

Dilational Properties of Sodium 2,5-Dialkyl Benzene Sulfonates at Air–Water and Decane–Water Interfaces

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ABSTRACT: The dilational properties of adsorbed film of sodium 2,5-dialkyl benzene sulfonates at the air–water and decane–water interfaces have been investigated by a Langmuir trough method. The influences of oscillating frequency and bulk concentration on dilational modulus and phase angle were expounded. The experimental results showed the hydrophobic chains located at different positions have different effects on dilational properties: for the air–water interface, the short alkyl chain ortho to sulfonate group produces a small increase in dilational modulus, while an increase in long para-alkyl plays an important role in the surface relaxation process, which leads to a higher surface dilational modulus. In the case of the decane–water interface, the interfacial dilational moduli are obviously lower than those of the air–water interface due to the insertion of decane molecules from oil phase. Moreover, the strong interactions between para-alkyl chains were weakened by decane molecules; therefore, the interfacial dilational moduli of sodium 2-ethyl-5-(1-butyl)octylbenzene sulfonate (4-7-2) and sodium 2-methyl-5-(1-hexyl)octylbenzene sulfonate (6-7-1) are approximately equal. The possible schematic diagrams of adsorbed molecules with different structures at the water–air and water–decane interfaces are proposed.

1. INTRODUCTION

Surfactants are among the most versatile products of the chemical industry, appearing in such diverse products as flotation, food production and processing, and the detergency of detergents and cosmetics. Sodium alkyl benzene sulfonates are the most commonly used synthetic anionic surfactant class, which have attracted multitudinous attention because of their well-known physicochemical characteristics and numerous industrial applications.¹ However, most of the studies on sodium alkyl benzene sulfonates have aimed to understand their adsorption at interfaces and low interfacial tension (IFT) activity,^{2–5} and far less attention has been paid to the surface and interfacial properties of these surfactants.

The rheological behavior at interfaces is an extremely complex phenomenon that has recently drawn the attention of many researchers.^{6–23} These dynamic properties are of fundamental interest in understanding the formation and stability of emulsions and foams.^{24–27} Moreover, the measurement of dynamic dilational behavior of surface-active molecules adsorbed at the interface had been proved to be a powerful technique to probe interfacial structure of the film formed.^{13,28}

The dilational rheological behaviors have been studied for more than half a century. However, the understanding of the relationships between surfactant structures and their interfacial properties is still a challenge, mainly due to lack of suitable pure surfactants with special structures as model compounds. In the present work, we have studied the dilational properties of three structure–designed branched-alkyl benzene sulfonates at the air–water and decane–water interface by means of a longitudinal method. The study was undertaken to provide insight into the effect of hydrophobic structure of a surfactant on interfacial dilational properties.

2. THEORETICAL BACKGROUND

The surface dilational modulus in compression and expansion is defined by the expression originally proposed by Gibbs for the surface elasticity of a liquid film as the increase in the surface tension for a small increase in area of a surface element:

$$\varepsilon = \frac{d\gamma}{d \ln A} \quad (1)$$

In this equation, γ is the interfacial tension, and A is the area of the surface element. The dilational modulus can also be defined as a complex function which can be written as:^{29,30}

$$\varepsilon = \varepsilon_d + i\omega\eta_d \quad (2)$$

where ε_d and $\varepsilon_\eta = \omega\eta_d$ correspond, respectively, to the real and the imaginary parts of the elasticity modulus. For a soluble monolayer, the elastic part ε_d accounts for the recoverable energy stored in the interface, and the viscous contribution, ε_η , represents the loss of energy in the relaxation process.

The phase angle θ was derived from the change in dynamic interfacial tension resulting from a small change in interfacial area (A), which means that the elastic and viscous contributions are given by:

$$\varepsilon_d = \varepsilon \cos \theta \quad (3)$$

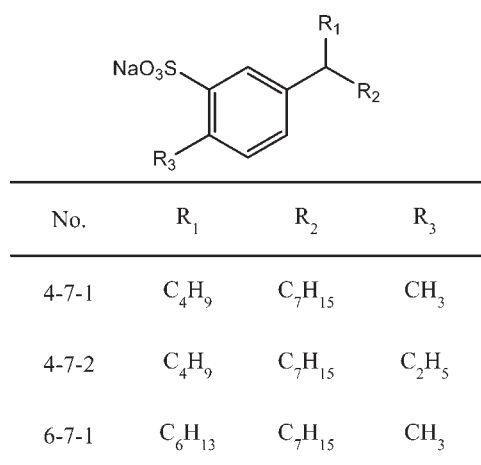
$$\varepsilon_\eta = \varepsilon \sin \theta \quad (4)$$

Received: December 22, 2010

Accepted: February 16, 2011

Published: March 07, 2011

Scheme 1. Sodium 2,5-Dialkyl Benzene Sulfonates with Different Structures



3. EXPERIMENTAL SECTION

3.1. Materials. A series of sodium 2,5-dialkyl benzene sulfonates were synthesized in our laboratory, as shown in Scheme 1.³¹ The purity of the compounds was checked by two-phase titration methods and ¹H NMR spectroscopy. *n*-Decane, analytical reagent grade, was obtained from Xingjin Chemical Reagent Ltd., Tianjin, China and used as the oil phase, which was further purified. The surface tension measured at 30 °C by a platinum plate method was $22.69 \cdot 10^{-3} \text{ N} \cdot \text{m}^{-1}$. Water used in the experiments was distilled twice from potassium permanganate solution.

3.2. Methods. The interfacial dilational viscoelasticities at the air–water and oil–water interfaces were recorded using JMP2000A (Powereach Ltd., Shanghai, China), combined with a Langmuir trough employing a pair of poly(tetrafluoroethylene) (PTFE) barriers for compression and expansion of the interface, as described previously.^{32,33} The dynamic interfacial tension was measured by the Wilhelmy plate method, using a Pt or PTFE plate, for air–water interface and oil–water interfaces, respectively, suspended in the middle of the trough area from a sensitive force transducer.

For the air–water interface, the water phase (about $90 \cdot 10^{-3} \text{ L}$) is poured into the trough, and the Pt plate is tangent to the top of the water surface. In the case of the decane–water interface, the water phase ($90 \cdot 10^{-3} \text{ L}$) and oil phase ($50 \cdot 10^{-3} \text{ L}$) were poured into the trough successively and carefully, and the PTFE plate should be completely submerged under the surface of the oil.

In the longitudinal wave method, the interfacial area can be changed in sine oscillating ((0.005 to 0.1) Hz) mode by the horizontal slide of the barrier. The dilational viscoelasticity measurements began after 6 h of pre-equilibrium of the air–water and oil–water interfaces by oscillating the interfacial area to a maximum change of 10 % of the original area ($\Delta A/A$, 10 %). In all cases, the standard deviation did not exceed 3 %. All experiments were performed at the temperature 30 °C.

4. RESULTS AND DISCUSSION

4.1. Frequency Dependence of Dilational Properties at Air–Water Interface. The variation of dilational properties with

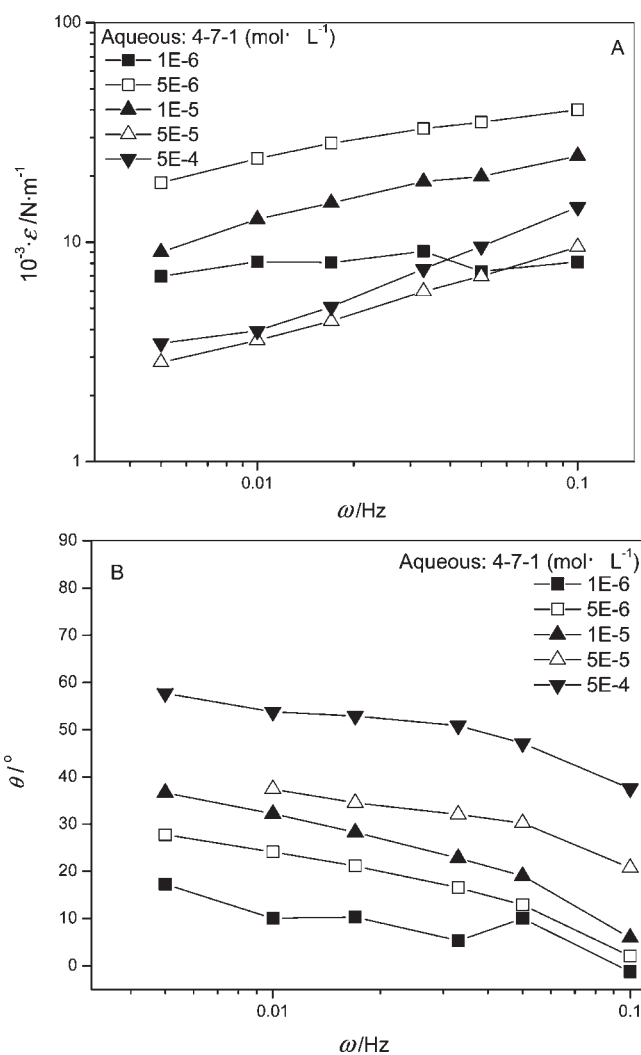


Figure 1. Frequency dependence of dilational rheological properties for 4-7-1 at the air–water interface. (A) Dilational modulus; (B) phase angle.

oscillating frequency could reflect the properties of interfacial adsorption film. To investigate the frequency dependence of the surface dilational properties, measurements in a range from (0.005 to 0.1) Hz have been carried out. For the sake of brevity, only the 4-7-1 surfactant will be discussed, and similar behaviors are found for the other surfactants investigated in this work. The dilational properties of the 4-7-1 solutions at the air–water interface are plotted as a function of the dilational frequency in Figure 1.

As seen from Figure 1, the dilational modulus is independent of frequency at low bulk concentrations, while there is a little change in the measured phase angle. These indicate that the adsorbed layer is predominantly elastic in nature. However, the dependence of frequency enhances at higher bulk concentration, and the phase angle decreases monotonically, which shows that the dilational properties of the adsorbed layer may be dominated by the exchange of the molecules between the bulk and the interface over the frequency range examined.

The modulus is a measure of the resistance against the creation of surface tension gradients. As the frequency grows higher, surfactant molecules have less time to respond to the surface

Table 1. Tendency of the Slope of $\log \varepsilon$ versus $\log \omega$ with Increasing Bulk Concentration for 4-7-1

$C/\text{mol}\cdot\text{L}^{-1}$	$\log \varepsilon - \log \omega$ slope
$1 \cdot 10^{-6}$	0.03
$5 \cdot 10^{-6}$	0.25
$1 \cdot 10^{-5}$	0.32
$5 \cdot 10^{-5}$	0.41
$5 \cdot 10^{-4}$	0.50

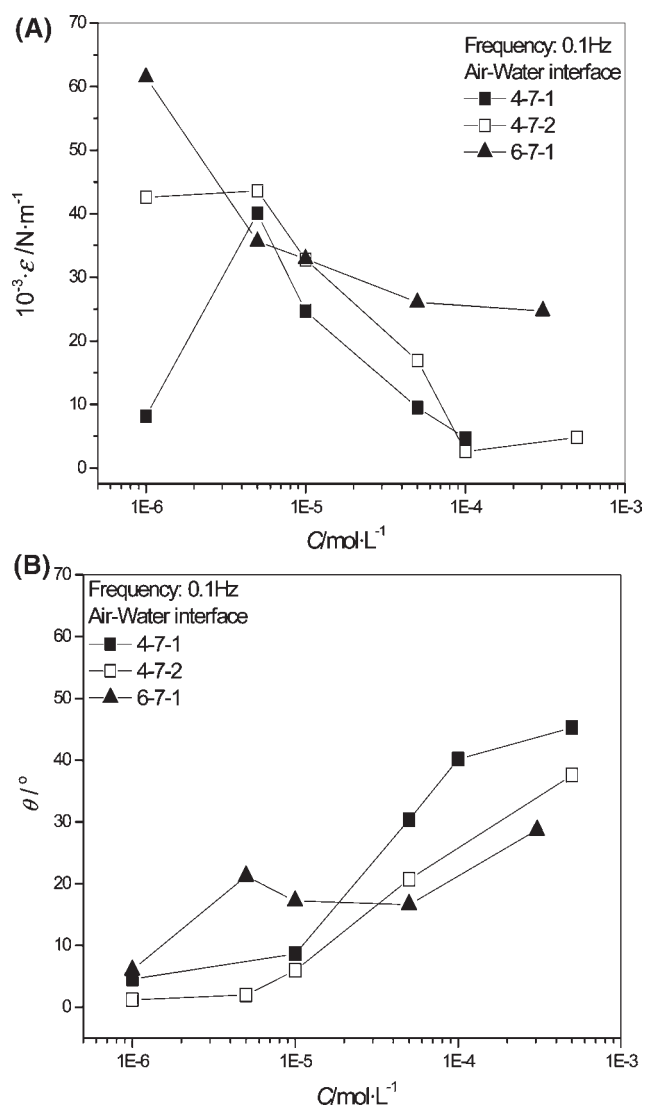
tension gradient resulting from the interface deformation. Thus, the dilational modulus increases with increasing oscillating frequency. On the other hand, the phase angle depicts the ratio of viscosity to elasticity. The exchange between interface and the bulk is responsible mainly for the viscosity of the soluble film, while the interaction in-interface is responsible for the elasticity of the film. The lower the phase angle is, the less the contribution of the dilational viscosity to the dilational modulus, due to the shorter time for the relaxation process of diffusion-exchange with increasing oscillating frequency.

If the characteristic frequency of the relaxation process at the interfacial layer exceeds the highest oscillating frequency used in this experiment (0.1 Hz), the curves of $\log \varepsilon - \log \omega$ are almost quasi-linear. The slope of curve for the diffusion-controlled relaxation process (the L-T model) was lower than 0.5.³⁴ When the interface had a special structure, the limiting slope of the $\log \varepsilon - \log \omega$ curve was 1.³⁵ Table 1 shows the tendency of the slope of $\log \varepsilon$ versus $\log \omega$ with increasing concentration at the air-water interface for 4-7-1. In the concentration range of the experiment, the slope increases with increasing 4-7-1 bulk concentration, which indicates strongly a decrease of elastic contribution to the dilational modulus.

4.2. Concentration Dependence of Dilational Properties at Air-Water Interface. The concentration dependence of dilational data for sodium 2,5-dialkyl benzene sulfonates with different structures at the air-water interface is presented in Figure 2. From Figure 2, we can see that the surface dilational modulus ε runs through a maximum with the increasing concentration except for 6-7-1 solutions due to the narrow experimental concentration range.

In general, an increase of surfactant concentration has two different effects on dilational data. On one hand, the increase of the surface concentration would lead to a higher interfacial tension gradient corresponding to the same interfacial deform, which results in an increase in dilational modulus. On the other hand, the molecular exchange between bulk and surface is increased with increasing surfactant concentration. The diffusion of surfactant molecules from the bulk to the interface can decrease the interfacial tension gradient, which creates a decrease in dilational modulus. Thus, at low surfactant concentration it is the increasing surface concentration, whereas at high surfactant concentration it is the molecular exchange that may play a dominant role in determining the dilational modulus. This "crossover" is mirrored in a maximum of the $\varepsilon_{(c)}$ curve. It could be speculated that there may be a maximum in concentration lower than the experimental range for 6-7-1 solutions.

As is known, by increasing hydrophobic chain length, the dilational modulus increases because of the increase of surface concentration and enhancement of hydrophobic interaction. However, it is worth noticing that the hydrophobic chains located in the different positions of the benzene have a different

**Figure 2.** Influence of bulk concentration on dilational properties of sodium 2,5-dialkyl benzene sulfonates at air-water interface. Frequency: 0.1 Hz. (A) dilational modulus; (B) phase angle.

effect on dilational properties. As shown in Figure 2A, for 4-7-2, the dilational modulus appears to cause no significant increase with increasing in length of ortho-alkyl; while for 6-7-1, the dilational modulus increases obviously with increasing in the length of the branched alkyl chain which is oriented para of the benzene. The obtained results suggest that the para-alkyl has significant effects on surface dilational rheology than those of ortho-alkyl for sodium 2,5-dialkyl benzene sulfonates.

As the concentration of the surfactant in the bulk increases, the transport of molecules from the bulk to the surface circumvents the opposing elastic force, leading to an increase in the measured phase angle, as shown in Figure 2B, which consists with a general regulation of concentration dependence on the dilational phase degree.

4.3. Concentration Dependence of Dilational Properties at the Decane-Water Interface. We have also investigated the interfacial dilational properties of employed series of sodium 2,5-dialkyl benzene sulfonates at the decane-water interface, and the results are shown in Figure 3. During our experimental

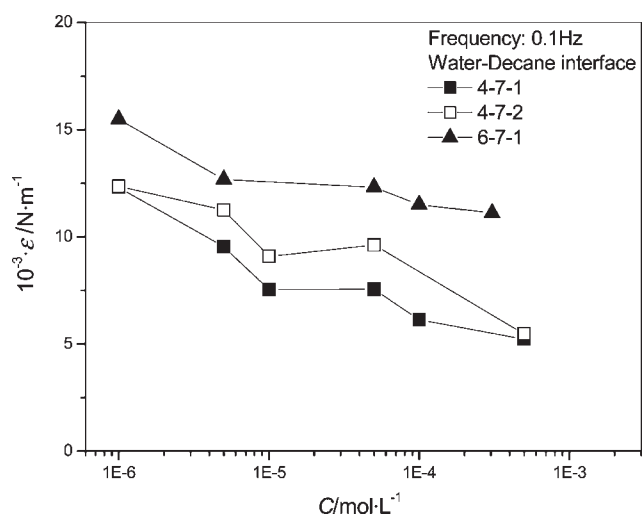


Figure 3. Influence of bulk concentration on dilational modulus of sodium 2,5-dialkyl benzene sulfonates at the water–decane interface. Frequency: 0.1 Hz.

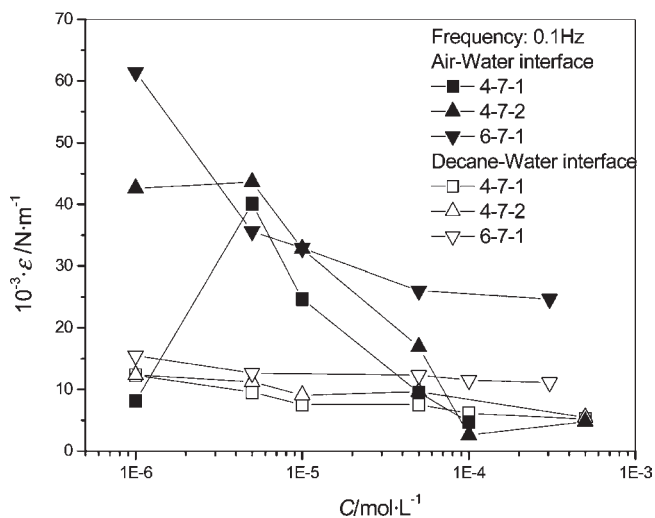


Figure 4. Concentration dependency of dilational modulus for sodium 2,5-dialkyl benzene sulfonates with different structures at air–water and decane–water interfaces, respectively. Frequency: 0.1 Hz.

concentration range, dilational modulus of three surfactants at the water–decane interface decreases monotonously with increasing concentration. Moreover, the dilational moduli at the water–decane interfaces are remarkably lower than those at the water–air interfaces for all surfactants.

To clarify the differences between dilational properties at air–water and decane–water interfaces, the concentration dependence of dilational modulus at both interfaces has been replotted at Figure 4.

From Figure 4, we can find that the dilational modulus is more than two times smaller at the decane–water interface as compared to the air–water interface. Moreover, it is very interesting that the values of dilational moduli of 6-7-1 are obviously higher than those of 4-7-2 at the air–water interface, while they are similar at the water–decane interface.

Moreover, the influence of bulk concentration on the slope of $\log \epsilon$ versus $\log \omega$ at air–water and decane–water interfaces is

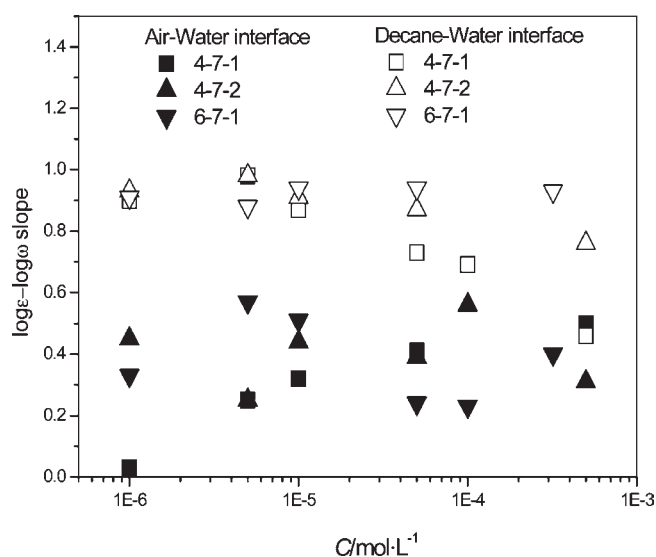


Figure 5. Influence of bulk concentration on the slope of $\log \epsilon$ versus $\log \omega$ at air–water and decane–water interfaces.

shown in Figure 5. The slope of $\log \epsilon - \log \omega$ curve reflects the viscoelastic nature of adsorption film. The lower the slope is, the more elastic the film appears. We can see clearly from Figure 5 that the slopes of decane–water interfaces are apparently higher than those of air–water interfaces for all three surfactants, which show strongly that the elasticity of adsorption film decreases due to the insertion of decane molecules into adsorbed surfactant molecules.

According to the above phenomena, we speculate that, at the air–water interface, an increase in the length of ortho-alkyl only increases hydrophobic interaction for 4-7-2 solutions, while in the case of 6-7-1 solutions, there exist strong interactions between the adjoining alkyls of 6-7-1 molecules except for van der Waals interaction. One can assume that the oil phase will be a better solvent for the hydrophobic chains of the adsorbed surfactant than air. Therefore, at the decane–water interface, for 6-7-1 solutions, the interposition of the oil molecules will weaken sharply the interactions between the branched alkyl chains which are oriented para of the benzene at interfaces.^{28,32}

The possible schematic of adsorbed 4-7-2 and 6-7-1 molecules at air–water and decane–water interfaces is proposed in Figure 6. As known, there exist some spaces among adjoining molecules because of the electrical repulsion between adjoined anionic molecules. As shown in Figure 6A, for 4-7-2 solution, the short ortho straight alkyl chain has little effect on the arrangement of molecules at the interface. In contrast, for 6-7-1 solution, the branched alkyl chain is much flexible, which are easily cross-linking and entangling, resulting in increment of interaction between 6-7-1 molecules. Therefore, the dilational modulus of 6-7-1 solutions is obviously higher than 4-7-2 solutions at the air–water interface.

In the case of the decane–water interface, as shown in Figure 6B, on one hand, the hydrophobic chains incline to be oriented toward the oil interface; on the other hand, the intervention of oil molecules will weaken the strong interactions between the long alkyl chains of interfacial 6-7-1 molecules and enhance diffusion-exchange between the interface and the bulk solution. As a result, the interfacial dilational moduli of 6-7-1 solutions decrease sharply.

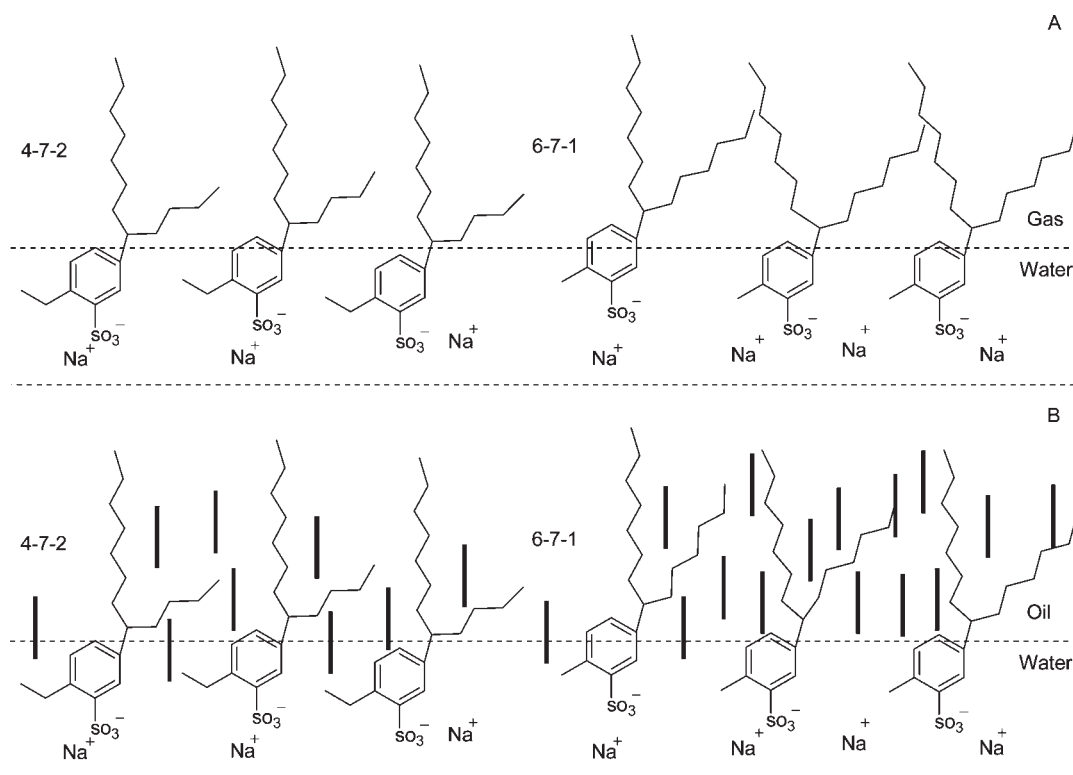


Figure 6. Schematic of adsorbed 4-7-2 and 6-7-1 molecules at the air–water (A) and decane–water (B) interface.

5. CONCLUSIONS

The dilational properties of sodium 2,5-dialkyl benzene sulfonates at the air–water and decane–water interfaces were investigated in the present work. The experimental results show that the dilational modulus increases with increasing the length of the hydrophobic group at air–water interface, and the same trend was observed at the decane–water interface. By comparison, the change in para-alkyl has significant effects on the nature of interfacial film than those of ortho-alkyl for sodium 2,5-dialkyl benzene sulfonates. Moreover, the dilational modulus at the decane–water interface is remarkably lower than those at the water–decane interface, due to intervening of oil molecules, which indicates that the nature of adsorption film is determined mainly by the structure of hydrophobic part. The possible schematic diagrams of adsorbed molecules with different structure at the water–air and water–decane interfaces are proposed.

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Funding Sources

The authors thank financial support from the National Science & Technology Major Project (2008ZX05011) and National High Technology Research and Development Program (2008AA092801) of China.

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