



A comparative study on the capacity of a range of food-grade particles to form stable O/W and W/O Pickering emulsions



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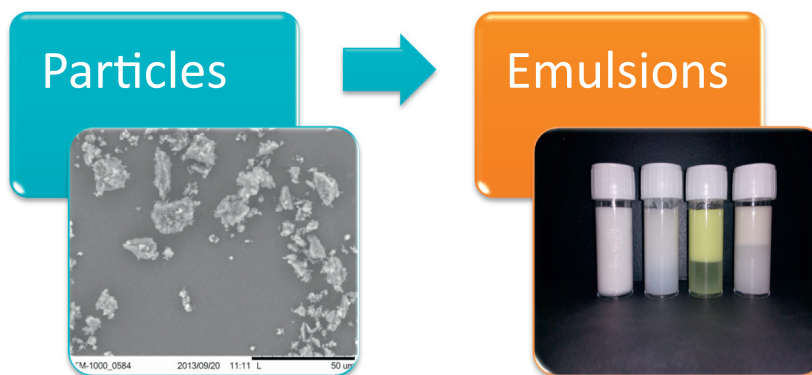
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HIGHLIGHTS

- Comparison of food-grade, as-obtained particles and subsequent emulsions.
- Development of design rules for Pickering particles for food applications.
- Production of stable food-grade Pickering nanoemulsions.

GRAPHICAL ABSTRACT



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ABSTRACT

Whilst literature describing edible Pickering emulsions is becoming increasingly available, current understanding of these systems still suffers from a lack of consistency in terms of the (processing and formulation) conditions within which these structures have been studied. The current study aims to provide a comparative analysis of the behaviour of different edible Pickering candidates and their ability to stabilise emulsion droplets, under well-controlled and uniform experimental conditions, in order to clearly identify the particle properties necessary for successful Pickering functionality.

More specifically, an extensive investigation into the suitability of various food-grade material to act as Pickering particles and provide stable oil-in-water (O/W) and water-in-oil (W/O) emulsions was carried out. Polysaccharide and flavonoid particles were characterised in terms of their size, ζ -potential, interfacial activity and wettability, under equivalent conditions. Particles were subsequently used to stabilise 20% w/w O/W and W/O emulsions, in the absence of added surfactant or other known emulsifying agents, through different processing routes.

All formed Pickering emulsions were shown to resist significant droplet size variation and remain stable at particle concentrations between 2 and 3% w/w. The main particle prerequisites for successful Pickering stabilisation were: particle size (200 nm – 1 μ m); an affinity for the emulsion continuous phase and a sufficient particle charge to extend stability. Depending upon the employed emulsification process, the resulting emulsion formation and stability behaviour can be reasonably predicted a priori from the evaluation of specific particle characteristics.

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1. Introduction

Pickering stabilisation [1,2] has long been recognised as a very useful method for producing emulsions with superior stability to conventional surfactant-stabilised emulsions. Pickering particles accumulate at the oil–water interface in the form of a densely-packed layer that protects against droplet flocculation and coalescence via a steric mechanism. In comparison, surfactants stabilise emulsions largely via an electrostatic mechanism: surfactants, comprised of a hydrophilic head group and a hydrophobic tail component, preferentially adsorb at the oil–water interface, reducing the free energy present when producing a high surface area during emulsification, and hence interfacial tension is reduced, allowing emulsions to form [3,4].

Several factors influencing Pickering emulsion stability have been identified. These include parameters such as oil phase composition, particle characteristics, emulsification technique, and storage conditions. However, arguably the most important factors are concerned with the particle characteristics, in particular particle size and wettability [5]. Recently, it was suggested that three main steps exist for convectional (i.e. non-diffusion controlled) adsorption to the liquid–liquid interface [6,7]. The first step involves particle collisions with free newly created interfacial areas during droplet formation. The second step is concerned with the initial adhesion of particles to this interface, where interparticle electrostatic interactions, as well as particle size properties, are extremely important. Finally, the third step involves water displacement from the particle surface by oil which is dependent upon the particle's contact angle, θ_{ow} , at the interface and hence the hydrophilic/hydrophobic character of the particle [8]. Where $\theta_{ow} < 90^\circ$ (measured through the water phase), the Pickering particle surface mainly resides in the water phase and can be classified as being predominantly hydrophilic; such particles will tend to stabilise an O/W emulsions. Adversely, if $\theta_{ow} > 90^\circ$, the (largely lipophilic) particle will predominantly remain within the oil phase and thus facilitate formation of W/O emulsions. Finally, in those cases where $\theta_{ow} = 90^\circ$, there is no net curvature of the interface and hence no preference for forming a specific emulsion type [9].

Understanding and/or modifying particle characteristics, such as wettability, surface charge, particle size and even surface activity properties, may be then used to control parameters relating to adsorption kinetics, such as the free energy of detachment (E_{det} ; Eq. (1)) of particles from the liquid–liquid interface [10,11]:

$$E_{det} = \pi r^2 \gamma_{ow} (1 \pm \cos \theta)^2 \quad (1)$$

where γ_{ow} is the interfacial tension between the oil and water phases; θ is the particle contact angle (positive or negative depending on which phase contact angle is measured through); and r is the particle radius.

Recently, research has focussed on using edible Pickering particles to stabilise simple emulsions such as hydrocolloids, rather than traditional Pickering particles such as clays [12], polystyrene [5], silica [13] and TiO₂ [14]. These edible hydrocolloid particles include colloidal hydrophobically modified starch particles [15–17], colloidal celluloses and various cellulose derivatives [18–20]. Such polysaccharide structures have been used to stabilise simple oil–water emulsions that were extremely stable to coalescence when compared to surfactant-stabilised systems. Additionally, particle stabilised emulsions were shown to possess an enhanced stability against lipid oxidation as well as an increased tolerance to shear [20,21]. Specifically fabricated colloidal particles have also been shown to successfully stabilise simple emulsions. These include chitosan nanocrystals [22], protein particles [23,24], and certain flavonoids [25,26], in addition

to fat crystals and wax microparticles [27,28] and various other crystalline species [29–31].

However, despite the currently increasing knowledge on the functionality and performance of edible Pickering particles and the emulsions that these structures can subsequently stabilise, a clear disconnect amongst the available literature persists. This is mainly as a result of reported studies differing greatly with respect to a number of important emulsion parameters, such as pH conditions, particle concentration, emulsification process and processing parameters, oil type and particle dispersion methods.

The aim of the present study is to assess the potential of a range of edible particulate structures to function as Pickering particles for the stabilisation of both O/W and W/O emulsions, under well-controlled and uniform (processing and formulation) experimental conditions. The specific particulate species studied were: three modified celluloses (colloidal microcrystalline cellulose, hydroxypropyl methylcellulose and ethylcellulose) and two flavonoids (rutin hydrate and naringin), as they are edible, commercially attractive and, in the case of flavonoids, also linked to particular health benefits [32]. Particles were characterised with regards to their size, ζ -potential, particle surface activity and wettability, while the emulsions that these structures formed were evaluated in terms of their droplet size and stability. Finally, this study assessed potential advantages that these particles would impart on emulsions produced through different processing routes (i.e. high pressure homogenisation or rotor–stator mixer).

2. Materials and methods

2.1. Materials

Distilled water and commercially available sunflower oil (purchased from a local supermarket) were used for the preparation of all emulsions. 100 g samples of emulsions were prepared at 20% w/w dispersed phase volume and materials were used without any further purification or modification. Particles used as emulsifying agents were rutin hydrate, naringin, colloidal microcrystalline cellulose (CMCC), ethylcellulose (EC) and (hydroxypropyl)methyl cellulose (HPMC) and all were obtained from Sigma, UK. Particle concentration and water and oil phase fractions, unless stated otherwise, are given as percentages of the weight of the individual constituent over the total weight of the final emulsion (% w/w).

2.2. Methods

2.2.1. Preparation of Pickering particle dispersions

All particles were introduced to and treated in the continuous phase prior to combination with the dispersed phase to form the emulsions. 80 g of these particle dispersions were prepared and then heated with a hot plate to 45–50 °C for 40 min whilst being agitated with a magnetic stirrer. Following this, particle dispersions were further treated by a high intensity ultrasonic vibracell processor (Sonics & Materials, Inc., CT, USA) operating at 750 W and 20 kHz.

2.2.2. Preparation of particle-stabilised emulsions

Following the particle dispersion, 20 g of the dispersed phase was added to the particle dispersion and the mixture was emulsified using a Silverson L4RT, with an emulsion screen of 19 mm diameter, for 2 min at 10,000 rpm. Following mixing in the rotor–stator mixer (RSM), emulsions were then passed through a high-pressure jet homogeniser (HPH) at 900 bar, where stated.

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