emulsions is a growing trend that can provide valuable insights into their stability and performance. This type of research is especially important for emulsions stabilized by drying techniques, offering a clearer understanding of how these systems function during digestion. Future research may focus on refining in vitro digestion models to better mimic human digestive processes and improving the predictability of in vivo outcomes. Additionally, combining in vitro digestion studies with other characterization techniques may provide a more comprehensive understanding of emulsion stability and behaviour in the digestive tract. Such advances will facilitate the development of more robust and effective encapsulation systems for functional lipophilic ingredients.

Emulsions are inherently thermodynamically unstable, making the study of technologies for their stabilisation a critical area of research. While the use of insoluble solid particles (Pickering emulsions) has been extensively explored, drying technologies offer an alternative approach by removing the aqueous phase, effectively preventing the movement of oil droplets and avoiding emulsion destabilization. Drying processes, which encapsulate emulsions by removing the liquid phase and preventing oil droplet coalescence, can also be considered as stabilisation methods. The exploration of innovative drying technologies, such as electrohydrodynamic drying, microwave-assisted drying, and infrared drying which offer rapid and uniform drying while maintaining product quality, holds considerable promise in emulsion stabilisation. An interesting option to stabilized and carrying lipophilic functional ingredients are the edible films. This application can be improved with technologies such as electro spinning.

Modifications to conventional drying methods such as the application of vacuum pressures in refractance window drying, the combination of different drying techniques such as convective drying and microwave drying can offer improved preservation of emulsion structure and bioactive content during drying. Given the central role of emulsions in various sectors such as food, cosmetics, and pharmaceuticals, improving their stability and shelf life remains a critical focus. These advanced drying techniques may contribute to achieving these objectives by minimizing heat damage and preserving sensitive bioactive ingredients. Their application in emulsion processing could result in safer, more effective, and more sustainable products. Continued investigation into these and other novel drying methods will play a key role in advancing the use of emulsions across industries and enhancing their overall performance.

Moreover, optimising operating conditions to minimize the degradation of functional compounds of interest will be crucial to ensuring the quality and efficacy of the final product. The challenge lies in balancing efficiency and cost-effectiveness with the preservation of the nutritional and bioactive properties of emulsions. As technology advances, there is potential for the emergence of new drying methods that meet these objectives and enhance the stability and functionality of emulsions for various applications.

Future research should prioritize exploring novel natural ingredients for encapsulating functional oils through emulsification, as this is increasingly important in the modern food and pharmaceutical industries. With consumer preferences trending towards clean

labels and sustainable practices, the search for effective nature-derived encapsulants has gained significant importance. These ingredients not only provide enhanced stability and controlled release of bioactive compounds but also align with the rising demand for healthier and more eco-friendly products.

CRediT authorship contribution statement

Alejandra Henao-Ardila: Writing – original draft, Investigation, Formal analysis. **María Ximena Quintanilla-Carvajal:** Writing – review & editing, Formal analysis, Conceptualization. **Fabián Leonardo Moreno:** Writing – review & editing, Supervision, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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