Adsorption Equilibrium and Kinetics on N-Lauroyl-N-Methylglucamide at Air/Water Interface

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Abstract—The adsorption equilibrium and kinetics of N-lauroyl-N-methylglucamide (MEGA-12) aqueous solution were studied. The critical micelle concentration, the maximum surface excess, and the minimum area per molecule of MEGA-12 were obtained as $2.48 \times 10^{-4}$ mol/l, $4.883 \times 10^{-6}$ mol/m$^2$, and 0.34 nm$^2$, respectively. The adsorption kinetics of MEGA-12 was studied by the maximum bubble pressure method. The result shows that in the initial stage or at small MEGA-12 concentrations, the adsorption process is diffusion-controlled; however, it changes to become adsorption-controlled at the end of the process. The effects of temperature, inorganic salts, alcohols, and ionic liquid on the adsorption kinetics were also discussed.

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INTRODUCTION

From both scientific and industrial perspectives, dynamic surface tension (DST) is an important property of surfactant solutions. Therefore, a lot of work has been done in this field [1–11]. Among the methods of measuring the adsorption dynamic surface tension, the maximum bubble pressure method (MBPM) is used more often [4–11].

N-acyl-N-alkylglucamides (NAGAs) are an interesting class of green nonionic surfactants, which are synthesized from renewable raw materials such as reducing sugars and fatty acid esters, and have gained increasing attention due to their advantages with regard to performance, consumer health, and environmental compatibility. NAGAs/acid/chlorocarbon/water microemulsion systems were studied and applied to surfactant-enhanced aquifer remediation (SEAR), many chlorocarbons in the microemulsion systems were observed to generate classical Winsor I  III  II phase behavior [12, 13].

N-acyl-N-alkylglucamides were also analyzed by reversed-phase liquid chromatography (LC) coupled to mass spectrometry ionization (ESMS) and a simple and sensitive analytical methodology was developed for the determination of NAGAs in municipal sewage treatment plant that were influent and effluent based on solid-phase enrichment, LC separation, and negative ion ESMS quantification [14]. Ko and his coworkers studied the synergistic adsorption effect between n-acyl (octyl, nonyl, and decyl)-N-glu- camides (MEGA-$n$, $n = 8, 9, 10$) and a top-heavy-type anionic surfactant, SDS, and a zwitterionic surfactant, CHAPS (a derivative of cholic acid), [15–19] by means of equilibrium surface tension measurements. It was found that a 10–20% addition of MEGA-$n$ to CHAPS led to the most effective synergism in lowering the critical micelle concentration (CMC) and reducing the surface tension.

In this paper, the adsorption equilibrium and kinetics of N-lauroyl-N-methylglucamide (MEGA-12) aqueous solutions at the air/water interface were studied and the effects of temperature, inorganic salts, alcohols, and ionic liquid on the adsorption kinetics were also discussed.

EXPERIMENTAL

Materials and Apparatus

We synthesized N-lauroyl-N-methylglucamide (CH$_3$CON(CH$_3$)$_2$CH$_2$(CHOH)$_2$CH$_2$OH) ourselves and recrystallized it twice with an alcohol/acetone mixture. Surface tension measurements of aqueous solutions suggested a high purity, particularly, the absence of any minimum and/or “should” around the CMC (Fig. 3a). Water was doubly distilled. The final distillation was performed over alkaline KMnO$_4$ in order to remove surface active impurities. The other materials used in this study are all of A.R. grade.

Fig. 1. The schematic diagram of maximum bubble pressure tensiometer: 1 – N$_2$ gas bomb; 2 – bumper; 3 – gas flowmeter; 4 – surfactant solution container and measuring capillary; 5 – pressure sensor; 6 – computer; 7 – printer.

1 The text was submitted by the authors in English.
An FA1104 electron balance, a 501 super thermostat, a Krüss K12 tensiometer, and a maximum bubble pressure tensiometer were used.

**Methods**

**Equilibrium surface tension.** The MEGA-12 solution was diluted with water to certain concentrations and stirred for 1 min then left motionless for 5 min. The equilibrium surface tension, $\gamma_e$, was measured as a function of surfactant concentration, $C$, with a Krüss K12 tensiometer at room temperature ($22 \pm 0.1^\circ C$).

**Dynamic surface tension.** The maximum bubble pressure method was used to determine the adsorption dynamic surface tension. In this study, we equipped the maximum bubble pressure tensiometer [20, 21] ourselves as shown in Fig. 1.

Samples were diluted to desired concentrations and thermostated at a given temperature for at least 12 h before measuring. When $N_2$ was injected into the aqueous solution of surfactant through a narrow capillary (radius $r = 0.318$ mm) immersed into the solution at constant depth, the bubbles formed, and the rate of the bubble formation was controlled by the flow rate of the gas. During the formation of bubbles, the change in the pressure between in and out of the bubbles was converted into digital signals with a pressure sensor and a digital conversion card, and the digital signals were gathered and put out in voltage by a computer. The dead time of the bubble $t_{\text{dead}} = 0.05t_{\text{life}}$, and the relationship between the metered pressure $\Delta P$ and the voltage $V$ is

$$\Delta P = 0.2451V - 0.1161.$$  

The DST, $\gamma_t$, can be obtained from following relationship:

$$\gamma_t = 0.03433V_{\text{max}} - 0.03019,$$  

where $V_{\text{max}}$ is the maximum voltage recorded by computer. The DST $\gamma_t$ was measured in the time range 0.1–90 s.

**RESULTS AND DISCUSSION**

*Synthesis and characteristics of N-lauroyl-N-methylglucamide*

N-lauroyl-N-methylglucamide was synthesized from N-methylglucamine and lauric acid methyl ester in a reactor with circumfluence and mix-round equipment in the presence of basic catalyst—sodium methoxide and methanol as a solvent. The reaction was carried out for 6 hours at constant temperature under continuous stirring. Then the methanol was distilled. The product was recrystallized twice with an alcohol/acetone mixture.

The IR spectrum of MEGA-12 is shown in Fig. 2. The absorption peak at 1079 cm$^{-1}$ is related to the stretch vibration of the C–N bond, while at 1620 cm$^{-1}$, it is related to the stretch vibration of the

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**Fig. 2.** IR spectrum of $N$-lauroyl-$N$-methylglucamide.

**Fig. 3.** Plots of equilibrium surface tension $\gamma_e$ (a) and the MEGA-12 surface excess $\Gamma$ (b) at air/water interface vs. the concentration of surfactant solution.