

## Probing the surface of aqueous surfactant-perfume mixed solutions during perfume evaporation



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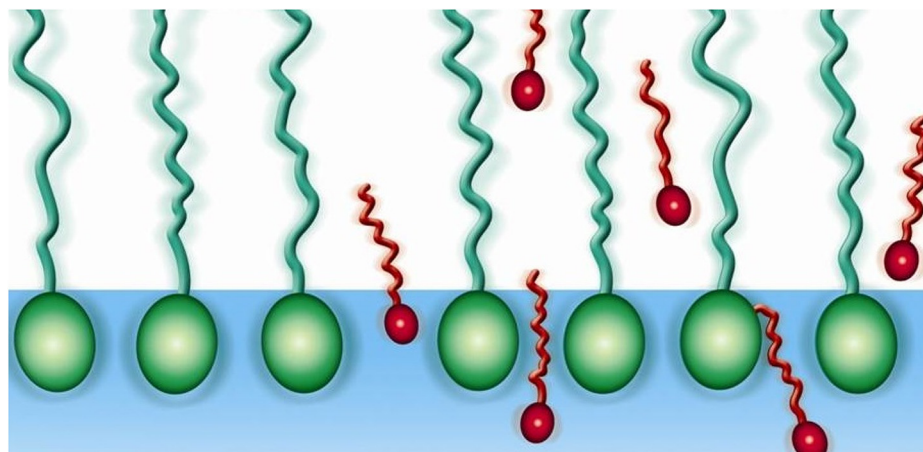
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### HIGHLIGHTS

- Perfume evaporation from mixed surfactant-perfume solution measured under forced air flow over stagnant headspace in limit of high flow rates.
- Amount of perfume adsorbed at interface decreases linearly with increasing time.
- Rate of decrease in adsorbed amount is dependent on perfume/surfactant solution composition.
- Diffusion to surface through a depleted surface region is the rate limiting factor.

### GRAPHICAL ABSTRACT



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### ABSTRACT

The rate of release or evaporation of perfume molecules from surfaces is a key factor in determining the impact of the perfume in a range of applications relating to home and personal care products. For mixtures of the anionic surfactant sodium dodecylsulfate and the model perfume linalool the rate of change of adsorption with time due to forced air flow over a fixed headspace was evaluated using neutron reflectivity over a period of up to ~250 min. The measurements were made in the limit of the high flow rates where the evaporation is independent of the air flow rate. The amount of perfume at the interface decreases with time, and this occurs at a faster rate as the initial amount of perfume in solution is decreased. The variation of the perfume adsorption with time for different

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## 1. Introduction

Most surfactant based home and personal care products contain perfumes as a key element of their performance [1–3], and this has stimulated extensive research into the different properties of a range of model perfume molecules. The solubilisation of different perfume molecules in a range of surfactant and mixed surfactant solutions has been studied [4–8], their location within aggregates identified [9–11], and their impact upon surfactant phase behaviour determined [12–15]. Optimised delivery is an important element and this has stimulated the development of novel encapsulation and delivery systems [16–19].

In optimising the performance of perfumes in many of the formulations, their efficient delivery to, and retention at surfaces are important factors to understand and optimise. A number of recent studies using neutron reflectivity, NR, have focussed on these aspects. Bradbury et al. [20,21] and Penfold et al. [22] have investigated the co-adsorption at the air-solution interface of some model perfume molecules, benzyl alcohol, phenylethanol, and linalool with different anionic surfactants, and notably sodium dodecylsulfate, SDS, and sodium dodecylbenzenesulfonate, LAS, and the cationic surfactant, cetyltrimethyl ammonium bromide, CTAB. More recently potential mechanisms or routes to enhance or manipulate perfume delivery to interfaces using surface multilayer structures [23] and polyelectrolyte-surfactant surface interactions [24] have been explored.

Perfume retention and evaporation from surfaces are key aspects of perfume performance in a range of home and personal care products [1–3]. The focus of this paper is on the evaporation of perfume molecules from the aqueous surface of a surfactant-perfume solution, and how it is related to the surface adsorption properties. Evaporation from surfaces has been extensively studied by indirect methods which are based on using gravimetric measurements or spectroscopic analysis of the headspace using chromatography or GC–Mass Spectroscopy [25–30]. Beverley et al. [25,26] have described a gravimetric method for the determination of evaporation rates for pure liquids and liquid mixtures, in which the evaporation rates are controlled by forced gas flow over a stagnant headspace. This approach was then applied to study the selective retardation of perfume oils from oil-in-water emulsions stabilised by surfactants or nanoparticles [27].

In this paper a new approach, in which the amount of perfume adsorption at the surface during evaporation is directly measured, is presented for the first time. We demonstrate how neutron reflectivity from the air-solution interface of a surfactant-perfume mixed solution can be used to follow the changes in adsorption during perfume evaporation from the surface. By selective deuterium labelling of the perfume molecules the variation in neutron reflectivity with time relates directly to the time dependence of the amount of perfume at the interface. This is in turn related to the mechanism for evaporation from the surface of the perfume and its delivery to the surface from the bulk solution, which has been determined for the model perfume, linalool, LL, in the presence of the anionic surfactant SDS, at different SDS/linalool compositions. Although the methods based on gravimetric measurements and headspace analysis has provided important insights into perfume and other volatile component evaporation, the approach adopted

here provides the opportunity to probe directly the role of the surface and delivery to the surface region on the evaporation process.

## 2. Experimental details

### 2.1. Neutron reflectivity

The neutron reflectivity, NR, measurements were made on the INTER reflectometer [31] at the ISIS neutron source. The reflectivity,  $R(Q)$ , was measured as a function of the wave vector transfer,  $Q$ , perpendicular to the surface (where  $Q = 4\pi \sin \theta / \lambda$ ,  $\theta$  is the grazing angle of incidence and  $\lambda$  the neutron wavelength). An accessible  $Q$  range  $\sim 0.03$ – $0.35 \text{ \AA}^{-1}$  was covered using a  $\theta$  of  $2.3^\circ$  and a  $\lambda$  range of  $1$ – $15 \text{ \AA}$ . The measurements were made at the air-water interface using a stainless steel trough to avoid excessive perfume adsorption on the trough surfaces, as described previously [20–22]. A fixed headspace volume with a series of baffles was attached to the trough, and the air flow through the headspace was at a fixed rate of  $40 \text{ L/min}$ . The trough volume was  $\sim 25 \text{ ml}$  and the sample temperature was  $25 \pm 1^\circ \text{C}$ . The samples were aligned and the data corrected and normalised to the direct beam and the reflectivity from a deuterium oxide,  $\text{D}_2\text{O}$ , surface using established procedures. Each individual measurement was made for a fixed counting period, in the range  $6$ – $15 \text{ min}$ , with a continuous air flow through the headspace above the trough.

The NR measurements were made for a combination of h-SDS and d-LL in null reflecting water, nrw, (water with a neutron refractive index identical to air,  $92 \text{ mol\% H}_2\text{O}/8 \text{ mol\% D}_2\text{O}$ ). Under these conditions the reflectivity is dominated by the presence of the deuterium labelled component at the interface, and the reflectivity is then directly related to the adsorbed amount of the deuterium labelled component [32], as used extensively in surfactant and mixed surfactant adsorption studies

### 2.2. Experimental arrangement

The stainless steel trough, with sample volume  $\sim 25 \text{ ml}$ , is mounted in a sealed container in which the usual lid is replaced by a fixed headspace containing a series of baffles, as illustrated in Fig. 1.

The detailed design and layout of the headspace and associated baffles is illustrated in Fig. 2. The distance between the liquid surface in the trough and the first baffle of the headspace box is  $35 \text{ mm}$ .

The design and layout of the baffles allows the headspace volume to be continuously replaced by a pump at a rate of  $40 \text{ L/min}$  without any disturbance of the free liquid sample surface below it. Within the timescale of the NR measurements, up to  $\sim 300 \text{ min}$ , the meniscus and surface of  $\text{D}_2\text{O}$  or a surfactant solution are unaffected by the air flow. This is indicated, in the absence of more volatile components, by an invariance in the NR data for a  $\text{D}_2\text{O}$  surface and an adsorbed layer of deuterium labelled surfactant in nrw with time. At longer pumping times than  $300 \text{ min}$  the forced air flow at  $40 \text{ L/min}$  was eventually sufficient to start to deplete the liquid surface level, resulting in a progressive misalignment of the incident neutron beam and an apparent reduction in the reflected signal. Hence data collection times were limited to a maximum  $\leq 300 \text{ min}$  to ensure that this additional complication was avoided.

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