

Interactions between colliding oil drops coated with non-ionic surfactant determined using optical tweezers

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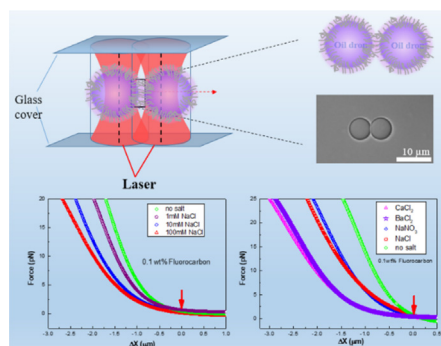
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HIGHLIGHTS

- This work provides novel observations of the charging behavior at O/W interface.
- The tetradecane drops coated non-ionic surfactant FS-30 are negatively charged.
- The effects of various salt cases on the interactions were studied systematically.

GRAPHICAL ABSTRACT



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ABSTRACT

Non-ionic surfactants are widely used in many industrial applications such as detergents, oil recovery, and mineral flotation techniques. Non-ionic surfactants readily adsorb onto the oil-water interface, although they cannot dissociate in solutions like ionic surfactants can. Nonetheless, the electrostatic double-layer (EDL) repulsive force between oil drops coated with the non-ionic surfactant can still provide stability against coalescence. In this work, the interaction forces between two tetradecane drops with diameter of 6.5 μm were measured in the presence of the non-ionic fluorocarbon-based surfactant FS-30 in various salt solutions using optical tweezers. The measured force curves were consistent with the presence of a surface charge, even though the surfactant was non-ionic in nature. The results clearly show that the EDL repulsive force is gradually screened with increasing salt concentration. It was also found that the EDL repulsive force was significantly screened in the divalent cation salt solutions (Ca^{2+} , Ba^{2+}) in comparison to in the monovalent cation salt solutions (Na^+). In addition, the absolute value of the measured zeta potential ($-\zeta$) of emulsified tetradecane drops gradually decreased with increasing FS-30 concentration. In all experimental processes, the emulsified tetradecane drops coated with FS-30 were stable against coalescence. These findings have significant implications for the stability of emulsions used in the food, cosmetic, and detergent industries.

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1. Introduction

Emulsions are widely used in various fields such as food (Gromer et al., 2010; Lissant et al., 1974), cosmetics (Lee et al., 2002), controlled-release substances and drugs (Gallarate et al.,

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1999; Laugel et al., 1998; Laugel et al., 2000; Muguet et al., 2001), advanced materials (Ge et al., 2014; Jiang et al., 2010; Pera-Titus et al., 2015), and biology (Shively, 2002). Stability of the emulsions plays a crucial role within these applications. Emulsions can be stabilised by using surfactants (Scamehorn, 1986), polymers (Tucker et al., 2012), and nanoparticles (Zhou et al., 2016), all of which are common and effective stabilisers. These stabilisers easily adsorb onto oil-water interfaces, resulting in an electrostatic double-layer (EDL) repulsive force or a steric barrier that prevents the coalescence of emulsion drops, which have been widely used in industrial production of emulsions. The stability of the emulsions including adsorbed surfactants, polymers, or nanoparticles relies on a well-developed understanding of interactions between single dispersed drops (Dagastine et al., 2006). It is thus vital to measure and analyse the interaction forces between emulsion drops in different stabiliser solutions.

Ionic surfactants readily dissociate in the aqueous phase, which leads to an EDL repulsive force that prevents the coalescence of emulsion drops. Non-ionic surfactants easily adsorb onto oil-water interfaces, although they do not dissociate in the aqueous phase like ionic surfactants; in the latter, the EDL repulsive force together with a steric barrier may synergistically stabilise the emulsion drops against coalescence (Mettu et al., 2017). Non-ionic surfactants are of interest as they are used in detergents, oil recovery, mineral flotation applications, and as a component of dispersants used in oil spill remediation (Fu et al., 1992). In many previous studies (Frostad et al., 2013; Giribabu and Ghosh, 2007; Karraker and Radke, 2002; Reichert and Walker, 2015), coalescence and other stability mechanisms in non-ionic solutions have been widely studied using traditional methods. Reichert et al. observed a long coalescence time for oil drops in the presence of the non-ionic surfactant Tween-80 (Reichert and Walker, 2015). Coalescence is characterised using a modified capillary tensiometer, allowing for accurate measurement of the coalescence time. Furthermore, the coalescence was shown to proceed more quickly in various 0.5 mM salt solutions than it did in a solution without any salt. This result clearly indicates that the EDL force can be screened in high salt solutions. However, the measurement of coalescence using a capillary tensiometer or other traditional methods is generally imprecise, because precise control of the drop position is not achieved.

Recently, atomic force microscopy (AFM) has made it possible to directly and quantitatively measure the interaction forces between two approaching surfaces in non-ionic solutions (Mettu et al., 2017; Rutland and Senden, 1993; Stubenrauch et al., 2004; Stubenrauch and Von Klitzing, 2003). The initial conditions for the approach of the drops are well controlled, allowing precise control of the drop position. Mettu et al. measured the interaction forces between two tetradecane drops in the presence of the non-ionic surfactant pentaethylene glycol monododecyl ether ($C_{12}E_5$) using AFM (Mettu et al., 2017). The tetradecane drops were negatively charged in the presence of $C_{12}E_5$, and the EDL force was gradually screened with increasing background electrolyte concentration. The experimental data was well fitted with the theoretical model.

However, these studies have mainly focused on force measurements between emulsion drops of 20–200 μm diameter (Bhatt et al., 2001; Butt et al., 2005; Chan et al., 2001; Chan et al., 2009, 2011; Dagastine et al., 2006; Mettu et al., 2017; Shi et al., 2017; Shi et al., 2016), whereas micron-sized emulsion drops (with diameter < 10 μm) have rarely been studied (Gillies et al., 2001, 2002; Nielsen-Nygaard et al., 2014). The relationship between Laplace pressure and deformation is significantly different for micron-sized drops than it is for 100 μm drops (Dagastine et al., 2006). The calculations of surface force and hydrodynamic interaction also require markedly different treatment for micron-sized drops than for larger drops (Chen et al., 2018b). Moreover, micron-size

drops are closer to the size of the drops in emulsions which have been widely used in many practical applications (Dong et al., 2013; Jensen et al., 2006; Ugelstad et al., 1992) such as toners, spacers for liquid crystal displays, and materials for biomedical and biochemical analyses. Therefore, there is an urgent need to systematically and quantitatively study the dynamic interaction forces between micron-sized oil drops.

Due to the limitations of AFM, the measurement of drops below 10 μm in diameter by AFM is relatively difficult. Optical tweezer technology offers a promising alternative. Optical tweezers are an optical operating platform that use the refraction and reflection of light to converge laser beams and capture the drops (Ashkin, 1970, 1978). With this technique, precise control of the drop position and quantitative measurement of the interaction forces between single oil drops below 10 μm in diameter can be readily achieved. In this work, we have pioneered the use of optical tweezers to study the interaction forces between two micron-sized (diameter 6.5 μm) tetradecane drops in the presence of the non-ionic fluorocarbon-based surfactant Capstone Fluorosurfactant FS-30 (FS-30; Dupont Co., Ltd.). The interaction forces between two micron-sized tetradecane drops, in various salt solutions and in the presence of FS-30, were measured precisely and analysed using the classical colloids and interface theory (Derjaguin, 1941; Derjaguin et al., 1954; Liang et al., 2007; Verwey et al., 1999). In addition, we measured the interface tension in the presence of different concentrations of FS-30 using pendant drop tensiometry, and the critical micelle concentration (CMC) on the interface of tetradecane/water was approximately 0.5 wt%. Further, the absolute value of the measured zeta potential of the emulsified tetradecane drops gradually decreased as the FS-30 concentration increased. Moreover, the measured force curves showed that the EDL repulsive force was significantly screened in the divalent cation salt solutions (Ca^{2+} , Ba^{2+}) compared to that within the monovalent cation salt solutions (Na^+). These findings have significant implications for the stability of emulsions applicable in the food, cosmetics, and detergent industries. This work provides a useful method for quantifying the interaction forces between emulsified micron-sized oil drops with non-ionic surfactant adsorbed at the oil/water interface in different salt solutions, and the results provide insights into the stabilisation mechanisms of oil-in-water emulsion drops.

2. Experimental

2.1. Materials and methods

Solutions of the fluorocarbon-based surfactant FS-30 (Dupont Co., Ltd, China) were prepared using Milli-Q water (resistance $\geq 18.2 \text{ M}\Omega \text{ cm}$) and used without purification. Sodium chloride (NaCl), sodium nitrate (NaNO_3), caesium chloride (BaCl_2), and calcium chloride (CaCl_2) of high purity were purchased from Aladdin (China) and used without purification. Tetradecane was also purchased from Aladdin and used without purification as the oil phase for all emulsion drops. The interfacial tension of tetradecane and different aqueous solutions was measured using a pendant drop tensiometer (OCAH200, Data Physics Instruments GmbH, Germany). Zeta potentials of tetradecane oil drops under different aqueous conditions were measured using a Zetasizer Nano ZSP (Malvern Instruments, UK). All experiments were carried out at 25 $^\circ\text{C}$.

2.2. Optical tweezers measurements

A NanoTracker™ 2 optical tweezers instrument (JPK Instruments, Berlin, Germany) was used to measure the interaction

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