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Applications of Mixed Surfactant Systems in the Construction and Regulation of High Internal Phase Emulsions (HIPEs)

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Abstract. High internal phase emulsions (HIPEs) are widely used in fields such as porous materials and food delivery, owing to their solid-like viscoelastic properties. Single low-molecular-weight surfactant systems require high emulsifier dosages and have limited biocompatibility, whereas Pickering particle systems face limitations such as difficulty in wettability regulation and insufficient stability at ultra-high internal phase fractions. Surfactant mixing, through synergistic effects among components, has become an important route for constructing high-performance HIPEs. This review summarizes the structural characteristics, preparation methods, and typical applications of HIPEs, compares the limitations of the two types of single stabilization systems, elaborates the synergistic stabilization mechanisms of the two types of mixed systems, and analyzes the regulation patterns of mixing ratio, concentration, environmental conditions, and emulsification process on the microstructure and macroscopic properties of HIPEs. Composite systems primarily enhance the compactness and mechanical strength of interfacial films through synergistic interactions between components at the interface; specifically, small-molecule combinations rely on interfacial molecular rearrangement, whereas particle-surfactant combinations achieve stability mainly through the modulation of wettability and the complementarity of the two-component interfacial film. This review can provide a reference for the rational design of mixed systems; however, the quantification of mechanisms and the development of green components still require further investigation and represent important directions for future research.

Keywords: High internal phase emulsions (HIPEs), Surfactant mixed systems, Synergistic stabilization

1. Introduction

High internal phase emulsions (HIPEs) refer to concentrated emulsion systems with an internal phase volume fraction exceeding 74%. Owing to their extremely high internal phase content, they exhibit solid-like viscoelastic properties and structural stability, and hold broad application prospects in porous materials, food delivery, cosmetics, and biomedicine [1]. The mainstream strategies currently employed to construct stable HIPEs can be categorized into three types: stabilization by low-molecular-weight surfactants, Pickering particle stabilization, and stabilization by surfactant mixed systems.

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Conventional HIPEs are mostly stabilized by low-molecular-weight surfactants such as Span 80 and CTAB. Because their interfacial adsorption is reversible and they are prone to desorption, relatively high dosages are required, which not only increases cost and poses safety risks but also limits their application in food and pharmaceutical fields.

Pickering particles stabilize emulsions through irreversible interfacial adsorption, which can reduce surfactant dosage and improve emulsion stability. However, single-particle systems have obvious limitations: inorganic particles are difficult to degrade, bioderived particles require modification, and their stabilizing performance in ultra-high internal phase systems is limited [2].

Surfactant mixing mainly includes two directions: the combination of different low-molecular-weight surfactants and the combination of surfactants with particles [1]. For surfactant–surfactant systems, combinations of nonionic and ionic surfactants can produce significant synergistic effects—nonionic components can insert between the head groups of ionic surfactants, weakening electrostatic repulsion and promoting compact packing of the interfacial film, thereby enhancing interfacial film strength and resistance to coalescence [1]. For surfactant–particle systems, Zou et al. successfully achieved Pickering HIPEs with an internal phase volume fraction of 98.5% by combining hydrophobic silica nanoparticles with Span 85 [3]. Each of the three routes has its applicable scenarios, but mixed systems offer the greatest tuning potential owing to their flexible designability and the wide selection of raw materials.

Nevertheless, research on mixed systems remains insufficiently systematic: on the one hand, there is no unified comparative framework for the applicable conditions and stabilization mechanisms of different mixed combinations; on the other hand, a systematic review of how the structural parameters of individual components in mixed systems synergistically affect interfacial assembly behavior and the macroscopic properties of HIPEs is still lacking [1].

Against this background, this review focuses on the application of surfactant mixed systems in the construction and regulation of HIPEs. It first briefly summarizes the mechanisms and limitations of the two types of single stabilization systems, then analyzes the synergistic stabilization mechanisms and key regulating factors of different types of mixed systems, generalizes the structure–property relationships between mixing parameters and the microstructures and macroscopic properties of HIPEs, and finally provides an outlook on future directions.

2. Overview of high internal phase emulsions

2.1. Definition and microstructural characteristics of HIPEs

HIPEs refer to concentrated emulsions with an internal phase volume fraction exceeding 74.05%, a critical value corresponding to the limit of the closest packing of monodisperse spherical droplets. As the internal phase fraction further increases, droplets are forced to squeeze and deform into polyhedral configurations, leaving only extremely thin continuous-phase films between them. Compared with conventional dilute emulsions, HIPEs possess a huge interfacial area, thin films, and mutually constrained droplets, and thus exhibit pronounced solid-like viscoelastic properties, with the ability to self-support, be molded, and resist phase separation over the long term [1, 4]. Droplet size, polydispersity, and interfacial film structure jointly determine the macroscopic properties: the more uniform the droplets and the denser the interfacial film, the more stable the system [1, 5].

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2.2. Preparation methods of HIPEs

HIPEs are mainly prepared by one-step emulsification, two-step emulsification, and concentration methods. One-step emulsification is simple and convenient to operate, being the most commonly used method; it typically employs high-shear homogenization combined with stepwise dropwise addition of the internal phase, in which the internal phase is added in batches to the continuous phase containing the emulsifier under continuous shearing to form a concentrated emulsion [5]. The shear intensity, dropping rate, and emulsifier dosage directly determine the droplet size and its distribution. However, this method suffers from high energy consumption and a broad droplet size distribution, and mismatches between the dropping rate and shear intensity readily trigger local coalescence and make size control difficult. Two-step and concentration methods can improve the droplet size distribution, but the processes are complex [5]. In comparison, membrane emulsification and microfluidic techniques can precisely control droplet size and monodispersity, yet their low throughput and high scale-up costs still pose challenges for industrial application [1, 5].

2.3. Application fields of HIPEs

On the basis of their high internal phase and solid-like characteristics, HIPEs are widely applied. In the preparation of porous materials, HIPEs can be used as templates for polymerization or drying to obtain PolyHIPEs with highly interconnected pore structures, which are of significant value in fields such as catalyst supports, adsorbent materials, and tissue engineering scaffolds [4, 6]. In the food sector, HIPEs can serve as fat replacers and delivery vehicles for bioactive factors: the high-internal-phase, solid-like rheological properties can mimic the texture and mouthfeel of fat, maintaining the system volume and sensory quality while reducing oil usage, and the dense interfacial film can isolate oxygen and light, inhibiting oxidative degradation of bioactive factors [5, 7, 8]. In cosmetics and biomedicine, HIPEs are used to construct cream matrices and sustained-release carriers: the high internal phase fraction imparts a rich, fine texture to creams; the solid-like characteristics and dense interfacial film improve product stability; and, after being encapsulated in the internal phase droplets, active ingredients can be released slowly through the barrier effect of the interfacial film, achieving a sustained-release effect.

3. Single stabilization systems and their limitations

3.1. Stabilization of HIPEs by low-molecular-weight surfactants

Conventional HIPEs mostly rely on low-molecular-weight surfactants such as Span 80, CTAB, SDS, and PGPR for stabilization. Their stabilization mechanism depends on the rapid adsorption of molecules at the oil–water interface and the reduction of interfacial tension. However, the adsorption–desorption equilibrium is reversible and the anti-coalescence ability of the interfacial film is limited; moreover, as the internal phase volume fraction increases, the required surfactant concentration rises markedly, typically reaching 5%–20% or even higher [2, 5]. The high dosage increases cost and raises concerns about residual toxicity, restricting its application in food and pharmaceutical fields. Food-grade W/O emulsifiers such as PGPR are also limited by their synthetic origin and dosage constraints [8].

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3.2. Stabilization of HIPEs by pickering particles

Solid particles adsorb at the oil–water interface in a nearly irreversible manner, with desorption energies far exceeding the thermal motion energy, and can form a dense particulate interfacial layer that enhances the anti-coalescence ability of droplets through steric hindrance and an interfacial network, thereby stabilizing HIPEs at relatively low dosages [2, 9]. Commonly used particles include inorganic particles such as silica and nanoclays, as well as bioderived particles such as cellulose, starch, and proteins. However, single-particle systems have obvious drawbacks: inorganic particles are difficult to degrade and have poor biocompatibility, whereas bioderived particles mostly require hydrophobic modification to improve wettability and provide insufficient interfacial coverage under ultra-high internal phase conditions [2, 9, 10]. These limitations have driven research toward mixed strategies: combining different types of surfactants can exploit synergistic adsorption and ordered arrangement between molecules to strengthen the interfacial film and improve stability at lower total concentrations, thereby compensating for the deficiencies of single systems. This has become an important research direction for the stability regulation of HIPEs.

4. Surfactant mixed systems

4.1. Surfactant–surfactant mixed systems

4.1.1. Nonionic/ionic combinations

The combination of ionic and nonionic surfactants is the most extensively studied class. Its synergistic stabilization mechanism lies in the insertion of the head groups of nonionic surfactants between the charged head groups of ionic surfactants, which weakens electrostatic repulsion between like charges, promotes compact and ordered packing of interfacial film molecules, and improves interfacial film strength and resistance to coalescence [1, 11]. Vilasau et al. confirmed that ionic/nonionic mixed systems can significantly improve the stability of paraffin emulsions, and that the mixed composition and emulsification process jointly determine the final performance [11]. Recent studies have further correlated interfacial molecular packing with macroscopic stability: Esposito et al. found that the degree of interfacial film densification in Span 80/Tween 80 systems corresponds closely to stability [12]; Wang et al. tuned the system HLB value toward the value required by the oil phase by adjusting the Span 80/Tween 80 ratio, achieving optimization of droplet size and stability [13]. In addition, combining small-molecule emulsifiers with structuring agents (e. g., co-stabilization by monoglyceride oleogel and PGPR) can significantly improve freeze–thaw stability while maintaining the W/O high internal phase structure, demonstrating the potential of mixed strategies in food-grade systems [14].

4.1.2. Other mixed types

Besides ionic/nonionic combinations, nonionic–nonionic mixtures can also produce synergistic effects: Bian et al. stabilized W/O HIPEs with a binary nonionic system of Span 80 and Triton X-100, in which the two surfactants co-adsorbed at the interface and strengthened the interfacial film, and the resulting emulsions exhibited excellent performance in coal slime flotation [15]. Anionic–cationic combinations possess an extremely low critical micelle concentration and more compact interfacial packing owing to the strong electrostatic attraction between oppositely charged head groups, but they are highly sensitive to the charge ratio, prone to precipitation and phase separation,

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and studies under high internal phase conditions remain scarce [1]. From the molecular mechanism perspective, the essence of synergistic stabilization in all types of mixed systems lies in the complementary adsorption and ordered arrangement of different surfactant molecules at the oil–water interface; the matching degree of molecular structure, HLB value, and charge properties determines the degree of interfacial film densification and thus affects macroscopic stability [12]. Different mixed types have their own applicable boundaries: ionic/nonionic combinations are the most mature but sensitive to composition ratio; nonionic–nonionic combinations are mild and safe, better suited to food scenarios; anionic–cationic combinations exhibit compact interfacial packing but are constrained by charge balance. Therefore, the rational design of mixed systems should optimize the composition and ratio according to the application scenario [1, 13].

4.2. Surfactant–particle combinations

4.2.1. Surfactant/inorganic particle combinations

Inorganic particles form a rigid particulate layer at the oil–water interface through nearly irreversible interfacial adsorption, but pure particle systems impose stringent requirements on the wettability matching of both the oil and water phases and often suffer from insufficient interfacial coverage under high internal phase conditions. Adsorption of a small amount of surfactant onto the particle surface can in situ tune the particle wettability and reduce interfacial tension, thereby promoting the migration and spreading of particles at the interface; the particles in turn provide a steric barrier formed by irreversible adsorption. The synergy between the two enables simultaneous enhancement of interfacial coverage and film strength at relatively low total dosages. Binks et al. confirmed that cationic surfactants can regulate the interfacial wetting behavior of silica nanoparticles and improve long-term stability [16]; Zou et al. constructed Pickering-type HIPEs with an internal phase fraction as high as 98.5% using hydrophobic silica and Span 85 [3]; Guan et al. found that pH can induce reversible transitions in the interfacial assembly behavior of lecithin–silica systems, thereby enabling regulation of the emulsion microstructure and the type of W/O/W double emulsions [17]. However, inorganic particles themselves are difficult to degrade, and their application remains constrained by biocompatibility.

4.2.2. Surfactant/biobased particle combinations

Compared with inorganic particles, bioderived particles such as lignin, cellulose, and proteins offer the advantages of being renewable, degradable, and biocompatible. However, native bioderived particles are usually too hydrophilic and have limited ability to stabilize HIPEs alone, so they often need to be combined with surfactants [9]: the surfactant adsorbs onto the particle surface through hydrophobic interactions, regulating particle wettability and promoting interfacial adsorption, thereby constructing stable high internal phase emulsions at relatively low dosages. Song et al. prepared Pickering-type HIPEs by combining ball-milled lignin with Tween 80 and found that the addition of the nonionic surfactant significantly improved the interfacial wettability and adsorption of lignin particles [10]; Yang et al. combined egg white protein with rhamnolipid, and the synergistic adsorption of the two components endowed the interfacial film with higher elasticity and excellent storage stability of the emulsions [7]; Kim et al. found that cellulose nanocrystals can markedly delay catastrophic phase inversion through interfacial jamming effects, and their synergistic interactions with surfactants of different charges determine emulsion stability [18]. However, bioderived particles are strongly influenced by the raw material source and pretreatment process,

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with pronounced batch-to-batch variability, and their long-term stability under demanding high internal phase conditions still requires improvement.

4.3. Generalization of synergistic stabilization mechanisms

Taking the surfactant–surfactant and surfactant–particle mixed systems together, the synergistic stabilization mechanisms can be classified into two categories. The first is interfacial molecular co-adsorption and rearrangement among small molecules: different surfactant molecules co-adsorb at the oil–water interface and reconstruct the molecular packing; ionic/nonionic combinations weaken electrostatic repulsion through the insertion of nonionic head groups, whereas nonionic/nonionic combinations optimize HLB matching through complementary molecular structures, both centered on increasing the densification of the interfacial film [11, 12]. The second is wettability regulation and construction of composite interfacial films in particle–surfactant systems: while reducing interfacial tension, the surfactant in situ tunes the particle wettability and promotes particle interfacial adsorption, whereas the particles compensate for the deficiency of the molecular film with a rigid steric barrier formed by irreversible adsorption, building a particle–molecule complementary composite interfacial film [16]. Although the two modes differ in composition, both inhibit droplet coalescence by enhancing the density and mechanical strength of the interfacial layer, achieving high internal phase stability at relatively low total dosages, which explains the general advantage of mixed strategies over single systems [19]. However, the synergistic effects of mixed systems are highly sensitive to environmental factors such as pH, ionic strength, and temperature; the boundary between synergy and antagonism among components remains unclear and lacks quantitative criteria, representing a weak link in current research.

5. Regulation of HIPE properties by mixing parameters

5.1. Effects of mixing ratio and concentration on microstructure

The total concentration and component ratio of the mixed system determine the microstructure of HIPEs: when the total emulsifier concentration is insufficient, interfacial coverage is inadequate and droplets are prone to coalescence; when the concentration is too high, a large number of micelles form, unnecessarily increasing the viscosity and cost of the system [19]. The component ratio directly determines the molecular packing mode in the interfacial layer: an excessively high proportion of ionic components introduces strong electrostatic repulsion that hinders compact molecular packing at the interface, whereas an excessively high proportion of nonionic components weakens the synergistic effect; only within an appropriate ratio window can HIPEs with small droplet size and narrow size distribution be obtained [11, 12]. An optimal ratio also exists in particle–surfactant systems: too little surfactant fails to effectively regulate particle wettability, while too much surfactant causes particles to be wrapped and detached from the interface, losing the Pickering stabilization advantage [16].

5.2. Correlation between structural parameters and macroscopic properties

Microstructural parameters—the average droplet diameter, polydispersity index, and interfacial film composition—determine the macroscopic properties of HIPEs: the smaller and more uniform the droplets, the larger the specific surface area and the denser the films, the stronger the viscoelasticity and anti-coalescence ability, and the better the storage stability [5]. The interfacial film composition

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is the core link between microstructure and macroscopic behavior: in small-molecule mixed systems, the more ordered the interfacial molecular packing, the stronger the resistance of the emulsion to environmental disturbance; in particle–surfactant mixed systems, the formation of a continuous particle–surfactant mixed network at the interface endows the system with stronger solid-like rheological behavior, which is favorable for preparing PolyHIPE materials with controllable structures [4].

5.3. Regulating factors

Besides formulation parameters, environmental conditions and the emulsification process also exert significant regulatory effects on the properties of HIPEs stabilized by mixed systems. pH alters the charged state of ionic surfactants and bioderived particles, affecting electrostatic interactions among interfacial molecules and thereby changing the synergistic stabilization effect [7]; studies on lecithin–silica systems have also shown that pH can induce reversible transitions of interfacial assembly and regulate the microstructure of HIPEs [17]. Salt ions weaken electrostatic repulsion by compressing the electric double layer: a moderate salt concentration promotes compact packing of interfacial molecules, whereas an excessive concentration induces emulsion flocculation and, by weakening the interfacial antioxidant barrier, aggravates lipid oxidation [8]. Temperature changes the solubility and HLB value of surfactants, directly regulating interfacial adsorption behavior, and the stability of mixed interfacial films under freeze–thaw cycling is particularly critical [14]. Regarding the emulsification process, the shear rate and the dropping rate of the internal phase determine the competition between droplet breakup and coalescence: insufficient shearing yields oversized droplets, whereas excessive shearing destroys the already-formed interfacial film, and even an optimal formulation cannot produce uniform and stable HIPEs under such conditions [5, 11]. Therefore, the preparation of HIPEs stabilized by mixed systems is a complex process coupling formulation, process, and environment, and controllable preparation can only be achieved through synergistic optimization.

5.4. Summary of key parameters and evaluation indexes for practical design

To strengthen the guiding value for practical formulation design, the key experimental parameters and commonly used evaluation indexes for mixed-system-stabilized HIPEs are summarized in Table 1. In general, the total emulsifier dosage, component ratio, and processing conditions should be optimized jointly rather than separately. The selection of evaluation indexes should also match the application scenario: storage stability and rheological properties are critical for food and cosmetic applications, while interfacial microstructure and mechanical strength are more relevant for porous material templating.

Table 1. Summary of key experimental parameters and evaluation indexes for different mixed systems

Mixed system type	Typical combination	Total emulsifier dosage	Optimal ratio window	Key evaluation indexes	Ref.
Ionic/nonionic surfactant	SDS/Tween 80, Span 80/CTAB	2-8 wt%	HLB matching oil phase; nonionic fraction = 0.3-0.6	Storage stability, droplet size, interfacial tension	[11-13]
Nonionic/nonionic surfactant	Span 80/Triton X-100	3-10 wt%	Synergistic adsorption range	Stability in flotation, rheology	[15]

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Table 1. (continued)

Surfactant/inorganic particle	Silica + cationic surfactant	Particle 1-5 wt% + surfactant 0.1-1 wt%	Contact angle ≈ 90°	Long-term stability, wettability, microstructure	[3, 16, 17]
Surfactant/bioderived particle	Lignin/Tween 80, cellulose NC + surfactant	Particle 1-3 wt% + surfactant 0.5-2 wt%	Surfactant wets particle surface	Pickering stability, phase inversion behavior	[10, 18]

5.5. Practical considerations: reproducibility, cost, and safety

Reproducibility. The reproducibility of HIPE preparation remains a key bottleneck for scale-up. Because HIPEs are highly sensitive to shear rate, internal-phase dropping speed, and temperature, small variations in processing conditions can lead to significant differences in droplet size and stability [5, 11]. In mixed systems, the synergistic effect further depends on the precise ratio and order of addition, which introduces additional batch-to-batch variability. Standardized protocols-including pre-hydration of surfactants, controlled temperature during emulsification, and fixed shear history-are therefore essential for reliable scale-up.

Cost. Mixed systems generally offer a cost advantage over single-component systems. In surfactant-surfactant combinations, synergistic interfacial adsorption reduces the total emulsifier dosage required compared with using a single surfactant. In surfactant-particle combinations, solid particles partially replace surfactant molecules, further lowering the total emulsifier loading to 2-5 wt% while maintaining stability [10, 16]. Bioderived particles such as lignin and cellulose are also relatively low-cost and renewable, making them particularly attractive for large-scale applications.

Safety risks. Safety considerations vary by application field. In food and pharmaceutical applications, residual synthetic surfactants (e.g., Triton X-100, CTAB) exhibit cytotoxicity and are strictly regulated. Both types of mixed systems can address this issue: surfactant-surfactant combinations can be formulated with food-grade emulsifiers such as lecithin and monoglyceride oleogels [7, 14], while surfactant-particle combinations can use bioderived particles that are biodegradable and biocompatible. Inorganic silica particles, although effective, are non-degradable and raise long-term biosafety concerns. Overall, green surfactants and bioderived particles represent a promising direction for safe and sustainable HIPE design.

Table 2. Comparison of reproducibility, cost, and safety among three stabilization systems

Criterion	Single surfactant system	Single Pickering system	Mixed systems (surfactant-surfactant / surfactant-particle)
Reproducibility	Moderate; sensitive to concentration and temperature	Low-moderate; particle batch variability	Moderate; requires precise ratio control; surfactant-particle systems also depend on particle batch consistency
Cost	High dosage→high cost	Moderate; modification steps add cost	Lower total dosage through synergy; cost-effective for both combination types
Safety	Residual toxicity; limited food/pharma use	Inorganic particles non-degradable	Both types can use food-grade or bioderived components

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Table 2. (continued)

Scalability	Mature process	Low throughput; membrane/microfluidic costly	Promising but needs standardized protocols for both combination types

6. Conclusion

This review summarizes the research progress on surfactant mixed systems in the construction and regulation of high internal phase emulsions. Both single low-molecular-weight surfactant systems and Pickering particle stabilization systems have inherent limitations, whereas the two types of mixed strategies can achieve interfacial film reinforcement and high internal phase stability at relatively low total dosages through synergistic interactions among components. The essence of synergy in mixed systems lies in improving the density and mechanical strength of the interfacial layer: small-molecule combinations rely on interfacial molecular rearrangement, while particle–surfactant combinations rely on wettability regulation and the complementarity of the two-component interfacial film. The mixing ratio, total concentration, environmental conditions, and emulsification process jointly determine the microstructure and macroscopic properties of HIPEs, providing a basis for the rational design of mixed systems. Current research on mixed systems still mainly relies on empirical formulation screening, and quantitative criteria for the synergy–antagonism transition remain to be established. Future research should focus on the quantification of mixing mechanisms and the development of green, degradable components, promoting their application in food, pharmaceutical, and porous material fields.

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