



Transient stability of surfactant and solid stabilized water-in-oil emulsions



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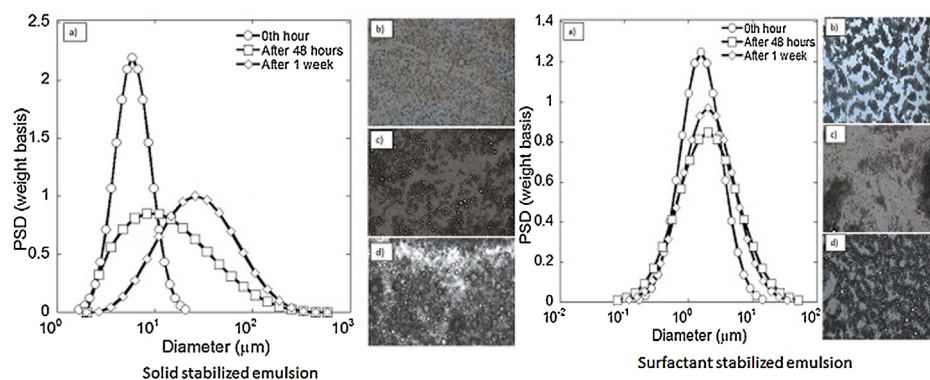
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HIGHLIGHTS

- Observation of dynamic emulsion behavior by acoustic and electroacoustic spectroscopy.
- Characterization of transient behavior of solid stabilized emulsions using acoustic spectroscopy.
- Quantification of de-stabilization mechanisms in solid stabilized emulsions.

GRAPHICAL ABSTRACT



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ABSTRACT

Water-in-oil emulsions with a dispersed phase fraction equal to 15 wt.% were prepared using either a non-ionic surfactant or solid particles under identical conditions of high energy mixing. Acoustic spectroscopy measurements were carried out on these emulsions to determine the initial droplet size distributions and their evolution over a period of one week. A transient stability test was conducted to compare the behavior of surfactant and solid particles at the oil-water interface. The objective of this paper was to elucidate the use of acoustic spectroscopy for characterizing emulsion stabilization/destabilization mechanisms in these emulsions. Experimental results indicate that destabilization mechanisms, such as coalescence, can be characterized using acoustic spectroscopy. Characteristics prevalent in the coalescing system, such as broad droplet size distributions and polydispersity, were captured by acoustic spectroscopy in terms of changes in the raw experimental attenuation. Complimentary bottle tests and optical microscopy experiments were conducted on these emulsions to validate the droplet size distributions and transient behavior of emulsions measured using acoustic spectroscopy.

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1. Introduction

Emulsions are metastable mixtures of two or more immiscible liquids. They can be classified as oil-in-water or water-in-oil based on the nature of the dispersed and continuous phase. An

emulsion is called stable if there is no phase separation against any destabilization phenomenon, such as coagulation and coalescence, phase inversion, creaming, sedimentation and Ostwald ripening, and if the emulsification process is possible [1]. Emulsifiers are additives that stabilize the oil–water interface. Surfactants are the usual choice for this purpose, and have been available in the market as an emulsifier for both water-in-oil and oil-in-water emulsions. Surfactants are either ionic, non-ionic, or zwitterionic in nature. Surfactants reduce interfacial tension, thereby promoting the creation of water–oil interface, and subsequently stabilize the interface to resist coalescence [2,3].

Emulsions stabilized using finely divided particles/nanoparticles are called solid stabilized emulsions (also known as Pickering emulsions) [2,3]. Solid stabilized emulsions have gained importance in the last two decades due to their applicability in many industries such as food, agriculture, pharmaceutical, and energy. These applications arise due to enhanced emulsion stability in some cases and the ability to produce droplets ranging from a few microns to several millimeters [4]. Solid particles function in a manner similar to surfactants. However, the preferential wetting of solid particles plays a major role in determining whether oil-in-water or water-in-oil emulsions are formed [5]. Solid particles such as pre-treated silica (available as Aerosil) have been widely used in the pharmaceutical and cosmetic industries as a stabilizer, and similar applications exist in the oil and gas industry [6]. Solid particles, such as hydrophobic silica, are known to be preferentially wetted by the oil phase and lead to the formation of water-in-oil emulsions; whereas, hydrophilic silica particles, which are preferentially wetted by the water phase, produce stable oil-in-water emulsions [3,7,8].

Solid stabilized emulsions can be more stable, depending on surface coverage, as compared to surfactant stabilized emulsions, due to the strong adhesion of solid particles to the oil–water interface. In solid stabilized emulsions, the most important factors to consider are the contact angle, coalescence kinetics, and particle interaction energy [7]. Stable emulsions are prepared when the particles are neither too hydrophilic nor hydrophobic and if there is complete surface coverage. Particles that form contact angle, $\theta > 90^\circ$ form a water-in-oil emulsion, whereas, $\theta < 90^\circ$ leads to an oil-in-water emulsion [5,7,8]. The particle shape, size and concentration greatly affect the emulsion stability and droplet size [9].

Acoustic and electroacoustic spectroscopy are some of the most reliable techniques for characterizing concentrated dispersions and emulsions in-situ [10]. These ultrasound based methods were chosen over other traditional particle sizing techniques, such as dynamic light scattering and electron microscopy, due to their ability to characterize polydisperse and concentrated systems without dilution [10,11]. For this study, we used an Acoustic/Electroacoustic spectrometer DT 1202 (produced by Dispersion Technology Inc.) for characterizing droplet size distributions of both surfactant and solid stabilized emulsions [10,11].

The motivation behind this work was to demonstrate how acoustic spectroscopy can be utilized to capture emulsion stabilization/destabilization phenomena in surfactant and solid stabilized emulsions, and characterize their transient stability over a period of time. For the purpose of this work, emulsions that exhibit two different kinds of stabilization/destabilization mechanisms were studied using acoustic spectroscopy: one that undergoes coalescence, and a second that exhibits high stability. A surfactant stabilized emulsion was used for investigating the transient behavior of a highly stable emulsion, since it is well known to produce stable and non-coalescing droplets when formed above the critical micelle concentration. Solid stabilized emulsions were chosen to evaluate coalescence using acoustic spectroscopy. Another important goal of this work was to investigate how acoustic spectroscopy captures characteristics prevalent in coalescing systems, such as

polydispersity and broad drop size distributions. For the purpose of this study, both coalescing and highly stable emulsions were prepared using similar mixing conditions and compositions.

In this paper, the droplet size distributions of water-in-oil emulsions stabilized using either hydrophobic fumed silica (aerosil R972) or a non-ionic surfactant, sorbitan monooleate (Span 80), were studied at different time intervals after emulsification. Bottle tests and optical microscopy measurements were also carried out to validate the results obtained from acoustic spectroscopy.

2. Materials and methods

2.1. Emulsion preparation

The water-in-oil emulsions prepared for this study contained decalin as the oil phase and deionized water as the aqueous/dispersed phase. Decalin (purchased from BDH chemicals) with 99% purity and a density of 0.786 g/cm³, and deionized water with a resistivity of 18.2 M Ω cm were used. Sorbitan monooleate (Span 80) and hydrophobic fumed silica (Aerosil R972) were the two different stabilizers used for this study.

For the surfactant stabilized emulsion, span 80, C₂₄H₄₄O₆ (molecular weight = 428.6 g/mol), supplied by Sigma Aldrich with a density of 0.99 g/cm³, was used as an emulsifier. The critical micelle concentration of span 80 in solvents such as decalin (dielectric constant = 2.2) was identified as 1.5–2 wt.% [12]. Hence, the oil phase was composed of 2 wt.% of span 80. Span 80 is used in industries as a surface-active emulsifier. span 80 is a non-ionic lipophilic emulsifier with a hydrophile-lipophile balance ratio (HLB) of 4.3. The low HLB ratio of span 80 is well known to produce w/o emulsion.

For Pickering emulsions, hydrophobic fumed silica (Aerosil R972) manufactured by Evonik Industries (Rheinfelden, Germany), with a tamped density of 1.06 g/cm³ was used as the stabilizing agent. Aerosil R972 is manufactured by flame pyrolysis of silica at 1000 °C and surface treated with dichlorodimethyl silane to impart hydrophobic functionality. The primary particle size of Aerosil R972 is 16 nm, and the material typically exists as a sintered aggregate with size varying in the size range 100–300 nm. To keep the emulsifier concentration constant for both kinds of emulsions, 2 wt.% aerosil R972 was dispersed in the oil phase.

Both types of emulsions were prepared using an Ultraturrax T25 homogenizer with a maximum operating power of 25,000 rpm. The oil phase of the emulsions was prepared by mixing 81.98 vol.% of Decalin with 2.12 vol.% of the stabilizer using a spatula, and homogenizing at 8000 rpm for 1 min to completely disperse the stabilizer in the oil phase. 15.9 vol.% of water was slowly added to the oil phase, and the emulsion was prepared using the homogenizer at 8000 rpm for 10 min.

2.2. Acoustic spectroscopy

For this paper, we used an acoustic/electroacoustic spectrometer DT-1202 (manufactured by Dispersion Technology, Inc.) for characterizing both kinds of emulsions. A detailed description of this device is available in the literature [13]. The spectrometer is used for measuring drop size distribution, zeta potential, and conductivity simultaneously. The acoustic technique utilizes acoustic sound waves for measuring particle and drop size distributions; whereas, the electroacoustic technique utilizes both electric and acoustic signals for measuring surface charge and zeta potential.

Acoustic particle sizing is regulated by two International Standards: ISO 20998 Parts 1 and 2 [10,11,13]. The two essential features of the acoustic particle sizing technique that make it absolute and not requiring calibration are pulse technique and variable gap method. The acoustic spectrometer works on the prin-

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