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The Physico-Chemical Properties of Sugar-Oxime-Ether Surfactants

Sugar oxime ether surfactants are a new class of surfactants produced by the reaction of a sugar with a hydrophobic alkoxyamine, producing an oxime ether linkage. We examined nine examples of this class of surfactants. The sugars used were maltose, sucrose, and glyceraldehyde; the alkoxyamines were decyloxyamine, dodecyloxyamine, and adamantyloxyamine. For the resulting surfactants we determined their thermal stability, melting point, water solubility, effect on surface tension, and critical micelle concentration. We found that all the compounds tested were thermally stable and decreased substantially the surface tension of water.

Key words: Adsorption, renewable, sugar, saccharide, alkoxyamine

Physikalisch-chemische Eigenschaften von Zuckeroximethertenside. Zuckeroximethertenside sind eine neue Tensidklasse, die durch Reaktion eines Zuckers mit einem hydrophoben Alkoxyamin unter Bildung einer Oximetherbindung hergestellt werden. Wir haben neun Beispiele aus dieser Tensidklasse untersucht. Die verwendeten Zucker waren Maltose, Saccharose und Glycerinaldehyd; die Alkoxyamine waren Decyloxyamin, Dodecyloxyamin und Adamantyloxyamin. Für die resultierenden Tenside haben wir die thermische Stabilität, den Schmelzpunkt, die Wasserlöslichkeit, den Einfluss auf die Oberflächenspannung und die kritische Mizellenbildungskonzentration bestimmt. Wir fanden, dass alle getesteten Verbindungen thermisch stabil waren und die Oberflächenspannung von Wasser wesentlich verringerten.

Stichwörter: Adsorption, Erneuerbar, Zucker, Saccharid, Alkoxyamin

1 Introduction

Modern society depends on ubiquitous use of chemicals in everyday life. Thus, the source and environmental fate of these chemicals (such as surfactants used in detergents, aircraft deicing fluids, and firefighting foams) are of great interest. Indeed, chemicals derived from renewable biomass have received recently a great deal of attention [1–6]. We became interested in sugar surfactants, a class of biocompatible nonionic surfactants which can be prepared by linking the *renewable* hydrophilic sugar moiety to a hydrophobic alkyl chain. Sugar-based surfactants are being considered for applications including personal-care products, food, and pharmaceuticals because they are considered environmentally sustainable [7]. In addition to their renewable nature, the sugar surfactants display physico-chemical properties that are insensitive to temperature, presumably due to the strength of the hydrogen bonding between the hydroxyl groups of the sugar headgroup and water [8]. Thus, sugar

surfactants have been used in a variety of applications. For example, the commercially available sugar surfactant *n*-dodecyl- β -D-maltoside (DDM) (Figure 1) is commonly used to solubilize membrane proteins without altering their activity for gel electrophoresis [9–11]. Existing sugar surfactants, such as those shown in Fig. 1, use covalent linkages such as acetals, ethers, esters, or amides to tether the hydrophilic sugar head group to a hydrophobic alkyl chain. The syntheses often require selective protection and deprotection steps due to the relatively small reactivity differences of the hydroxyl groups on the sugar moiety [12]. Furthermore, the common covalent linkages of the existing sugar surfactants (Fig. 1) are prone to cleavage in aqueous acidic or basic media. This tendency often limits their use to neutral media.

To ameliorate the disadvantages of the existing sugar surfactants, we recently developed a synthesis route to a new class of sugar surfactants, sugar oxime ether surfactants (SOESurfs), that use the robust oxime ether bond to link the hydrophilic sugar headgroup to a hydrophobic alkyl chain (Fig. 2) [13]. The chemoselective formation of the oxime ether bond [14–18] obviates the synthetic complications involving selective protection and deprotection steps. Furthermore, the well-documented hydrolytic stability of the oxime ether bond [19–21] allows for the use of SOESurfs in acidic and basic media.

In this paper, we describe the SOESurf physicochemical characteristics, specifically their water solubility, thermal stability, melting point, effect on the surface tension of water, and the tendency to form micelles. We quantify the effect on surface tension using the model discussed below.

If the surfactant's affinity for the air-water interface is not dependent on surface coverage, the Gibbs adsorption isotherm can be integrated to yield the Szyszkowski equation: [22]

$$\gamma = \gamma_0 - \frac{RT}{\omega} \ln(K_{ad}c + 1) \quad (1)$$

In this equation, γ is the surface tension of the solution, γ_0 is the surface tension of pure water, ω is the molar lateral surface area at full surface coverage, K_{ad} is the equilibrium constant for surface adsorption, and c is the concentration. Thus, isotherms can be used to calculate the standard free energy change for adsorption to the air-water interface.

$$\Delta_{ad}G^\circ = -RT \ln K_{ad} \quad (2)$$

For surfactants that form micelles, the critical micelle concentration (CMC) can be used to calculate the standard free energy change for micelle formation.

$$\Delta_{mic}G^\circ = RT \ln CMC \quad (3)$$

Based on our results, we are gratified to find that six of the SOESurfs, 1a–3a and 1b–3b, were quite good surfactants: all decreased the surface tension of water to less than 40 mN m⁻¹ at sub-micromolar concentrations.

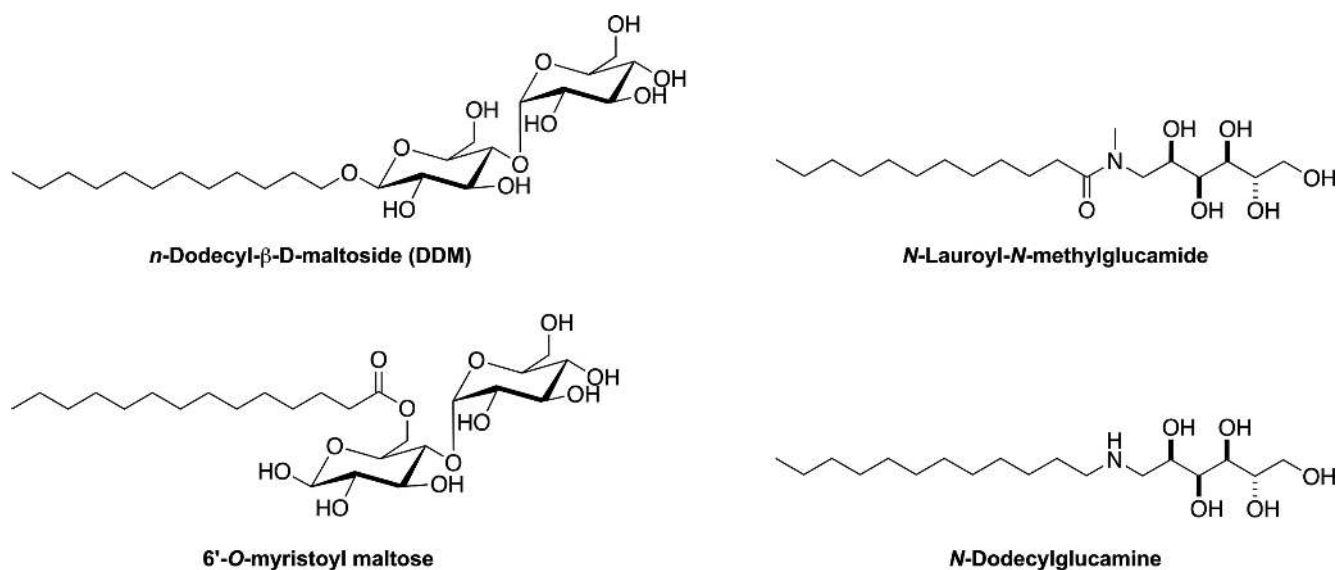


Figure 1 Representative sugar surfactants

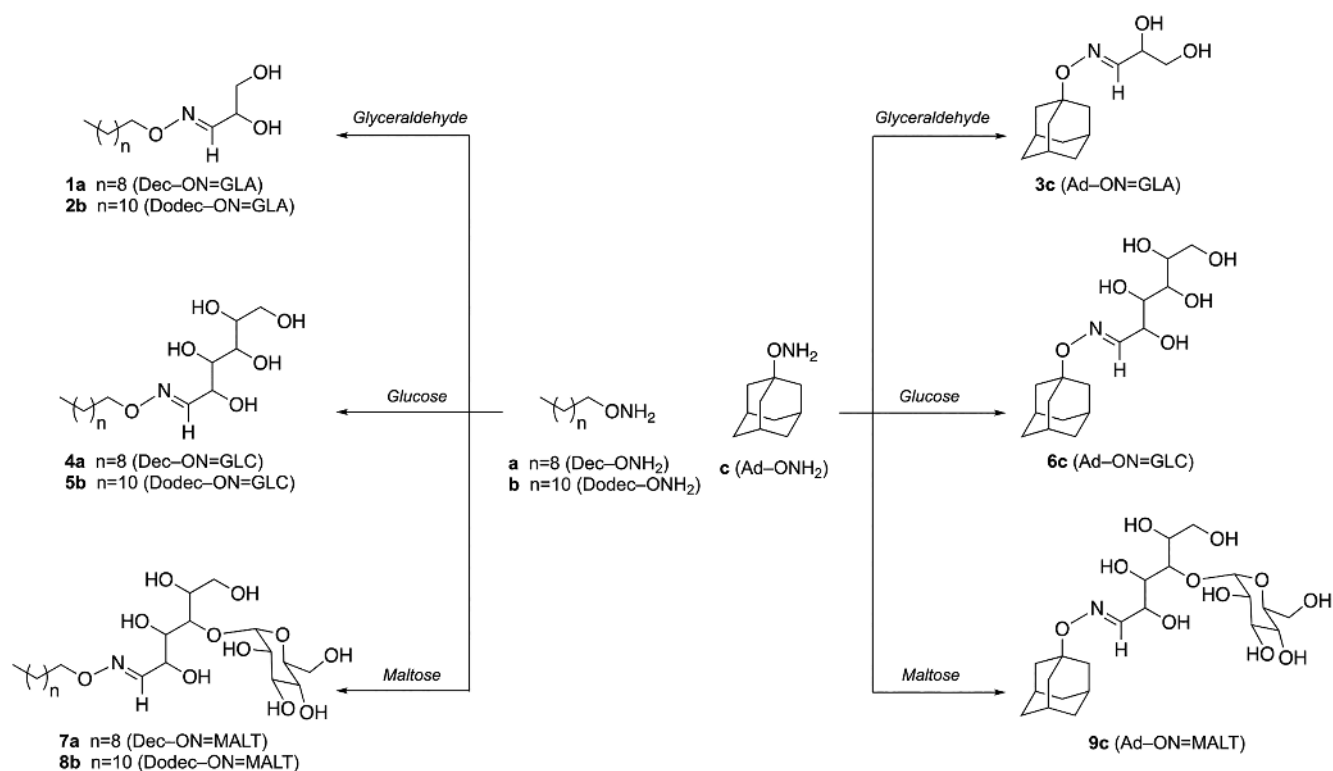


Figure 2 Sugar oxime ether surfactants (SOESurfs). The hydrophobes are decyl-, dodecyl-, and adamantyl-groups, while the sugar hydrophiles are glycerinaldehyde, glucose, and maltose. The abbreviations for the sugar hydrophiles on SOESurf: GLA = glycerinaldehyde; GLC = glucose; MALT = maltose

2 Materials and Methods

2.1 Materials

All SOESurfs were synthesized and purified as described in our other work [13]. The water used for all experiments was 18 MΩ cm produced using a Barnstead NANOpure system.

2.2 Methods

2.2.1 Melting point

The melting points were determined by placing the compounds in 100-mm glass capillary tubes. The tubes were heated while being observed under 6x magnification using Vernier Melt Stations (Vernier Software & Technology, Beaverton, Oregon).

2.2.2 Solubility

The procedure used to determine solubility was adapted from a literature procedure [23]. Approximately 20 mg of the surfactant to be tested was mixed with 20 mL of nanopure water in a vial. The mixture was magnetically stirred overnight at 25 °C. If all solids had dissolved, an additional 10 mg of solid was added, and the overnight stirring was repeated. The mixture was then stirred for 24 h at 25 °C. If the mixture did not have excess solid after 24 h, the addition and stirring steps were repeated. Once a saturated solution had formed, the solution was filtered using a Pasteur pipette packed with glass wool. A 5-mL aliquot of the glass wool filtered, saturated solution was filtered through a 45 µm syringe filter and lyophilized to constant mass over 24–72 h. The mass of the dry residue was used to calculate the solubility.

2.2.3 Thermogravimetric analysis

Thermogravimetric analysis (TGA) was used to determine the thermal stability of the surfactants in an inert (dry nitrogen) atmosphere. A TA Instrument TGA Q500 was used for all experiments, with a nitrogen flow rate of 90 mL min⁻¹ in the sample chamber. The samples were taken from ambient temperature to 300 °C at a rate of 10 °C per minute.

2.2.4 Modeling analysis

The solvent-accessible polar and non-polar area for each surfactant was estimated using SpartanTM (Wavefunction, Inc, Irvine, CA). First, each molecule was subjected to a simple molecular mechanics minimization. Then, the geometry was optimized using density functional theory (B3LYP using a 6–31* basis set), with water as the solvent. The polar and non-polar solvent-accessible areas were calculated using the built-in Quantitative Structure-Activity Relationship (QSAR) option in SpartanTM. The probe radius for calculating the accessible area was 1.0 Å. The cutoff for polar area was a calculated electrostatic potential with an absolute value greater than 75 kJ/mol.

2.2.5 Surface tension isotherms

A KSV Sigma 703 tensiometer employing a platinum DuNoüy ring was used for all surface tension measurements. The DuNoüy ring was cleaned by heating with a propane torch flame until red hot. The instrument was set to report all surface tension measurements using the Zuidema-

Waters correction factor. Stock solutions of each surfactants were prepared at concentrations approximately half of the solubility limit for each compound. A series of 2:1 serial dilutions were prepared, continuing until the most dilute solution was less than 5 µM in surfactant. Solutions were held in a 25 °C water bath until use.

3 Results

3.1 Melting Point and Thermal Stability

The results of melting point and thermogravimetric analysis are shown in Table 1. All the surfactants are stable in dry nitrogen below 200 °C. All surfactants were solids at room temperature.

3.2 Solubility and Surface Adsorption

All the SOESurfs decreased the surface tension of water. One measure of surfactant efficiency is the concentration of surfactant needed to produce a given decrease in surface tension. A commonly used parameter is pC₂₀, the negative base 10 logarithm of the surfactant concentration (in mol L⁻¹) that induces a 20 mN m⁻¹ decrease in surface tension [24]. Efficient surfactants will have high values of pC₂₀. The solubility and the surfactant efficiency, pC₂₀, of SOESurfs in water are summarized in Table 2. The surfactants with maltose headgroups had solubilities that were, on average, an order of magnitude more than the surfactants made from glyceraldehyde or glucose.

The adsorption isotherms of the SOESurfs are shown in Fig. 3. Four of the SOESurfs (**7a**, **8b**, **1a**, **2b**) formed micelles, as shown by the breaks in the isotherms in Fig. 3.

The results from the analyzing the isotherm data in Fig. 3 are shown in Table 3.

Five of the SOESurfs did not form micelles: surfactants with an adamantyl tail (**3c**, **6c**, **9c**) or with a glucose headgroup (**4a**, **5b**). The isotherms for these surfactants are shown in Fig. 4.

The results from the analyzing the isotherm data in Fig. 4 are shown in Table 4.

4 Discussion

4.1 Melting Point and Thermal Stability

For a given hydrophobe, the melting point increased with the size of the sugar in the head group. In addition, the decyl and dodecyl maltose SOESurfs **7a**, **8b** (Fig. 2) became translucent at approximately 80 °C. This may be a solid to liquid-crystal phase transition; similar surfactants, such as decyl α-D-glucopyranoside, dodecyl α-D-glucopyranoside, decyl β-D-glucopyranoside, and dodecyl β-D-glucopyranoside are known to display a solid to smectic liquid crystalline phase transition [25, 26]. However, we did not attempt to characterize this transition.

4.2 Solubility and Surface Adsorption

As expected, SOESurfs with maltose head groups all have relatively high solubility in water. This is likely a consequence of the large polar area of this group, shown by the calculated solvent-accessible polar area (Table 2). Surprisingly, SOESurfs with the adamantyl hydrophobe display high solubility in water despite the rigid nature of the adamantyl group. For solid compounds, solubility normally is lower for rigid compounds, since flexible molecules gain more entropy when

Surfactant(tail-ON=sugar)	T _{dec} /°C	T _m /°C
Ad-ON=GLA (3c)	215	81-82
Ad-ON=GLC (6c)	217	104-107
Ad-ON=MALT (9c)	221	114-117
Dec-ON=GLA (1a)	214	39-41
Dec-ON=GLC (4a)	219	169-171
Dec-ON=MALT (7a)	204	decomposes
Dodec-ON=GLA (2b)	202	52-54
Dodec-ON=GLC (5b)	237	146-148
Dodec-ON=MALT (8b)	215	decomposes

Table 1 Decomposition and Melting Temperatures of SOESurfs

SOESurf (tail-ON=sugar)	Solubility/mM	Efficiency (ρC_{20})	Polar Area/ \AA^2	Nonpolar Area/ \AA^2
Ad-ON=GLA (3c)	14 ± 1	2.54 ± 0.05	27	131
Ad-ON=GLC (6c)	26 ± 2	2.16 ± 0.06^a	59	137
Ad-ON=MALT (9c)	77 ± 4	1.99 ± 0.10	79	161
Dec-ON=GLA (1a)	5.9 ± 0.5	4.60 ± 0.10	31	172
Dec-ON=GLC (4a)	2.4 ± 0.2	4.06 ± 0.06	48	185
Dec-ON=MALT (7a)	94 ± 2	3.70 ± 0.11	84	215
Dodec-ON=GLA (2b)	1.7 ± 0.2	4.60 ± 0.12	32	194
Dodec-ON=GLC (5b)	1.6 ± 0.1	4.42 ± 0.12	47	206
Dodec-ON=MALT (8b)	80 ± 8	4.61 ± 0.03	84	237

^a This value was obtained by extrapolation.

Table 2 Solubility and Adsorption Efficiency at 25 °C

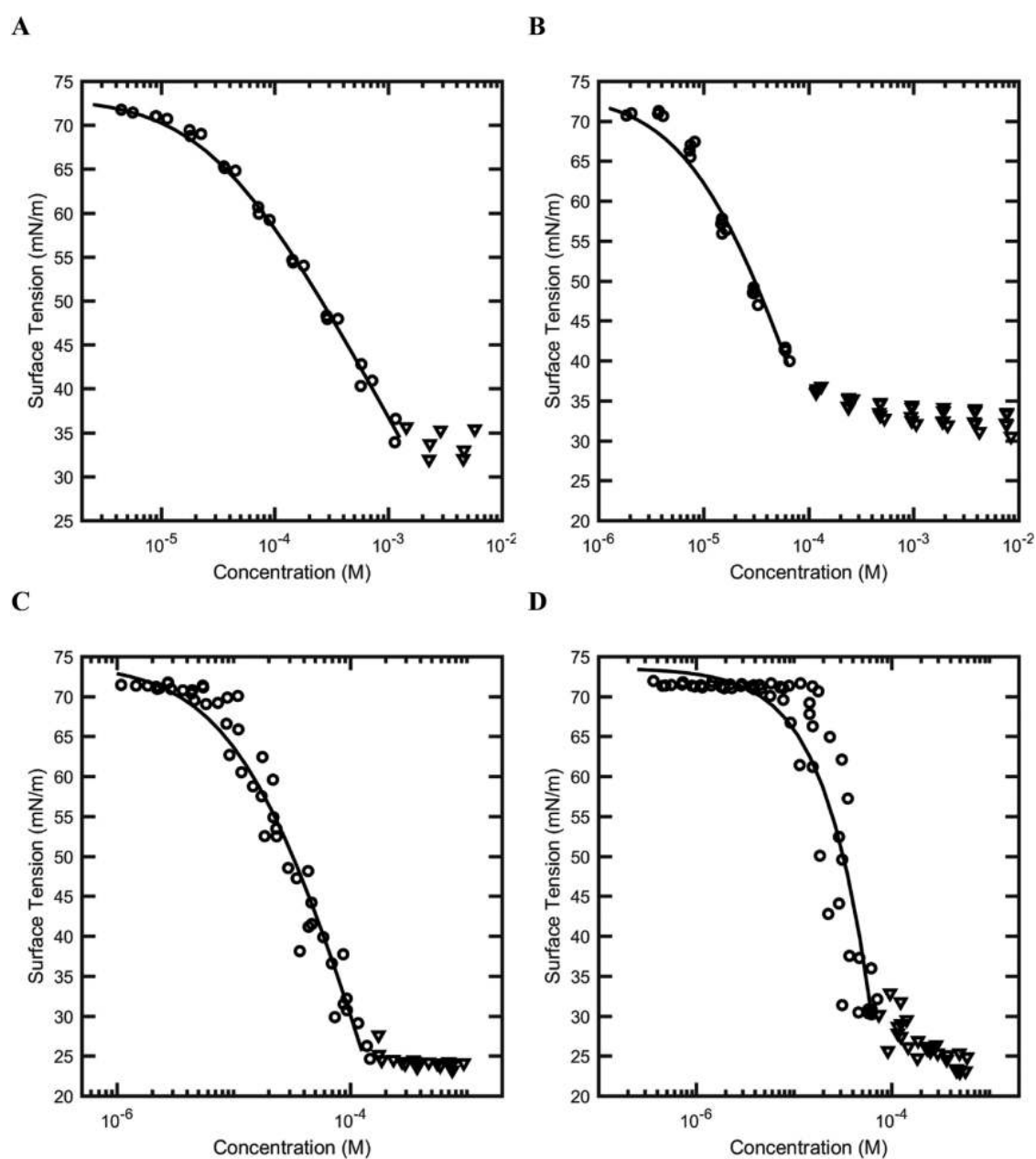


Figure 3 The surface tension isotherm data for four of the SOESurfs show a break at the critical micelle concentration. Data at concentrations lower than the CMC (o) were fit to the Szyszkowski equation; data above the CMC (∇) were not. (A) Dec-ON=MALT (7a), (B) Dodec-ON=MALT (8b), (C) Dec-ON=GLA (1a), (D) Dodec-ON=GLA (2b)

they dissolve [27]. However, SOESurfs with the adamantyl hydrophobe have a lower solvent-accessible non-polar area than those with a straight chain hydrophobe (Table 2), which probably accounts for their higher than expected solubility in water.

The adsorption isotherms of all nine SOESurfs indicate that they are effective surfactants. The SOESurfs with a dodecyl hydrophobe have surfactant efficiencies very close to that of the commercially-available *n*-dodecyl- β -D-maltoside, with a pC_{20} of 4.52 [28]. The SOESurfs with a decyl hydrophobe have efficiencies similar to that of the commercially-available *n*-decyl- β -D-maltoside, with a pC_{20} of 3.77 [29].

For surfactants with the same hydrophilic headgroup we observed that the polar area varies very little with the type of hydrophobic tail. For a given headgroup, the polar areas are essentially the same for surfactants with dodecyl and decyl tails; there was no clear pattern for the adamantyl tails. In contrast, the non-polar area increased in the order adamantyl < decyl < dodecyl regardless of the headgroup. Since adsorption is driven by the hydrophobic effect, we were not surprised to see that, in general, the surfactant efficiency increased in the same order: adamantyl \ll decyl < dodecyl.

For surfactants with the same hydrophobic tails we observe that, as expected, polar area increased with the num-

ber of hydroxyl groups of the sugar hydrophilic headgroup: GLA < GLC < MALT. Nonpolar area also increased, but not nearly as much. The increasing polar area appears to decrease the affinity of the surfactants for the surface, as the surfactant efficiency decreased in the same order: GLA > GLC > MALT. The sole exception to this trend was compound **8b**, which had the highest efficiency of the surfactants despite bearing a maltose headgroup.

We see in Table 3 that the SOESurfs with the bulky maltose headgroups had larger molar surface areas than those with glyceraldehyde. The magnitudes of the free energies for micelle formation and adsorption at the air-water interface are higher for SOESurfs with dodecyl tails than for those with decyl tails.

Finally, it is important to note that the magnitude of the micelle formation free energy is always less than that for adsorption at the air-water interface. A rough estimate of this difference in energy can be calculated by accounting for the portion of $\Delta_{ad}G^\circ$ due to the decrease in surface energy at the air-water interface:

$$\Delta_{ad}G^\circ - \Delta_{mic}G^\circ \approx (\gamma_{CMC} - \gamma_0)\omega \quad (4)$$

SOESurf	ω /m ² mmol ⁻¹	$\Delta_{ad}G^\circ$ /kJ mol ⁻¹	CMC/ μ M	$\Delta_{mic}G^\circ$ /kJ mol ⁻¹
Dec-ON=GLA (1a)	103 \pm 8	-26.2 \pm 0.9	160	-21.6 \pm 0.5
Dec-ON=MALT (7a)	220 \pm 40	-24.9 \pm 0.8	1460	-16.1 \pm 0.4
Dodec-ON=GLA (2b)	80 \pm 40	-26 \pm 2	56	-23.7 \pm 0.8
Dodec-ON=MALT (8b)	190 \pm 30	-29.1 \pm 0.9	110	-22.4 \pm 0.5

^a All free energy values were calculated using a 1.0 M standard state.

Table 3 Molar Area and Free Energy of Adsorption and Micelle Formation at 25 °C

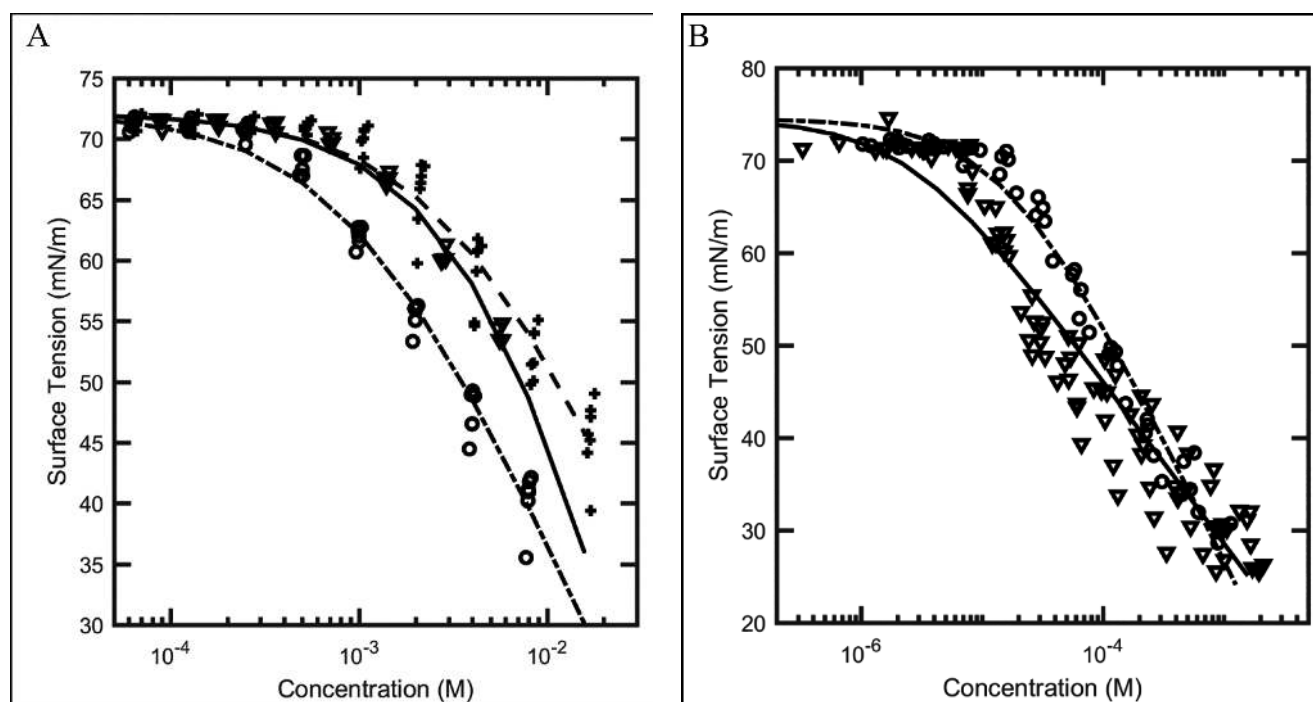


Figure 4 The surface tension isotherm data for non-micelle forming surfactants: (A) Ad-ON=GLA (**3c**) (o), Ad-ON=GLC (**6c**) (∇), Ad-ON=MALT (**9c**) (+) and (B) Dec-ON=GLC (**4a**) (o) and Dodec-ON=GLC (**5b**) (∇). Note that surfactants with an adamantyl tail (**3c**, **6c**, and **9c**) are much less effective at reducing surface tension significantly at low concentration.

SOESurf	$\omega/\text{m}^2 \text{ mmol}^{-1}$	$\Delta_{\text{ad}}G^\circ/\text{kJ mol}^{-1}$
Ad-ON=GLA (3c)	166 ± 8	-16.8 ± 0.4
Ad-ON=GLC (6c)	80 ± 20	-12.1 ± 0.2
Ad-ON=MALT (9c)	120 ± 30	-12.6 ± 0.8
Dec-ON=GLC (4a)	200 ± 30	-26.1 ± 0.8
Dodec-ON=GLC (5b)	300 ± 20	-30.7 ± 0.9

^a All free energy values were calculated using a 1.0 M standard state.

Table 4 Molar Area and Free Energy of Adsorption at 25 °C

Here γ_{CMC} is the surface tension at the CMC. For example, the molar area for Dec-ON=GLA is $103,000 \text{ m}^2 \text{ mol}^{-1}$ and γ_{CMC} is $\sim 25 \text{ N m}^{-1}$; thus, the predicted difference between of $\Delta_{\text{ad}}G^\circ$ and $\Delta_{\text{mic}}G^\circ$ is 4.8 kJ mol^{-1} . The observed difference is 4.6 kJ mol^{-1} . This difference is larger for the maltose-based SOESurfs, which is expected as a consequence of their large headgroups.

The presence of the oxime ether linkages does not have a measurable effect on the value of the free energy of micelle formation, as shown by a comparison of the value for Dodec-ON=MALT, $-22.4 \text{ kJ mol}^{-1} \pm 0.5 \text{ kJ mol}^{-1}$, with that of the commercially-available surfactant *n*-dodecyl- β -D-maltoside, $-21.8 \text{ kJ mol}^{-1}$ [29].

The low affinity of the SOESurfs with adamantyl tails for the air-water interface is striking. These surfactants are less hydrophobic than the others because of their lower hydrophobic accessible area, as shown in Table 2.

4.3 CMC and molecular structure

We expected that surfactants with longer chains would have smaller CMC values because of their greater hydrophobicity. This was observed in the data: changing from a decyl to a dodecyl chain decreased the CMC for all surfactants that formed micelles. The size of the decrease was different for different headgroups: a factor of approximately three for surfactants with GLA headgroups and a factor of approximately 13 for surfactants with MALT headgroups.

The headgroup type affects the CMC: Surfactants with a GLA headgroup had smaller CMC values than surfactants with MALT headgroups. The decrease in CMC was a factor of about nine for decyl-chain surfactants and about two for dodecyl-chain surfactants.

5 Conclusions


The oxime ether linkage provides a route to chemically robust surfactants sourced from a renewable feedstock. All nine SOESurfs tested were effective at decreasing surface tension. SOESurfs with straight-chain hydrophobes all resulted in substantial surface tension decreases at micromolar concentrations; SOESurfs with adamantyl hydrophobes did this only at millimolar concentrations. Our results suggest several areas for future work. First, the suspected liquid crystalline phase behavior for Dec-ON=MALT and Dodec-ON=MALT could be characterized. Second, since the most promising surfactants were based on maltose and a straight-chain hydrophobe, we would like to synthesize and characterize maltose-based SOESurfs with 14 and 16-carbon chains. Finally, SOESurfs with large hydrophobes would be water insoluble. It is possible that such compounds would form bilayers and vesicles.

Acknowledgements

We gratefully thank the Department of the Navy, Office of Naval Research, under Award # N00014-11-1-0359 for sponsoring this work.


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Received: 18. 06. 2019

Revised: 08. 10. 2019

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DOI 10.3139/113.110660
Tenside Surf. Det.
57 (2020) 1; page 6–12
© Carl Hanser Verlag GmbH & Co. KG
ISSN 0932-3414

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