



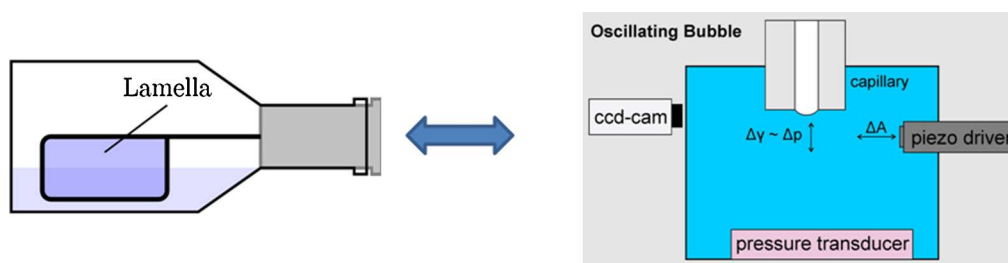
Surface rheology and its relation to foam stability in solutions of sodium decyl sulfate



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GRAPHICAL ABSTRACT



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ABSTRACT

The anionic surfactant sodium decyl sulfate (SDeS) at concentrations below its critical micellar concentration (cmc) was studied in a surface chemically purified state at the air–water interface in terms of its static and dynamic interfacial properties. Equilibrium surface tension was determined using a pendant drop tensiometer. An oscillating bubble capillary pressure tensiometer allowed accessing the surface dilatational properties of the surfactant's adsorption layers in a frequency range from 2 to 500 Hz. The estimation of single foam lamella stability was obtained from visual observation of films formed within a rectangular glass frame in a saturated atmosphere. Upon increasing surfactant concentration, the foam lamellae were found to rupture at prolonged lifetimes. The dramatic difference in foam stability goes along with a pronounced transition from a surface elastic to a surface visco-elastic state of the adsorption layers. These findings were indicative for a correlation between foam lamella stability and surface viscoelasticity also for this model surfactant. These results may be of importance to further shed light on the processes governing foam and foam lamella stability.

1. Introduction

Surfactants play a major role in the stabilization of foams and emulsions. Recently, they also found application as directional agents in the synthesis of nano-particles [1]. Sodium decyl sulfate (SDeS) as member of the commercially available homologous series of sodium-*n*-alkyl sulfates is a frequently studied model substance for anionic surfactants. Critical micellar concentrations (cmcs) and the corresponding heats of micellation have been determined as a function of temperature [2] as well as its mixed adsorption with perfluorooctanoate towards γ -alumina [3] and mixed

micellation with *N,N*-dimethyldodecylamine oxide [4].

Its adsorption dynamics have been studied at the water-*n*-hexane interface [5]. At low bulk concentrations it was found to exhibit ideal behavior [6] at both the oil–water and air–water interface [7]. Next to studying the properties of pure SDeS at different interfaces, also its interaction with other components has been of interest to previous studies. Penfold et al. studied the interaction of sodium-*n*-alkyl sulfate surfactants with oppositely charged polyelectrolytes at air–water interface [8]. Foam enhancement upon addition of an oppositely charged polymer to sodium-*n*-alkyl sulfates has been observed previously and correlated to measurements of surface

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viscoelasticity [9]. Mixtures of sodium alkyl sulfates (C_{10} , C_{12} , C_{14} and C_{16}) with non-ionic surfactants of the Triton series have been studied in terms of their dynamic surface tension properties by means of the maximum bubble pressure method [10]. The validity of a diffusion controlled adsorption regime was proven from the linear relation between dynamic surface tension γ and the reciprocal square root of adsorption time $1/\sqrt{t}$. Electron paramagnetic resonance (EPR) spectroscopy of aqueous mixtures containing SDeS and the non-ionic polymer poly(vinylpyrrolidone) were found to form more ordered and compact micelles compared to the respective sulfonate surfactant [11]. Also the interaction between carbon-soot nano-particles and the structurally related surfactant 1-decane sodium sulfonate was subject to a dynamic surface properties study [12]. Therein, dilatational characteristics up to a perturbation frequency of 0.2 Hz and transient heights of foam columns were determined.

Characterization of foams is a field of high complexity and a several characteristic numbers for the qualification of foaming and foam properties have been introduced. Karakashev et al. [13] therefore suggested a new parameter unifying characteristics during the foam formation and foam decay by definition of the so called foam production as ratio of foamability and the rate of foam decay relying on a concentration scale relative to the cmc. Foam stabilization due to the presence of the homologous series of alkyl sulfates was studied using the Ross-Miles method and foam lifetime at constant pressure [14]. Badwan et al. found the maximum foamability of the members of this series to occur at a defined temperature depending on the chain length [15]. Also the presence of long-chain alcohols was found to modify the drainage rate in foam films and the generated foam volume [16,17]. The breakdown of foams can be accelerated by the action of bulky anti-foaming agents such as tributyl phosphate or 2-ethyl-1-hexanol [18]. The two-dimensional micro-structure within plain foam films leading to film stabilization and stepwise film thinning can be determined by means of an interferometric setup [19]. Studies of this kind have been conducted earlier by Goddard and coworkers [20,21].

In this article, we measure the surface dilatational modulus in an extended frequency range from 2 to 500 Hz by means of a novel design of a capillary tensiometer and relate the complex modulus to the visually observed stability of individual foam lamellae. The aim is to establish a relation between foam stability and the surface dilatational characteristics.

2. Experiment

2.1. Materials

Sodium decyl sulfate (SDeS) was obtained from Acros organics (HPLC grade). Due to the component's susceptibility to sulfate ester hydrolysis, a purification procedure to exclude the parent compound, *n*-decanol, as described by Lunkenheimer et al. [22] was used to ensure the state of surface chemical purity. The procedure includes repeated adsorption steps towards a large surface area followed by a suction cleaning of the compressed interfacial area. Thereupon, the preferentially adsorbed *n*-decanol impurities gradually become less abundant. The process is conducted in a specially designed and fully automated apparatus. Adsorption times of 600 s between the 400 subsequent expansion and compression cycles were chosen. To ensure validity of the purification procedure, it was carried out at a concentration of $30.00 \text{ mmol L}^{-1}$, which is below the surfactant's critical micellar concentration (cmc). Depending on the method chosen (titration calorimetry or conductometry), this value differs between 24.5 and 36 mmol L^{-1} [2,23,24]. The aqueous solutions were prepared using deionized water with a resistivity of $18.2 \text{ M}\Omega \text{ cm}$. The molecular structure of SDeS is shown in Fig. 3.

The studied aqueous solutions of SDeS at concentrations of 0.2, 1.5, 15.0 and 30.0 mmol L^{-1} were obtained from diluting the stock solution with deionized water.

2.2. Surface tension

The equilibrium surface tension isotherm was obtained using a drop profile analysis tensiometer (PAT1M, Sinterface GmbH, Germany). Starting from the stock solution having a concentration of 30 mmol L^{-1} , lower concentrated samples were prepared by successive dilution with

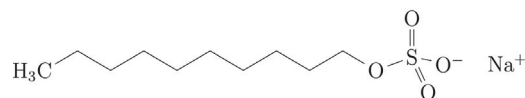


Fig. 1. Molecular structure of SDeS.

deionized water. The equilibrium values of surface tension of the respective solutions were obtained from averaging the instantaneous surface tension values over 100 s starting at a surface age of 200 s measured at a temperature of $22 \pm 0.5 \text{ }^\circ\text{C}$.

2.3. Foam lamella stability

Foam lamella stability was estimated from the life time of a macroscopic plain foam lamella ($3 \text{ cm} \times 1.5 \text{ cm}$) formed at a metal frame placed in an aqueous solution of the respective surfactant concentration [25]. The life time of the studied foam lamella is defined as the time from lamella formation until its rupture. A schematic view of the home-built device is given in Fig. 2. The metal frame is contained in a flask to ensure saturation of the gas phase. Visual observation was used to determine the time from lamella formation to the rupturing event. A conventional stop watch was used to measure the foam lamella life time.

At each concentration, a single foam lamella was formed within the metal frame by rotating the flask, thereby moving the frame through the sample liquid. After its formation, the lamella was brought into an upright position remaining in contact with the solution as shown in Fig. 2. The measurements were carried out at room temperature and repeated 25 times for each concentration.

2.4. Oscillating bubble

For the measurement of surface rheological characteristics, an oscillating bubble device was used [26]. At each studied concentration, three independent measurements were carried out, *i.e.*, after each measurement, the sample chamber was opened and refilled with the respective sample solution. In the following, the averaged values obtained from these three measurements will be shown. Within one measurement, the frequency scanning mode was applied, *i.e.*, amplitude and phase angle of the pressure response were measured at a certain frequency. Before moving on the next frequency, the bubble size was corrected to half sphere geometry by an implemented control loop. All measurements were carried out at room temperature at a fixed relative deformation amplitude of 5%.

3. Results

3.1. Surface tension isotherm

The experimentally obtained equilibrium surface tension isotherm of sodium decyl sulfate (SDeS) and a comparison to published literature data for the same surfactant in a purified state is shown in Fig. 3.

The experimentally obtained values of equilibrium surface tension γ against the logarithm of concentration $\log C$ curves can be described by the frequently used Frumkin adsorption isotherm accounting for intermolecular interactions between the individual adsorbed decyl sulfate ions. From the fitting parameters a minimum surface area of 22.2 \AA^2 is obtained, which compares well with the 21.5 \AA^2 from the published equilibrium isotherm measured at $21.9 \text{ }^\circ\text{C}$. The error due deviations in temperature between the experiment and published literature data is negligible.

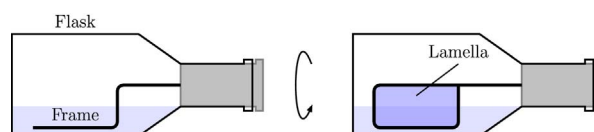


Fig. 2. Metal frame mounted in a flask for the determination of foam lamella stability.

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