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Phase inversion emulsification: Current understanding and applications

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ABSTRACT

This review is addressed to the phase inversion process, which is not only a common, low-energy route to make stable emulsions for a variety of industrial products spanning from food to pharmaceuticals, but can also be an undesired effect in some applications, such as crude oil transportation in pipelines. Two main ways to induce phase inversion are described in the literature, i.e., phase inversion composition (PIC or catastrophic) and phase inversion temperature (PIT or transitional). In the former, starting from one phase (oil or water) with surfactants, the other phase is more or less gradually added until it reverts to the continuous phase. In PIT, phase inversion is driven by a temperature change without varying system composition. Given its industrial relevance and scientific challenge, phase inversion has been the subject of a number of papers in the literature, including extensive reviews. Due to the variety of applications and the complexity of the problem, most of the publications have been focused either on the phase behavior or the interfacial properties or the mixing process of the two phases. Although all these aspects are quite important in studying phase inversion and much progress has been done on this topic, a comprehensive picture is still lacking. In particular, the general mechanisms governing the inversion phenomenon have not been completely elucidated and quantitative predictions of the phase inversion point are limited to specific systems and experimental conditions. Here, we review the different approaches on phase inversion and highlight some related applications, including future and emerging perspectives.

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1. Introduction

Emulsions, i.e., mixtures of two immiscible liquids in which one is usually present as droplets (dispersed phase) immersed into the other one (continuous phase), are ubiquitous in many industrial applications, e.g., in the food and biomedical sectors. Typically, one phase is organic (the oil phase), and the other is an aqueous solution. In some cases, the two phases are continuously interpenetrated into each other, thus making it impossible to distinguish between the dispersed and the continuous phase. These emulsions are referred to as co-continuous or bicontinuous. Due to oil/water immiscibility, emulsions tend to be phase separated at thermodynamic equilibrium, hence emulsion stability is one of the main problems in several industrial applications. Many phenomena drive phase separation, e.g., creaming and sedimentation due to density difference of the phases, coalescence when two droplets merge into one [1,2], flocculation when droplets tend to aggregate in clusters, and Ostwald ripening when the smaller droplets diffuse into the bigger ones through the continuous phase [3,4]. As shown by Stoke's sedimentation law, reducing the density difference between the phases, lowering droplet radius, and increasing continuous phase viscosity are all factors acting to hinder phase separation, making the emulsion meta-stable, i.e., the separation process "is slow enough" [5]. Such kinetic stability can also be achieved by exploiting surfactants, which are amphiphilic molecules stabilizing droplets by lowering interfacial tension and eliciting Marangoni stresses [6]. The role of surfactants and amphiphiles in general in lowering interfacial tension [7–11], their adsorption behavior as well as the interface behavior both in static and dynamic conditions [12–25] have been widely studied. Emulsions having fine droplet size are more stable against phase separation and widely exploited in application also due to their capability to incorporate hydrophilic or hydrophobic species. Such small-sized emulsions are mainly divided in two broad categories: nanoemulsions and microemulsions. Nanoemulsions have a size range between 20 nm–200 nm [26], can be almost transparent or rather turbid and are kinetically stable. Microemulsions are thermodynamically stable (interfacial tension nearby zero [27]) and transparent with droplet size ranging from 10 to 100 nm. Although this terminology is rather confusing and not always agreed upon in the literature [28], it can be stated that microemulsions exhibit smaller droplet size with respect to nanoemulsion and are thermodynamically stable, while nanoemulsions are kinetically stable.

Several methods are used to obtain emulsions with stability levels suitable to industrial applications. Here, we focus on phase inversion emulsification, a quite popular technique characterized by low energy requirements. In this process, an inversion between the continuous and the dispersed phase is achieved by either changing temperature or composition, thus obtaining finely dispersed emulsions in a more sustainable way with respect to the classical high energy mixing emulsification routes.

The phase inversion pathways have been described as catastrophic or transitional. The former is a term introduced by Salager [29] to describe emulsion inversion reached by changing the water/oil ratio

(this is also referred to as phase inversion composition or PIC, and emulsion inversion point or EIP) [30]. The term catastrophic comes from Dickinson [30–33], following the hypothesis that catastrophe theory might be useful to describe emulsion phase inversion. Despite all the efforts, catastrophe theory can only predict qualitative features of catastrophic phase inversion, but it cannot be used as a predictive model [30,34]. This is due to the lack of a kinetics coupled thermodynamic approach as opposed to a mere thermodynamic one [34]. In transitional phase inversion the inversion is considered to be brought about by changing the surfactant affinity for the two phases [30,35], e.g., non-ionic surfactants becoming more lipophilic when heated (phase inversion temperature or PIT). The latter assumption was stated clearly by Shinoda in 1968 [36].

However, in spite of the widespread use and the extensive investigation of phase inversion (dating from the pioneering work of Shinoda, who in turn refers to earlier work of Langmuir), its governing mechanisms are still debated. As stated by Nienow [37] and Orr [38], notwithstanding all the efforts made in order to model the catastrophic phase inversion, and phase inversion in general, satisfactory models are not available. One of the key issues is the high number of variables, such as concentration of surfactant(s), oil and water, temperature, agitation rate, flow type, vessel geometry, surface wetting and electrostatic charge [33]. The main approaches to model phase inversion in the literature can be related to three main areas: phase behavior, interfacial properties and flow-induced morphological changes, such as droplet breakup and coalescence. Most studies focus on one of these areas at a time and their interplay is still to be fully elucidated.

This review is organized as follows: in the first section phase and interfacial behavior in phase inversion will be reviewed, in the second section studies of flow-induced morphological changes will be considered, finally concluding remarks and future perspectives will be given.

2. The physico-chemical approach to phase inversion

2.1. Phase behavior and its role in phase inversion

Phase behavior plays an essential role in studying emulsion properties. In general, amphiphilic molecules like surfactants or phospholipids show characteristic cooperative association in aqueous solutions leading to supermolecular structures such as micelles in a given surfactant concentration range. Hydrophobic interactions are the driving force of the phenomenon and are important for a thermodynamic description [39,40]. The polar head groups of surfactants are indeed hydrated to allow appreciable water–amphiphile contact in the micelles [39]. An isotropic micellar solution is also named L_1 [41] or L_2 if made of reverse micelles [41]. For higher surfactant concentrations surfactant self-assembly leads to lyotropic liquid crystalline phases, i.e., larger equilibrium structures with high degree of organization such as hexagonal (long rods of surfactants forming hexagonal pillars, H_1 or H_{II} if made of reverse micelles) [39,42], reverse anisotropic nematic N_2 [43], discontinuous cubic (spherical aggregates packed with a cubic symmetry) [39], bicontinuous cubic (spherical interconnected aggregates packed

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