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Advances in Double Emulsion Technology for Encapsulation and Controlled Delivery System

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Abstract

Double emulsions, commonly referred to as "emulsions of emulsions," have garnered considerable interest in the food, pharmaceutical, and biotechnology industries owing to their unique structural complexity and functional versatility. These systems, particularly water-in-oil-in-water (W/O/W) configurations, offer a promising platform for the co-encapsulation of both hydrophilic and hydrophobic bioactive compounds, the development of low-calorie food products, and targeted drug delivery. However, the inherent thermodynamic instability of double emulsions remains the primary challenge limiting their widespread commercial exploitation. This review comprehensively examines the physicochemical properties of double emulsions, the conventional and emerging fabrication techniques, the mechanisms of instability, and the effects of processing parameters and storage conditions on emulsion stability. Special emphasis is placed on the applications of double emulsions in food formulation, including encapsulation of vitamins, minerals, and bioactive compounds, fat replacement, and development of polymersomes. Case studies on curcumin stability and vitamin D3 encapsulation using psyllium gum are also discussed. The review concludes with future perspectives highlighting the need for novel stabilization strategies to advance the commercial viability of double emulsions.

Keywords: Double emulsions, W/O/W, emulsification, stability, encapsulation, food formulation, fat replacer, bioactive compounds

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Introduction

The modern food consumer increasingly demands products that are healthy, convenient, and nutritionally wholesome. In response to this evolving demand, food technologists and scientists have explored novel delivery and structuring systems capable of improving the bioavailability of nutrients, reducing caloric density, and enhancing sensory appeal. Among such systems, double emulsions have emerged as a highly versatile platform with substantial potential across food, pharmaceutical, and cosmetic applications.

An emulsion is a dispersed system comprising two or more immiscible liquids, with one liquid dispersed as droplets within another. Conventional examples include butter (W/O) and ice cream (O/W). Double emulsions, in contrast, are structured systems in which the dispersed droplets themselves contain smaller, enclosed droplets of a different phase, hence the descriptor "emulsion of emulsion." The most technologically relevant double emulsion type is the water-in-oil-in-water (W/O/W) configuration, in which small inner aqueous droplets (W1) are enclosed within oil droplets, which are in turn dispersed in an outer aqueous continuous phase (W2). An analogous but less commonly exploited configuration is oil-in-water-in-oil (O/W/O).

The key advantage of double emulsions over conventional single emulsions lies in their ability to simultaneously encapsulate hydrophilic and hydrophobic compounds within a single delivery vehicle. Single emulsions are generally unstable and have limited capacity for encapsulating water-soluble compounds. Double emulsions overcome these limitations through their multi-compartmental architecture (Tyowua *et al*., 2017). However, the complexity of this architecture also renders double emulsions thermodynamically unstable, susceptible to multiple degradation mechanisms including coalescence, Ostwald ripening, and phase inversion.

This review provides a comprehensive account of the physicochemical properties, fabrication strategies, stability challenges, and food application of double emulsions, drawing on peer-reviewed literature to synthesize current knowledge and identify areas requiring further research.

Properties of Double Emulsions

Particle Size

Particle size is a fundamental parameter governing the physical stability, optical appearance, and functional performance of double emulsions. It is collectively measured through dynamic light scattering (DLS) using an optical detector, wherein light passed through the sample is scattered and analysed to determine droplet diameter. The mean particle size of emulsion droplets is characterized by the volume-weighted mean diameter (d4,3) and the surface-weighted mean diameter, also known as the Sauter mean diameter (d3,2) (Giroux *et al*., 2016). These metrics reflect different aspects of the droplet size distribution and are particularly relevant for emulsions intended for encapsulation, where the surface area influences the release rate of encapsulated compounds.

Zeta Potential

Zeta potential (ζ, mV) is the electrical potential measured at the shear plane of an emulsion droplet, the boundary where the counter-ions remain strongly attached to the droplet surface in an electric field (Bamba *et al*., 2018). A high absolute zeta potential value indicates strong electrostatic repulsion between droplets, which resists aggregation and confers physical and biochemical stability to the emulsion system. This phenomenon is also referred to as electrostatic stabilization. Measurement of zeta potential typically requires the double emulsion to be diluted approximately one hundred-fold, and values are strongly influenced by the pH and the type of emulsifier present in the system (Prichapan *et al*., 2021). More negative zeta potential values are generally associated with greater stability.

Microstructure

The microstructure of a double emulsion directly influences the fabrication quality and the controlled release profile of encapsulated active substances. Two major techniques are employed to assess double emulsion microstructure: Confocal Laser Scanning Microscopy (CLSM) and Scanning Electron Microscopy (SEM). In CLSM, fluorescent dyes are used to visualise the aqueous and oil phases, aqueous regions typically appear dark while oil regions appear bright (Gharehbeglou *et al*., 2019). SEM, on the other hand, provides information on the surface morphology of emulsion droplets or dried microcapsules, revealing a smooth, relatively spherical and asymmetrical surface for particles of varying sizes (Reichhardt & Parsek, 2019).

Encapsulation Efficiency

Encapsulation efficiency (EE) is a measure of the extent to which a double emulsion can retain the encapsulated compound within its core. It is commonly determined by centrifugation of the emulsion to separate the free (non-encapsulated) compound, followed by spectrophotometric quantification of the released material (Assadpour *et a*l., 2016; Maghamian *et al*., 2021). High encapsulation efficiency is desirable for minimising the loss of bioactive material during processing and storage.

Viscosity

Viscosity is a critical rheological parameter for double emulsions and is significantly affected by shear rate and dispersed phase concentration. Double emulsions can exhibit either Newtonian or non-Newtonian (specifically shear-thinning) behavior depending on the dispersed phase concentration. At low dispersed phase concentrations, emulsions generally behave as Newtonian fluids, wherein viscosity remains constant regardless of shear rate. As dispersed phase concentration increases, or at higher shear rates, viscosity decreases, a hallmark of non-Newtonian, pseudoplastic behavior. The addition of buffers and salts to the inner aqueous phase creates osmotic gradients that influence the migration of the outer aqueous phase inward, causing droplets to pack more closely and thereby increasing viscosity. Phosphate buffer and NaCl are commonly added to the inner phase to equilibrate osmotic pressure, minimise inter-phase transfer, reduce dielectric constant, and decrease droplet collision frequency.

Fabrication of Double Emulsions

The fabrication of double emulsions involves the sequential stabilisation of two distinct interfaces- the primary interface between the inner dispersed phase and the middle phase, and the secondary interface between the middle phase and the outer continuous phase. Emulsifiers are indispensable in this process: lipophilic emulsifiers with a hydrophilic–lipophilic balance (HLB) of 2–7 stabilise the primary W/O interface, while hydrophilic emulsifiers with an HLB of 6–16 stabilise the secondary O/W interface (Nguyen & Assadi, 2018; Saffarionpour & Diosady, 2021).

Two-Step Emulsification

The conventional two-step emulsification method is the most widely employed strategy. In the first step, a W/O or O/W single emulsion is prepared using a high-speed homogenizer or disperser with an appropriate hydrophobic or hydrophilic emulsifier. In the second step, this primary emulsion is re-emulsified into an outer aqueous or oil phase under gentle homogenisation conditions to prevent droplet disruption and maintain the integrity of the inner compartment (Chong *et al.*, 2015).

Phase Inversion Technique

The phase inversion technique involves the addition of water to a pre-formed W/O emulsion until a change in phase volume concentration triggers the spontaneous formation of an intermediate W/O/W emulsion. This approach is also termed catastrophic phase inversion or the emulsion inversion point method. A related strategy; the phase transition temperature technique which involves heating the W/O emulsion to form a lamellar phase, followed by rapid cooling (Saffarionpour & Diosady, 2021).

Membrane Emulsification

Membrane emulsification is an advanced technique enabling the production of monodisperse double emulsions with tuneable droplet size distributions. The dispersed inner phase is forced under low pressure through a microporous membrane into a continuous phase. Two modes exist: direct membrane emulsification, in which the dispersed phase is pumped through the membrane under pressure, and premix membrane emulsification, in which a coarse emulsion is passed across a packed bed of microparticles to reduce droplet size (Vladisavljević, 2019). This technique is particularly advantageous for sensitive bioactives that cannot withstand the shear forces of conventional homogenisation.

Stability of Double Emulsions

Double emulsions are inherently thermodynamically unstable systems that tend to degrade through a variety of physical and chemical mechanisms. The stability of a double emulsion is affected by numerous factors including the type and concentration of emulsifiers, dispersed phase concentration, processing conditions, and storage environment (Aditya *et al*., 2014). Instability manifests through mechanisms such as flocculation, coalescence, creaming, sedimentation, Ostwald ripening, and phase inversion.

Effect of Composition

At high dispersed phase concentrations, viscosity increases and droplets pack densely, promoting droplet size growth and emulsion instability. Conversely, electrolytes incorporated in the inner aqueous phase enhance repulsive forces between inner water droplets, thereby improving stability. The balance between osmotic pressure and interfacial tension is critical in determining the extent of water transfer across the oil film separating the inner and outer aqueous phases (Evageliou *et al*., 2019).

Lipid Oxidation and Phase Inversion

Phase inversion, the conversion of a W/O emulsion to an O/W emulsion or vice versa, constitutes a primary instability event in double emulsions. Lipid oxidation is another major concern; oxidative reactions at the oil droplet surface generate off-flavors and off-colors, while reactive intermediates promote further emulsion destabilization through prooxidant–substrate interactions at the oil–water interface (Berton-Carabin *et al*., 2014).

Effect of Homogenization Pressure and Shear

Homogenization pressure critically determines final droplet size, zeta potential, and encapsulation efficiency. Excessive pressure which is a phenomenon termed over-processing, promotes coalescence and Ostwald ripening, increasing particle size. At high shear rates, the emulsifier is unable to adsorb sufficiently onto the newly generated droplet surfaces, and elevated temperatures induced by shear can denature protein-based emulsifiers, leading to reduced electrostatic stabilization and increased coalescence (Wang *et al.*, 2015). A balanced shear speed is therefore essential for producing stable double emulsions.

Thermal and Storage Stability

Thermal treatment promotes an increase in droplet size and favors bimodal size distributions. The incorporation of biopolymers within the inner aqueous phase has been shown to improve thermal stability by physically reinforcing the inner interface. During storage, instability in double emulsions is primarily driven by water diffusion, coalescence, and Ostwald ripening. Fat-containing double emulsions are particularly prone to storage instability due to the high melting point of the lipid phase (Cofrades *et al*., 2016).

Applications of Double Emulsions in Food Formulation

Encapsulation of Bioactive Compounds

Many bioactive compounds of nutritional and nutraceutical importance suffer from poor stability, low solubility, limited bioavailability, and susceptibility to degradation during food processing. Double emulsions offer a means of protecting and delivering such compounds. Spray drying is the most widely used encapsulation method due to its scalability, low water activity of the product, and suitability for transport and storage, though it is associated with poor agglomeration of microcapsule powder and limited wall material options (Eun *et al*., 2019). Freeze-drying preserves sensitive encapsulants well but entails high energy costs and long processing times. Chemical methods such as complex coacervation involving phase separation of oppositely charged polymers at a stable pH, are particularly useful for lipophilic materials such as turmeric oleoresin, palm oil, and β-carotene, achieving loading capacities of 40–90% (Shaddel *et al*., 2018; Giri *et al*., 2013; del Carmen Razola-Díaz *et al*., 2021).

Specific bioactive compounds encapsulated using double emulsions include epigallocatechin gallate (EGCG), the predominant catechin in tea (Evageliou *et al.*, 2019); glycyrrhizic acid, a natural sweetener derived from liquorice root (Maghamian *et al*., 2021); and oleuropein, a hydrophilic phenolic compound responsible for the bitterness of olive leaf extract (Souilem *et al.*, 2014).

Fortification with Vitamins and Minerals

Water-soluble vitamins including vitamin C, vitamin B12, and riboflavin are highly susceptible to oxidative and photodegradative damage during food processing and storage. Double emulsions offer an effective delivery vehicle for these vitamins, enhancing their stability and controlled release. Riboflavin deficiency remains prevalent in developing nations, and its photo-lability necessitates protection through encapsulation (Kheynoor *et al*., 2018). For minerals, rancidity associated with iron fortification is a major quality concern, and encapsulation using double emulsions mitigates the unacceptable color and flavor changes. Synergistic effects between co-encapsulated compounds, such as the enhanced intestinal absorption observed for calcium combined with vitamin D3 further justify multi-compound double emulsion formulations (Dima & Dima, 2018, 2020).

Drug Delivery

In pharmaceutical applications, double emulsions produced using two-step emulsification avoid the immediate dissolution of active ingredients into the aqueous phase, resulting in extended dissolution times and prolonged biological half-life. This approach addresses challenges such as bitter taste, unpleasant odor, poor stability, and low bioavailability of various drugs. Double emulsions also prevent the premature degradation of sensitive nucleotides and peptides, while imparting improved therapeutic efficacy with reduced systemic side effects (Iqbal *et al* ., 2015).

Development of Low-Calorie Foods

Double emulsions are a powerful tool for reducing sugar and fat content in foods without compromising sensory quality. Sweeteners encapsulated within double emulsions possess significantly higher sweetness indices than those in single emulsions, enabling sugar reduction. Encapsulation also mitigates common drawbacks of artificial sweeteners such as off-flavors, aftertaste, hygroscopicity, and thermal instability, for instance, improving the thermostability of aspartame (Ilyasoglu Buyukkestelli & El, 2021). As fat replacers, double emulsions formulated with polysaccharides, gums, or pectin can replace up to 80% of the fat in ice cream and up to 60% of fat in meat systems, while maintaining similar sensory characteristics and providing a healthier fatty acid profile (Eisinaite *et al* ., 2017).

Polymersomes and Microreactions

Beyond encapsulation, double emulsions serve as templates for the fabrication of polymersomes- polymer-based vesicular structures used in controlled drug and gene delivery. They also provide a scalable and controllable environment for conducting millions of monodisperse micro-reactions at the nanolitre scale. The coalescence efficiency of emulsion droplets permits precise control over reaction conditions, protecting ongoing reactions from external disturbances (Hou *et al*., 2017).

Research-based comparative table

Fabrication method	Typical droplet size	Encapsulation efficiency (%)	Advantages	Disadvantages	Industrial applicability
Microfluidization	30–200 nm	85-99	Produces highly uniform droplets, excellent stability, continuous process	Expensive equipment, multiple passes often require	Excellent industrial scalability for food and pharmaceuticals
Ultrasonication	20–200 nm	75-98	Rapid emulsification, simple laboratory setup, low solvent requirement	Difficult scale-up, localized heating, probe erosion	Mainly laboratory and pilot scale; limited industrial use
Phase Inversion Temperature (PIT)	20–100 nm	85–99	Very small droplets, low energy, good stability	Applicable mainly to nonionic surfactants, temperature-sensitive process	Cosmetic and pharmaceutical formulations
Membrane emulsification	1–10 µm	85-98	Uniform droplet size, low shear, suitable for sensitive biomolecules	Low production rate, membrane fouling	Specialty pharmaceutical and food applications
Microfluidics	10–100 µm	90-100	Precise droplet control, excellent reproducibility, monodispersity	Very low throughput, expensive fabrication	Research, precision drug delivery, diagnostics

Current Knowledge Gaps in Double Emulsions

Despite extensive research, several challenges limit the commercial translation of double emulsions (DEs). The primary knowledge gap is poor long-term stability, as DEs are susceptible to coalescence, flocculation, Ostwald ripening, creaming, and osmotic water transfer between the internal and external aqueous phases, leading to leakage of encapsulated compounds and reduced shelf life. Another critical gap is the limited understanding of encapsulation and controlled release mechanisms, particularly under processing, storage, and gastrointestinal conditions, where interfacial transport and osmotic effects remain difficult to predict. In addition, scalable fabrication technologies that maintain uniform droplet size and high encapsulation efficiency are still lacking, as high-energy methods compromise droplet uniformity, whereas low-energy techniques and microfluidics suffer from low throughput and high production costs. The development of sustainable, food-grade emulsifiers capable of providing long-term stability without affecting product quality also remains an important research priority. Furthermore, the absence of standardized protocols for measuring droplet size, encapsulation efficiency, stability, and release kinetics limits comparison among studies. Finally, in vivo validation, regulatory assessment, and economic feasibility studiesare scarce, highlighting the need for comprehensive investigations to facilitate industrial adoption of double emulsions in food, pharmaceutical, and cosmetic applications.

Future Perspectives and Conclusion

Double emulsions represent an eco-friendly and functionally versatile technology with demonstrated potential for the encapsulation of diverse nutrients, the development of natural food systems, and targeted delivery of bioactive and pharmaceutical compounds. Their ability to act simultaneously as fat replacers, sweetness enhancers, and delivery vehicles for both hydrophilic and hydrophobic compounds position them as a transformative tool in food formulation and nutraceutical science.

Nevertheless, the long-term thermodynamic instability of double emulsions remains the principal challenge constraining their commercial-scale application. The multiple instability mechanisms coalescence, Ostwald ripening, phase inversion, and lipid oxidation interact in complex ways that are not yet fully understood. Future research must focus on the development of novel stabilization strategies, including the use of plant-based biopolymers and natural emulsifiers, Pickering stabilization using solid particles, and the optimization of processing parameters through advanced modeling and real-time monitoring techniques.

Advances in microfluidics and membrane emulsification offer promising avenues for producing monodisperse double emulsions with precisely controlled droplet architectures. Coupling these fabrication advances with encapsulant-specific release engineering could unlock novel applications in personalized nutrition, functional foods, and precision drug delivery. It is anticipated that with continued interdisciplinary research, double emulsions will transition from laboratory-scale curiosity to mainstream commercial food processing technology.

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