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Study on Stability and Application of Pickering Emulsion

Yujing Zhou

School of Chemistry and Chemical Engineering, Hunan Institute of Science and Technology, Yueyang, Hunan, 414006, China

ABSTRACT
Pickering emulsion is an emulsion stabilized by ultrafine solid nanoparticles instead of traditional emulsifiers developed in the last two decades, with strong stability and high safety. At present, it has a good application prospect in cosmetics, food, medicine, agriculture, petroleum and other fields.

KEYWORDS
Pickering emulsion; Stabilization mechanism; Stabilizing factors; Double phase; Application

1 Introduction

In recent years, the application and demand for emulsions have been increasing across various production and daily life fields [1]. An emulsion is a dispersion system composed of two immiscible liquids, where the dispersed phase (i.e., the internal phase) is dispersed as droplets within the continuous phase (i.e., the external phase) [2-3]. Influenced by the free energy of liquid-liquid interface systems [4], emulsions exhibit thermodynamic instability [2], making them prone to phenomena such as demulsification and coalescence. To enhance product stability, emulsifiers must be added to reduce oil-water interface tension and minimize energy consumption during emulsification [5]. For decades, researchers have explored emulsifiers, with traditional ones primarily being surfactants like sodium dodecylbenzenesulfonate (SDBS) and sodium lauryl sulfate (SLS). Pickering emulsions are a category of emulsions stabilized by extremely fine solid nanoparticles . Since their discovery in the early 20th century by Ramsden [6] and Pickering [7], this field has become a focal point for extensive research and exploration. Traditional emulsions typically rely on surfactants or surfactant substances for stabilization, but these often suffer from issues like strong irritancy, accumulation of ionic impurities, poor biodegradability, and low stability. Consequently, Pickering emulsions have gradually replaced some traditional emulsions in production and daily applications in recent years due to their non-irritating, high safety, environmental friendliness, and superior stability. Pickering emulsions are distinguished by their outstanding stabilizing properties. In recent years, with the rapid advancement of nanotechnology, researchers have conducted more detailed investigations on Pickering emulsions using various advanced equipment. Existing studies indicate that the stabilization mechanism of Pickering emulsions can be summarized as solid particle-stabilized emulsions composed of three phases: water, oil, and solid particles. These are generally categorized into water-in-oil (O/W) and oil-in-water (W/O) types. Additionally, there are special emulsion systems such as W/O/W, O/W/O bicontinuous types, and water-in-water (W/W) systems [8]. The stabilization mechanism of Pickering emulsions primarily involves the formation of a film structure at the oil/water interface by solid particles. This film prevents droplet merging and phase separation, thereby maintaining the emulsion's stable state. As shown in the Figure1:

The applicability of Pickering emulsions is closely tied to their stabilization mechanisms. By understanding these mechanisms, we can optimize their structure and properties to expand their applications. This paper examines three stabilization mechanisms in Pickering emulsions, synthesizes literature on factors influencing their stability and phase

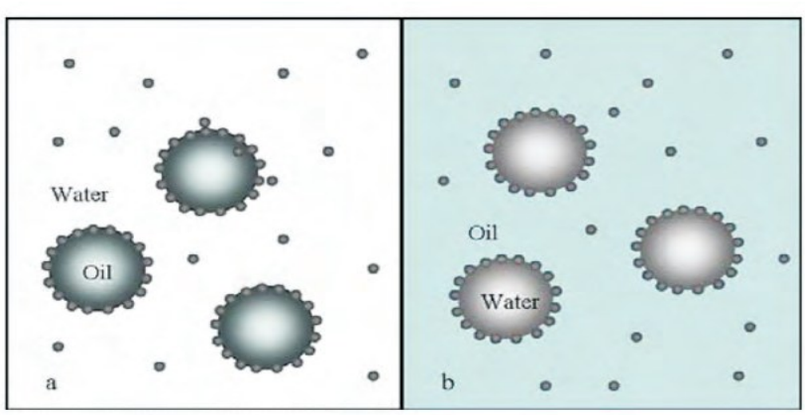

Figure 1 Schematic of Pickering emulsion stability mechanism(a)O/W emulsion(b)W/O emulsion

transition characteristics, and details their diverse industrial applications. Finally, it outlines current challenges in Pickering emulsion technology while offering insights into its future development prospects.

2 Stability mechanism of pickering emulsion

2.1 Interface film theory

The interfacial film theory describes how solid particle emulsifiers spontaneously aggregate at oil-water phase interfaces, forming compact and elastic single or multi-layer membrane structures [9]. These structures create spatial steric hindrance that prevents droplet collisions. The elastic properties of the films also ensure droplets remain intact during impacts, undergoing only localized deformation rather than rupture. This mechanism effectively prevents droplet aggregation and emulsion demulsification, thereby enhancing emulsion stability. The structural integrity relies on interparticle interactions including van der Waals forces, dipole repulsion, and Coulombic forces. In a two-dimensional hexagonal packed single-layer structure, particles maintain stable positioning at interfaces through equilibrium between attractive and repulsive forces, driven by their higher free energy at the phase boundary [10]. As shown in the Figure2:

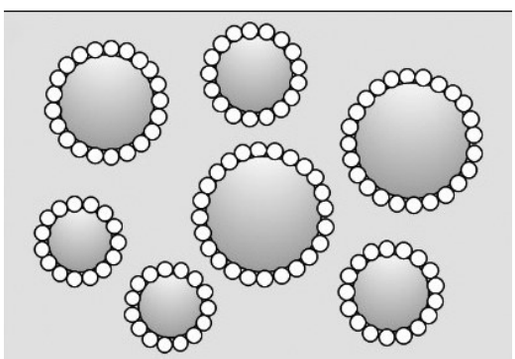

Figure 2 Schematic diagram of interfacial membrane theory

2.2 Contact angle theory

Flinke[11-12] pointed out in 1923 that the key factor enabling solid emulsifier particles to effectively stabilize emulsions is their surface wettability. Surface wettability refers to the spontaneous interaction between solid particles and oil/water phases, where both phases spread across the particle surface, meaning the particles become wetted. However, oil and water differ in their spreading capabilities on solid surfaces. Most particles preferentially wetting the more favorable phase causes the interface to bend toward the less-wettable phase, forming droplets that determine emulsion types. Wettability is measured by the contact angle θ between solid particles and liquid phases. When $\theta<90°$, particles are preferentially wetted by water, forming O/W emulsions; conversely, when $\theta>90°$, they are preferentially wetted by oil, forming W/O emulsions[13]. See Figure3:

From an energy perspective, Blinks demonstrated through research that the adsorption of Pickering emulsion particles at oil/water interfaces is irreversible. This occurs because the energy required for particle desorption at the interface far

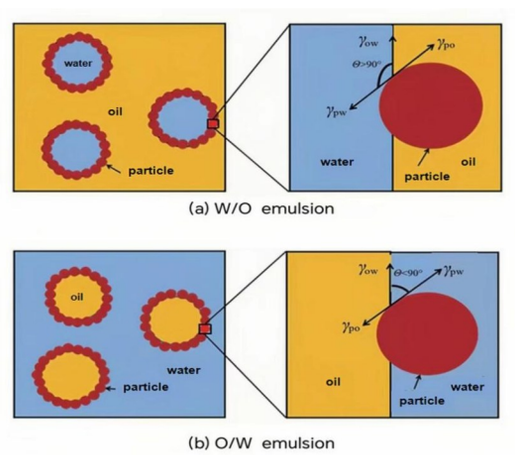

Figure 3 Microscopic schematic diagram of O/W and W/O Pickering emulsion.(a)W/O emulsion,(b)O/W emulsion,γ_{po}—particle-oil interfacial tension; γ_{pw}—particle-water interfacial tension; γ_{ow}—oil-water interfacial tension

exceeds their thermal energy, making particles reluctant to detach. This characteristic contrasts with surfactant molecules, which exhibit dynamic adsorption-desorption within short timeframes (dynamic equilibrium at interfaces)[14]. When solid particles reach the oil/water interface, they enter one phase and form a new two-phase interface. The energy change during this process is expressed as $\Delta E= \pi R^2 \gamma ow(1\pm\cos\theta)^2$ (Equation 1), where R is the particle radius, γ_{ow} represents the interfacial tension between oil and water, and the "+" sign indicates particle migration from the original interface into the oil phase, while the "-" sign denotes migration into the water phase. A negative ΔE value corresponds to adsorption, whereas a positive value with the same absolute magnitude indicates desorption. Chen Zhao et al. calculated the relationship between desorption energy and contact angle θ for particles with a diameter of 1×10^{-8} m and γ_{ow} of 5×10^{-2} N/m, as shown in the Figure4. With θ=90° as the boundary, desorption energy initially increases before decreasing. At θ=90°, desorption energy reaches its maximum value, indicating maximum emulsion stability due to firm particle adhesion. Using 1000KT as the benchmark, higher values above 1000KT indicate stronger surface activity, while values below 1000KT suggest weaker activity. Particles exhibit higher surface activity at θ=60°-120°, lower activity at θ=30°-60° or θ=120°-150°, and negligible surface activity at θ<30° or θ>150° [15]. In summary, contact angle θ determines emulsion type and particle surface activity, serving as the key factor in maintaining Pickering emulsion stability.

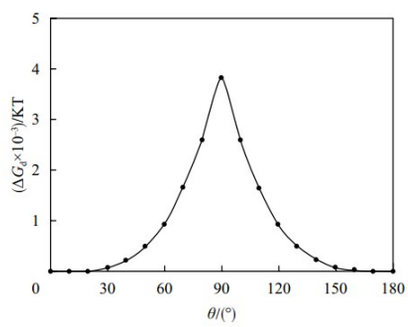

Figure 4 Desorption free energy of a nanoparticle from hydrocarbon/water interface as a function of its contact angle

2.3 Three-dimensional network structure theory

Three-dimensional network structures can be categorized into two types: ① In Pickering emulsions, some particles remain unadsorbed on the liquid interface. These particles interact with each other to form highly elastic three-dimensional networks between particles. This structure reduces collisions and aggregation among dispersed droplets while increasing viscosity of the continuous phase, a common phenomenon in clay-based solid particle-stabilized Pickering emulsions [13,16]. ② Adhered solid particles also interact with adjacent droplet particles. Under their influence, interconnected particles bridge neighboring droplets, forming shared three-dimensional network structures that enhance both viscosity and stability of the emulsion [17-18].See Figure5:

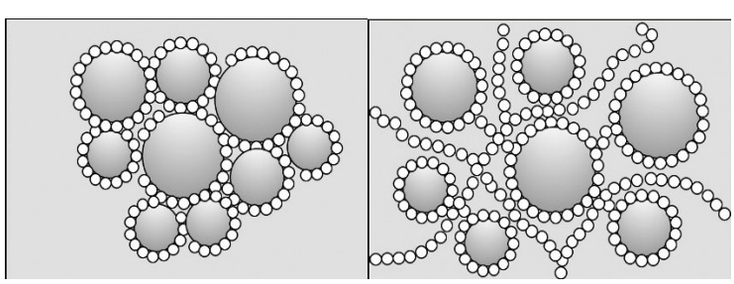

Figure 5 The diagram of Three-dimensional network structure ,(a)three-dimensional network structure formed by particle-particle interaction,(b) three-dimensional network structure formed by bridging different droplets

3 Factors affecting pickering emulsion stability

The influencing factors of Pickering emulsion stability are mainly the wettability and contact angle θ of solid particles, the size of solid particles, in addition to the type of solid particles, pH, oil-water ratio [13,19] etc.

3.1 Types of solid particles

3.1.1 Organic solid particles and inorganic solid particles

The most common types of solid particles in stabilized Pickering emulsions are inorganic and organic particles.

Commonly used organic solid particles for preparing Pickering emulsions include proteins, starch, ultra-fine soybean meal, chitosan, and various modified organic solid particles. Inorganic solid particles include: SiO_2, Ag, nanoTiO_2, graphene oxide, and most modified inorganic particles [13,20]. The inherent or modified properties of different solid particles influence the performance of the prepared Pickering emulsion. For example, Wang Sen and Chen Ying et al. modified nanoTiO_2 particles with salicylic acid to form TiO_2-SA particles with excellent amphiphilic properties, which were then used to prepare mosquito-repellent microcapsules through the Pickering emulsion method, achieving an encapsulation rate of 84.02% [21]. Biomacromolecules like proteins inherently possess good biocompatibility. After modification, they gain hydrophobic and lipophilic properties, forming hydrophobic-lipophilic aggregates. Through specific interactions, these aggregates combine with various protein molecules to form spherical, rod-shaped, and other interfacial layer structures. The resulting Pickering emulsion effectively encapsulates poorly water-soluble substances like curcumin, a method now widely applied in pharmaceutical research [22].

3.1.2 Organic-inorganic nano-composite particles

To impart specific properties to Pickering emulsions, two complementary organic polymers and inorganic nanomaterials can be combined through fine structural design into organic-inorganic composite nanoparticles, endowing the final product with broader application potential. For instance, polyacrylamide molecules contain abundant-NH2 groups that can form chemical bonds with Hg^{2+} ions, enabling effective adsorption. The modified SiO2 not only exhibits excellent surface wettability but also demonstrates adsorption capacity for Hg^{2+}. By integrating these advantages, Zhang Kui and colleagues developed polyacrylamide (core) -silica (shell) composite microspheres using an inverse-phase Pickering emulsion polymerization method. They employed acrylamide aqueous solution as the internal phase, liquid paraffin as the external phase, and modified silica particles as stabilizers. When 0.1g of composite particles were tested with 150mL of Hg^{2+} wastewater at an initial concentration of 20μg/mL, the results showed that the composite microspheres achieved saturation adsorption capacity significantly higher than silica particles. The adsorption process was rapid, reaching equilibrium within 30 minutes. This composite has been applied in Hg^{2+} adsorption and separation research [23].

3.1.3 Janus particles

Janus particles are functional materials characterized by two or more distinct chemical compositions or properties on their surfaces [13,24]. This asymmetry primarily manifests in shape and properties. In terms of morphology, they exhibit spherical, dumbbell-shaped, fibrous, and other configurations. These materials also feature adjustable structural zones such as positive/negative charges, polar/neutral regions, and hydrophilic/hydrophobic areas. Regarding properties, they demonstrate differences in optical, electrical, thermal, magnetic, and surface wetting characteristics [25]. Beyond surface activity, Janus particles possess amphiphilic properties. Blinks and Fletcher adjusted the partitioned area on Janus particle surfaces to modify their amphiphilicity. Comparative studies with conventional solid particles revealed that when the average contact angle Δθ (the average of two distinct contact angles formed by different zones at the phase interface) reaches approximately 90°, their desorption efficiency triples that of ordinary solid particles. Even when Δθ approaches 0° or 180°, they maintain strong surface activity [26]. Consequently, compared to stable emulsions of conventional solid particles, Janus particles exhibit significantly enhanced stability and surface activity. Today, Janus particles have found extensive applications in environmental monitoring, medical fields, sensors [19], catalysts, and other domains.

3.2 PH

The water phase pHvalue significantly affects the average droplet size in emulsions, which in turn impacts their stability. Larger droplet sizes indicate aggregation and reduced stability, while smaller droplets maintain better stability. Taking a Pickering emulsion stabilized by ultra-fine soybean dregs as an example: when the oil phase fraction was 0.6 and the soybean dregs particle fraction remained fixed at 1%, adjusting the water phase pH value between 3-8 and leaving the emulsion undisturbed for 7 days. As shown in the Figure6 ，measurements showed an initial increase followed by a decrease in average droplet size across different pH values. In acidic conditions (pH=3-6), the growth rate slowed due to the high charge frequency at the adsorption interface caused by electrostatic repulsion from soybean dregs ζ -electrostatic potential. The emulsion remained relatively stable under these conditions, but reached its maximum average droplet size at pH=7, leading to coalescence and decreased stability [27].

3.3 Oil-water ratio

The oil-to-water ratio not only affects emulsion stability but may also trigger phase transitions that alter emulsion properties. In O/W emulsions, increasing the oil-to-water ratio increases the average particle size, whereas in W/O emulsions, it decreases it. For example, in O/W emulsions, when the oil-to-water ratio rises while the solid emulsifier content remains constant, the emulsion fails to fully coat droplet surfaces with an interfacial film. Without adequate surface coverage, droplets tend to aggregate and collide due to surface interactions, thereby compromising emulsion stability [13,27].

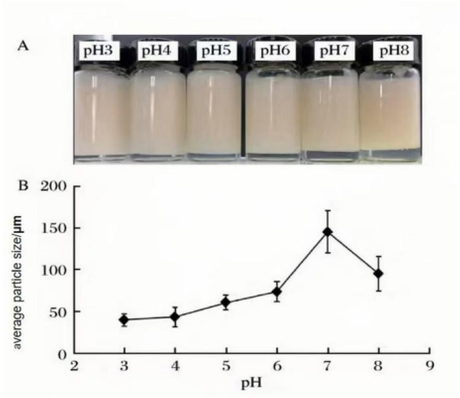

Figure 6 Digital images of emulsions prepared under different aqueous pH values(sampled after 7 d settlement)and relationship between the average particle size of the emulsion and aqueous pH values

4 Phase transition of emulsion

Affected by oil to water ratio, emulsifier concentration, temperature and other factors, the emulsion will have a phase transition phenomenon in which the internal phase and the external phase transform into each other. In the production process, it is very important to control the phase transition of emulsion.

4.1 Mutation phase shift and transition phase shift

Emulsion phase inversion is typically categorized into two types: abrupt phase inversion and gradual phase inversion. Abrupt phase inversion occurs by altering the oil-to-water ratio while maintaining constant emulsifier concentration, causing the internal phase to transition to the external phase. Gradual phase inversion, however, involves modifying the emulsifier's particle properties or composition while preserving the oil-to-water ratio [28-30]. The critical parameter determining phase inversion is known as the phase transition point, which is determined by the emulsifier's characteristics and types, including parameters such as HLB value, HLD, and conductivity. Binks et al. investigated both inversion types and discovered that W/O emulsions stabilized by hydrophobic SiO_2 particles transition to O/W type when the water phase volume fraction reaches 70%, while hydrophilic SiO_2-stabilized O/W emulsions shift to W/O type at 30%. During this process, emulsions demonstrate excellent stability and anti-agglomeration properties, with optimal performance achieved near the phase transition point. In phase transition studies, researchers investigated toluene-water emulsions stabilized by two differently wettable SiO_2particles. When maintaining a 1:1 oil-to-water volume ratio, adding hydrophilic SiO_2particles to an oil-in-water (W/O) emulsion stabilized by hydrophobic SiO_2particles transformed it into an oil-in-water (O/W) type. Conversely, introducing hydrophobic SiO_2particles into an O/W emulsion stabilized by hydrophilic SiO_2particles converted it into a water-in-oil (W/O) type. Both transformations demonstrated stable sedimentation and anti-aggregation properties, though performance deteriorated most significantly at the phase transition point [31].

4.2 Dual rotation phase

Beyond the two aforementioned phase inversion phenomena, there exists a distinct variant known as dual-phase inversion. This phenomenon occurs when solid emulsifying particles and surfactants simultaneously act as emulsifiers in an emulsion. By adjusting parameters such as particle concentration, pH, temperature, or aqueous phase salt concentration, the emulsion undergoes sequential phase transitions: first transitioning from O/W to W/O, then back to O/W [29,31]. Binks et al. first observed this dual-phase inversion in a 1:1 volume ratio of dodecane-water emulsion stabilized by a SiO_2 particle dispersion (Ludox HS-30 particles) and the surfactant di-C10DMAB. As shown in the Figure7, experimental data demonstrate that as di-C10DMAB concentration increases, the emulsion evolves through three distinct phases: initial O/W phase, followed by W/O phase, and ultimately back to O/W. Studies indicate that dual-phase inversion primarily stems from changes in the hydrophilic and hydrophobic properties of emulsifier particles. Through precise surfactant concentration adjustments, both SiO_2 particles and amphiphilic surfactants can induce dual-phase inversion in stabilized emulsions [32-33].

5 Pickering emulsion application

After getting familiar with the stabilizing mechanism, stability factors and phase transition phenomenon of Pickering emulsion, we can effectively regulate its characteristics and apply it widely in various production and life fields.

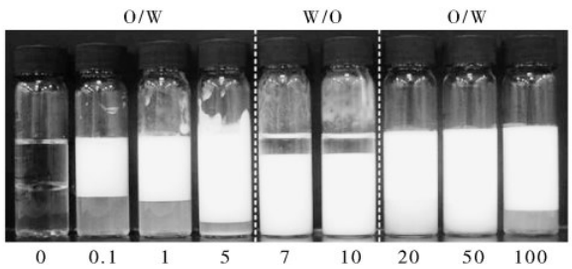

Figure 7 Appearance of dodecane — water emulsions stabilized by a mixture of Ludox HS — 30 silica nanoparticles and di — C10DMAB surfactant

5.1 Medicine

Bioactive substances widely used in the pharmaceutical field, such as nutrients, drugs, and enzymes, are often affected by environmental changes like light exposure and oxygen concentration, leading to alterations in stability and bioavailability. Encapsulation technology utilizes stable carriers to transport active components into a stable system for precise delivery. W/O and W/O/W types of Pickering emulsions can encapsulate hydrophilic active substances, establish stable systems, and ensure accurate drug delivery. For instance, Chen Juanbo et al. encapsulated W/O-type Pickering emulsions in calcium alginate capsules, utilizing the three-dimensional network structure of calcium alginate hydrogel to restrict emulsion movement. This created a carrier system combining Pickering emulsions with calcium alginate hydrogel, which can carry hydrophilic active substances while forming multi-layer protective structures like calcium alginate gel layers, particle interface membranes, and an outer oil phase to prevent external interference [34]. Li Taining mentioned in a medical research review that curcumin, a crucial active ingredient in anti-tumor fields, achieves a bioavailability of 65.67% when encapsulated by self-organized membrane structures formed through succinylation and glycosylation of whey-modified proteins, significantly enhancing drug efficacy [22].

5.2 Food sector

In today's world, growing public concern over food safety and health has led to increasingly stringent market demands for product quality, shelf life, and packaging. In recent years, Pickering emulsions have gained traction in the food industry due to their exceptional stability. Chitosan, a hallmark of food packaging materials, offers advantages like non-toxicity, biodegradability, and excellent film-forming properties, though it still lacks sufficient antibacterial and antioxidant capabilities. To fully leverage chitosan's potential, researchers Zhou Chuang et al. selected two natural components: gallic acid (GA), an antioxidant metabolite from plants, and zein—a hydrophobic macromolecular biopolymer capable of self-assembly into nanoparticles. They modified zein with GA to create a conjugated compound Zein-g-GA that exhibits notable antioxidant and stabilizing properties. Using this modified compound as an emulsifier, they formulated a Pickering emulsion loaded with cinnamon essential oil. By combining this cassia bark-essential oil Pickering emulsion with a chitosan solution, they developed a chitosan-active coating liquid. Experimental applications on mango surfaces demonstrated that this coating effectively shields fruits from environmental influences while significantly reducing respiration intensity, minimizing moisture and nutrient loss, delaying ripening, and ensuring optimal storage quality [35].

5.3 Cosmetics

Traditional cosmetics using surfactants as emulsifiers often suffer from issues like high irritation, keratin layer itching, and short shelf life. The application of Pickering emulsion effectively addresses these problems while aligning with current cosmetic R&D needs. Firstly, its solid emulsifier particles are predominantly derived from biopolymers and natural solid particles, demonstrating excellent biocompatibility and high safety [18,20,36]. Secondly, the interfacial film formed by these emulsifier particles not only maintains emulsion stability and viscosity but also encapsulates and protects photosensitive, pH-sensitive, and ion concentration-sensitive bioactive ingredients (such as nicotinamide and salicylic acid, which are whitening efficacy components). This minimizes external interference while enabling precise control over active ingredient release location and speed through thickness and structure regulation of the interfacial film, achieving targeted delivery, reduced irritation, and extended product shelf life [18,36-37]. Additionally, the efficacy of emulsifier particles themselves is crucial. For instance, nanoTiO2 particles exhibit excellent anti-aging properties, enhancing sun protection and powder adhesion, making them commonly used in sunscreen formulations [38]. Starch particles, with their fine texture, create more skin-friendly creams [39-40], while volcanic mud can emulsify water-insoluble vitamin E [41], enabling its effective release in cosmetics.

5.4 Paper industry

Emulsification technology has long been a cornerstone of paper chemical production. Today, emulsion-based

additives are extensively utilized in the paper industry, with emulsifiers predominantly consisting of inorganic solid particles that are widely available and easily manufactured [42]. In papermaking, the treatment of sizing agents is particularly critical, as it directly affects the paper's liquid barrier properties. Researchers led by Zhao Zhenhuan employed Pickering emulsification technology using nanoTiO2 particles as stabilizers to stabilize industrial-grade sizing agent ASA emulsions. Experimental results demonstrated that nanoTiO2-stabilized ASA emulsions showed improved sizing performance, with paper sizing degree significantly enhanced when the dosage increased from 0.4% to 0.5%. Additionally, they developed a synergistic stabilization system combining nanoTiO2 particles and chitosan for sizing agent AKD emulsions. Their study revealed that these emulsions not only exhibited excellent stability but also achieved high sizing efficiency at lower concentrations, with optimal sizing levels maintained through precise dosage control. Unlike surfactants that tend to generate foam during production—a condition detrimental to paper machinery—and are difficult to degrade, Pickering emulsions not only ensure superior paper quality but also align with modern eco-friendly manufacturing standards [43].

6 Summary and outlook

While Pickering emulsions outperform surfactant-based formulations in multiple aspects, they still face practical challenges in current applications. Regarding stability, the sustained effectiveness of Pickering emulsions may not be guaranteed, as other functional components and preservatives in formulations could potentially compromise their stability. Moreover, certain molecules inherently lack hydrolytic stability, and emulsifier particles may not effectively address this issue. From a safety perspective, the use of solid nanoparticles remains controversial in human health applications [44-45], particularly in cosmetics where strict control over dosage, duration, and formulation methods is essential. Improper preparation could lead to severe toxic side effects. For instance, nanoTiO2 particles can generate reactive oxygen species (ROS) under photocatalysis, which may damage cell membranes, trigger inflammation, or even harm DNA [44]. Therefore, rigorous safety evaluations and systematic research must be conducted when formulating with Pickering emulsions. Additionally, production challenges persist due to complex procedures, immature technologies, high costs, and difficulties in achieving large-scale manufacturing [13]. It is hoped that future research can find some more environmentally friendly, more practical and better product stability preparation methods, and conduct further research on the stability mechanism and stabilizing factors, and explore more application directions.

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