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RESEARCH ARTICLE

Straight and Branched Sucrose Monoamide Derivatives as New Surfactants

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Abstract: We have synthesized monoamide analogs of sucrose. The synthesis applied a multi steps synthesis scheme ended by Staudinger reaction based coupling of a sucrose azide with fatty acid chlorides. Sucrose monoamide surfactants are easily accessible surfactants from sucrose, which is consider as the cheapest natural carbohydrate resources. The assembly behavior of synthesized monoamide derivatives was investigated by optical polarizing microscopy, Differential scanning calorimetry DSC and surface tension studies. These surfactants exhibit very good solubility in water at room temperature and value of CMC within the range of disaccharides surfactants, which make those surfactants fitting the target application as water-in-oil emulsions.

Keywords: Bioactive surfactants, Monoamide surfactants, Staudinger reaction, Carbohydrate surfactants, Oil -in-water emulsion, Hexagonal phase and sugar based surfactants

Introduction

Sucrose is a non-reducing disaccharide that occurs naturally in most fruit and vegetable. The structure is shown in Figure 1. It is produced in large quantities, *e.g*. in sugar cane and sugar beets, from which it is isolated for commercial use. The annual production of sucrose is about 168 million tons. Sucrose is the less expensive carbohydrate on the world market. Sucrose production exceed world market demand by over million tons[1]. Because of that, more application of sucrose is needed[2]. The aim of this series is to synthesize sucrose monoamides by replacement of the hydroxyl group at position 6 of fructose with straight and branched C_{12} alkyl chains via Staudinger reaction. The target application of sucrose surfactants are water -in oil emulsifier.

Due to the three primaries and five secondary hydroxyl groups (Figure 1), sucrose will be a great candidate for water-in-oil emulsions, which is fitting the target application for those surfactants, but the chemical reactions of unprotected sucrose in a single hydroxyl group is a big challenge. The suitable reactivity differences between primary and secondary hydroxyl groups have been generalized such that the three primary ones alkylated, acylated, oxidized and displaced by halogen in the order 6-OH glucose = 6-OH fructose >>1-OH fructose, an over generalization as this order of reactivity mainly cover comparatively bulky reagents, which necessarily favor reaction at the 6-OH glucose and 6-OH fructose groups[3].

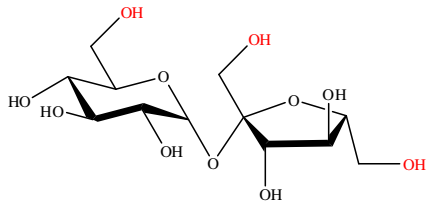

Figure 1. Chemical structure of sucrose

Experimental

Melting points were determined using a manual melting point apparatus and are uncorrected. Optical rotations were measured at 589 nm in 10 cm cells at room temperature. NMR spectra were recorded on Jeol and Bruker spectrometers at 400 MHz for ^{1}H and 100 MHz for ^{13}C, respectively. Assignments of ^{13}C-signals are based on HMQC spectra[4,5]. High-resolution mass spectra were recorded on an LC–MS system, applying MeOH/water eluents. Phase transition temperatures were determined by DSC in replicated heating–cooling cycles at a heating/cooling rate of 10 ^{o}C min. Lyotropic phases were investigated using the contact penetration technique under OPM observation[6]. The determination of Krafft points applied heating 20 mL samples of the surfactant in water at a concentration of about 10% above the CMC in an oil bath under moderate stirring until the mixture cleared. Critical micelle concentrations were determined by surface tension measurements[7-9]. The intersection of the concentration dependent and the high concentration independent region in the plot of the surface tension *versus* the logarithmic concentration determines the CMC. Surface tension measurements were measured at r.t. in 5 replicates with a standard deviation below 0.1 mN m.

General procedures

Tert-butyldiphenylsilylation

4-Dimethylaminopyridine (0.026 eq.) was added to the mixture of sucrose (1.0 eq.) and tert-butyldiphenylsilyl chloride (1.1 eq.) in pyridine (25 mL per one gram). The mixture was stirred at room temperature and monitored by TLC using (ethyl acetate-acetone-water, 10:10:1) as eluent. When the TLC showed consumption of the starting materials, the mixture was concentrated and used for the next reaction without further purification.

Acetylation

Acetic anhydride (2 mL per one hydroxyl group) was added to sugar derivatives (1.0 eq.) in pyridine. The mixture was stirred at room temperature and monitored by TLC using hexane-ethyl acetate 3:1 as an eluent. When the TLC showed the consumption of the starting materials, the solvent was removed at reduced pressure. The residue was dissolved in dichloromethane and washed with sodium hydrogen carbonate solution and water, dried over magnesium sulfate and concentrated. The compound was used for the next reaction without further purification.

Desilylation

The solution of tert-butyldiphenylsilyl-sucrose peracetate (1.0 eq.) in tetrahydrofuran (25 mL per one gram) was treated at room temperature with tetra-butylammonium fluoride (1.1eq. 1 M solution in THF). The reaction was monitored by TLC (hexane- ethyl acetate, 3:1), When the starting material was consumed (about 5 hours), the solvent was evaporated and the residue was dissolved in dichloromethane (DCM). The organic solution was washed with water, dried over magnesium sulfate and concentrated. The compound was utilized for the next reaction without further purification.

Tosylation

To the solution of sugar derivatives (1.0 eq.) in pyridine (25 mL per gram) was added tosyl chloride (1.0 eq.). After about 24 hours TLC (hexane-ethyl acetate 3:1) showed consumption of the starting material. The mixture was poured into ice water and extracted with dichloromethane for three times. The combined extracts were evaporated and the residue was subjected to azidation without further purification.

Azidation

A suspension of sugar tosylate (1.0 eq.) and sodium azide NaN_3 (6.0 eq) in *N,N*-dimethylformamide DMF (20 mL per gram) was heated to 80 ºC for 24 hours. The solution was cooled to room temperature, diluted with water and extracted with dichloromethane. The organic layer was washed with water, saturated $NaHCO_3$ solution and water, dried over $MgSO_4$ and concentrated under reduced pressure. After acetylation with acetic anhydride (2.0 eq.) in pyridine (20 mL per gram) and recrystallization with ethanol NMR pure white azide was obtained in very good yield.

Staudinger reaction

Fatty acid chloride (1.6 eq.) in (5 mL) of dichloromethane was added drop wise at room temperature to a mixture of acetylated sugar azide (1 eq.) and triphenylphosphine (1.2 eq.) in (20 mL) of dichloromethane. Stirring was continued for about 15 hours at room temperatureto obtain cloudy solution. The solid was filtered of and the solution was washed with 5% solution of sodium hydrogen carbonate, dried over magnesium sulfate and evaporated to dryness. The resulting syrup was chromatographed on silica gel with 6:1 ethyl acetate- acetone as eluent to affordthe protected derivatives as white NMR pure crystals.

Deacetylation

The peracetylated amide derivatives was dissolved in methanol (25 mL per one gram) and treated with a catalytic amount of sodium methoxide to obtain a basic medium.The mixture was stirred for 24 hours at room temperature and monitored by TLC (dichloromethane: ethanol 9:1). The basic catalyst was removed with Amberlite IR 120 (H^+), the solid was filtered and methanol was evaporated to furnish the surfactant in approximately quantitative yield.

*Synthesis of 1',3',4'-hepta-O-acetyl-6'-O-dodecanamido-sucrose (**7a**)*

Compound **6** (1.5 g, 2.27 mmol) was coupled with dodecanoyl chloride $C_{11}H_{23}COCl$ and triphenylphosphine PPh_3 according to the Staudinger reaction to produce compound **7a** (1.25 g, 68%) as NMR pure white crystals. $[\alpha]_D^{25}$= + 33.0 (c 0.15, $CHCl_3$).^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.62 (d, 1H, H-1), 5.42 (dd~t, 1H, H-3), 5.39 (dd~t, 1H, H-3'), 5.19 (dd~t, 1H , H-4), 5.02 (dd~t, 1H, 4'-H), 4.82 (dd, 1H, 2-H), 4.05 - 4.44 (m, 6H, H-1'A, H-1'B, H-5 , H-5', H-6B, H-6'A), 3.70 (ddd, 1H, H-6A), 3.32 (ddd, 1H, H-6'B), 2.17 (m. 2H, α-CH_2), 2.14 , 2.09, 2.08, 2.07, 2.06, 2.02, 1.99 (7s, 21H, Ac), 1.61 (m, 2H, β-CH_2), 1.22 (s, 16H, bulk-CH_2), 0.85 (t, 3H, CH_3). $^3J_{1,2}$=3.5, $^3J_{2,3}$ =10.0, $^3J_{3',4'}$=9.5, $^2J_6$ =14, $^2J_{6'}$=13.0, $^3J_{6',NH}$ =5.5. Hz.^{13}C NMR (100 MHz, $CDCl_3$) δ =173.54 (CONH), 170,66 x2, 170.11 x2, 196.98, 196.63, 196,56 ((C=O), 103.86 (C-2'), 89.80 (C-1), 79.90 (C-5'), 75.85 (C-3'), 75.83 (C-4'), 70.41 (C-2), 69.37 (C-3), 68.67 (C-5) , 68.29 (C-4), 63.40 (C-6), 61.76 (C-1'), 41.37 (C-6'), 36.50 (α-CH_2),31.88 (ω-2), 29.61, 29.59, 29.48, 29.36, 29.31 x2 (bulk-CH_2), 25.54 (β-CH_2), 22.66 (ω-1), 20,75, 20.73, 20.66 x2, 20.63, 20.60, 20.57(Ac), 14.09 (ω). Elemental analysis for $C_{38}H_{59}NO_{18}$: C55.81, H 7.27, N 1.71; found C 55.84, H 7.33, N 1.75.

*Synthesis of 6'-dodecanamido-sucrose (**7b**)*

Compound **7a** (1 g) was deacetylated with sodium methoxide in methanol according to Zemlplen reaction to give **7b** (0.6 g, 94%) as NMR pure white crystals.mp 204 ^{o}C (dec.).$[\alpha]_D^{25}$= +43.0 (c 0.15, CH_3OH).^{1}H NMR (400 MHz, CD_3OD) δ= 3.5 (d 1H, H-1 , 4.07 (d, C-5'), 3.89- 3.59 (m, 7H, H-2', H-3', H-3, H-6, H-4, 2H-6'), 2.42 (dd~t, 1H, H-2), 3.48 (dd~t, 1H, H-1'A), 3.28 (m, 1H, H-1'B), 2.21 (m, 2H, α-CH_2), 1.61 (m, 2H, β-CH_2), 1.31 (s, 16H, bulk-CH_2), 0.91 (t, 3H, CH_3) Hz, $^3J_{1,3}$=3.5, $^3J_{2,3}$ =10.0, $^2J_6$=13.5).

^{1}H NMR (400 MHz, CD_3OD) δ= 175.58 (CONH), 104.67 (C-2'), 92.16 (C-1), 80.19 (C-3'), 78.10 (C-5', 77.00 (C-5), 73.37 (C-3), 73.08 (C-4'), 72.08 (C-2), 70.64 (C4), 63.23 (C-6), 61.42 (C-1'), 42.29 (C-6') ,35.75 (α-CH_2), 31.73 (ω -2), 29.40 x3, 29.12 x2, 28.99 x3, 28.94 x2 (bulk-CH_2), 25.64 (β-CH_2), 22.38 (ω-1), 13.08 (ω). HRMS: $[M+H]^+$: calcd for $C_{24}H_{45}NO_{11}$: 524.3065 (100%), 525.3098 (28 %); found : 524.3039 (100 %), 525.3064 (25 %); $[M+Na]^+$: calcd for $C_{24}H_{45}NO_{11}Na$: 546.2884 (100%), 547.2917 (28%; found: 546. 2878 (100%), 547.2905 (28%).

*Synthesis of 2,3,4,6,1',3',4'-hepta-O-acetyl-6'-O-(2-butyl-octanamido)-sucrose (**8a**)*

Compound **6** (1.g, 2.27 mmol) was coupled with 2-butyloctanoyl chloride C_6H_{13} (COCl) C_4H_9 and triphenylphosphine PPh_3 according to the Staudinger reaction to furnish compound **8a** (1.g, 65%) as NMR pure white crystals.$[\alpha]_D^{25}$= + 27.0 (c 0.15, $CHCl_3$).^{1}H NMR (400 MHz, CD_3OD) δ = 5.17(d, 1H, H-1), 5.48 (dd~t, 1H, H-3), 5.46 (dd~t, 1H, H-3'), 5.06 (dd~t, 1H, H-4, 5.32 (dd~t, 1H, 4'-H), 4.94 (dd, 1H, 2-H), 4.23-4.14 (m, 4H, H-1'B, H-5 , H-5', H-6B, H-6'A), 3.71 (ddd, 1H, H-6A), 2.22 (m. 2H, α-CH_2), 3.32 (1H,-CH), 2.19, 2.12, 2.11 ,2.09, 2.08, 2.06. 202 (7s. 21H, Ac), 1.58 (m , 2H, β-CH_2), 1.29 (m, 16H, bulk-CH_2), 0.91 (t, 3H, CH_3). $^3J_{1,2}$=3.5, $^3J_{2,3}$ =10.0, $^3J_{3',4'}$=9.5, $^2J_6$ =1, Hz.^{13}C NMR (100 MHz, CD_3OD) δ = 177.39 (CONH), 170,.41, 170.34 x2, 170.30, 196.92, 196,90 (C=O), 88.96 (C-2'), 79.92 (C-1), 76.27(C-5'), 76.07 (C-3'), 70.25 (C-4'), 69.58 (C-2), 68.69 (C-3), 68.48 (C-5), 68.29 (C-4), 62.95 (C-6), 62.07 (C-1'), 41.71 (C-6), 32.84. 32.74 (α-$CH_2$29.61, 29.59, 32.50, 32.37, 31,98 (bulk-CH_2), 31.4, 31.31 (β-CH_2), 22.66 (ω-1), 29.47, 29.00, 28.95 (Ac), 19.30, 19.15 (ω). Elemental analysis for $C_{38}H_{59}$ NO_{18}: C55.81, H 7.27, N 1.71; found C 55.85, H 7.38, N 1.74.

*Synthesis of 6'-(2-Butyl-octanamido)-sucrose (**8b**)*

Compound **8a** (1 g) was deacetylated with sodium methoxide in methanol according to the Zemlplen reaction to give compound **8b** (0.59 g, 92%) as NMR pure white crystals. $[\alpha]_D^{25}$= + 43.0 (c 0.15, CH_3OH).^{1}H NMR (400 MHz, CD_3OD) δ= 5.38 (d , 1H , H-1), 4.07 (d, C-5'), 3.89- 3.49 (m, 7H, H-2', H-3',H-3, H-6, H-4, 2H-6'), 3.44(m, 1H, H-2), 3.29 (m, 2H, H-1'A, 1'B), 2.20 (m, 2H, α-CH_2), 1.56 (m, 2H, β-CH_2), 1.35 (s, 16H, bulk-CH_2), 0.91 (t, 3H, CH_3) Hz, $^3J_{1,3}$=3.5, $^3J_{2,3}$ =10.0 , $^2J_6$=13.5).

^{1}H NMR (400 MHz, CD_3OD) δ= 177.88 (CONH), 104.11 (C-2'), 92.08(C-1), 80.28 (C-3'), 78.52 (C-5'), 77.26 (C-5), 73.34 (C-3), 73.16 (C-4'), 71.97(C-2), 70.46 (C4), 62.97 (C-6), 61.50 (C-1'), 42.50 (C-6'), 32.86, 32.76, 32.53, 32.44 (α-CH_2), 31.53x2 (ω-2), 29.49x3, 29.42, 29.11, 29.05 (bulk-CH_2), 27.21, 27.14 (β-CH_2), 22.39, 22.35, 22.72 (ω-1), 13.01, 12.94 (ω). HRMS: $[M+H]^+$: calcd for $C_{24}H_{45}NO_{11}$: 524.3065 (100%), 525.3098 (28%); found : 524.3163 (very weak); $[M+Na]^+$: calcd for $C_{24}H_{45}NO_{11}Na$: 546.2884 (100%), 547.2917 (28%); found : 546. 2988 (100%), 547.3017 (24%).

Results and Discussion

Synthesis

The synthetic scheme is pictured in Figure 2. The reaction of sucrose with 1.1 eq. of the bulky reagent *tert*-butyldiphenylsilyl chloride (TBDPSCl) led to the sole protect of the hydroxyl group at position 6 of the fructose moiety by yield 6'-O-tert-butyldiphenylsilyl sucrose (**2**)[10-12]. Afterword remaining hydroxyl groups can be easily acylated with acetic anhydride in pyridine to form the fully protected sucrose compounds 1',2,3,3',4,4',6-hepta-*O*-acetyl-6'-*tert*-butyldiphenylsilyl-sucrose (**3**)[13]. The hydroxyl group at position 6 of fructose can be selectively deprotection with tert-butylammonium fluoride (TBAF) in tetrahydrofuran (THF) to obtain the partially protected 1',2,3,3',4,4',6-hepta-*O*-acetyl-6'- sucrose (**4**)[14].

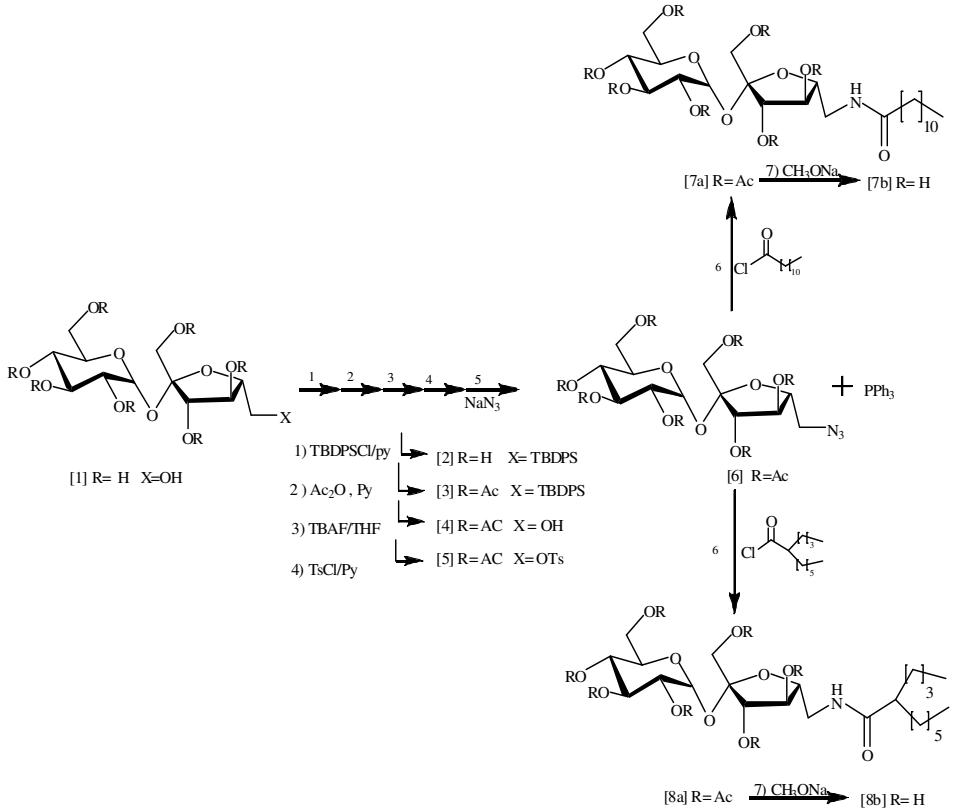

Figure 2. Synthesis of sucrose monoamide surfactants

Activation of 6'-OH applied in pyridine to furnish 1',2,3,3',4,4',6-hepta-*O*-acetyl-6'-tosyl-sucrose (**5**)[15]. The tosyl was substituted with sodium azide in *N,N*-dimethylformamide DMF at 80 ^{o}C to give the surfactant precursor 1',2,3,3',4,4',6-hepta-*O*-acetyl-6'-azido-sucrose (**6**)[16,17]. Coupling of the latter with dodecanoyl chloride and 2-butyloctanoyl chloride via Staudinger reaction in presence of tryphenylphosphine furnished the actylated surfactants 1',2,3,3',4,4',6-hepta-*O*-acetyl-6'-dodecanamido-sucrose **(7a)** and 1',2,3,3',4,4',6-hepta-*O*-acetyl-6'-2-butyl-octanamido-sucrose (**8a**) respectively[18-20]. Final deacetylation under Zamplen conditions led to NMR pure surfactants 6'-dodecanamido-sucrose (**7b**) and 6'-2-butyl-octanamido-sucrose **(8b)**[21-23].

^{1}H NMR of the acetylated surfactants (Figures 3 and 4) shows the signals of the amide proton at about δ 6.00. The sugar protons appear between δ 5.62 (H-1) and δ 4.40 (H-5). The primary proton of fructose found at 3.32 (H-6'B), which indicate the replacement of the oxygen with amide. Acetyl groups are found at about δ 2.00. The alkyl chain protons appear between δ 2.2 (α-CH_2) and δ 0.85 (terminal-CH_3). In ^{13}C NMR (Figures 5 & 6) the signal for the amide carbon appears at about δ173, whereas acetyl carbons of the pyranose are found between δ 170.5 and δ 170, while for furanose found between δ 197 and δ 196.5, Sugar carbons appear between δ 103 (C-2') and δ 61 (C-1'). The signal of (C-6') found at δ 41 which indicate the replacement of oxygen with amide. Alkyl chain carbons appear between δ 36.5 (α-CH_2) and δ 14.00 (terminal CH_3).

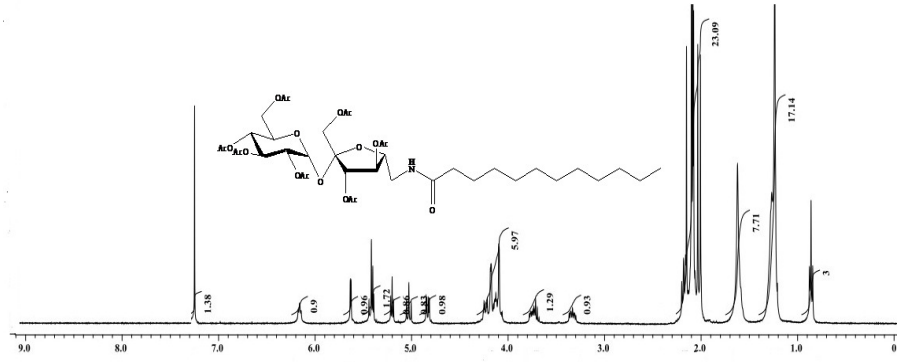

Figure 3. ^{1}H NMR of the acetylated surfactant (**7a**)

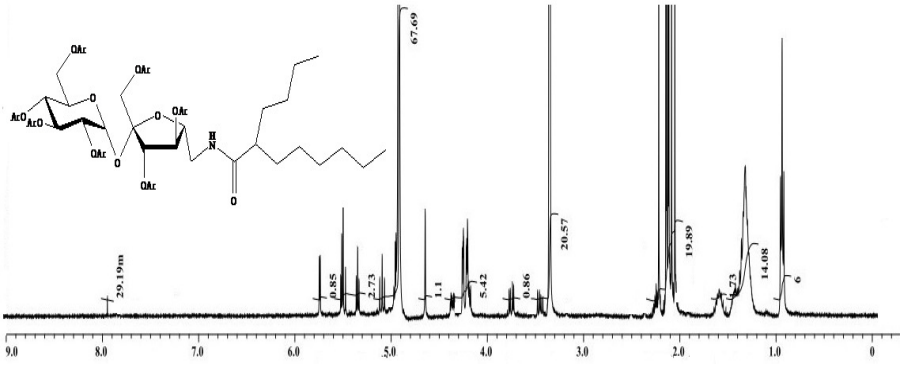

Figure 4. ^{1}H NMR of the acetylated surfactant (**8a**)

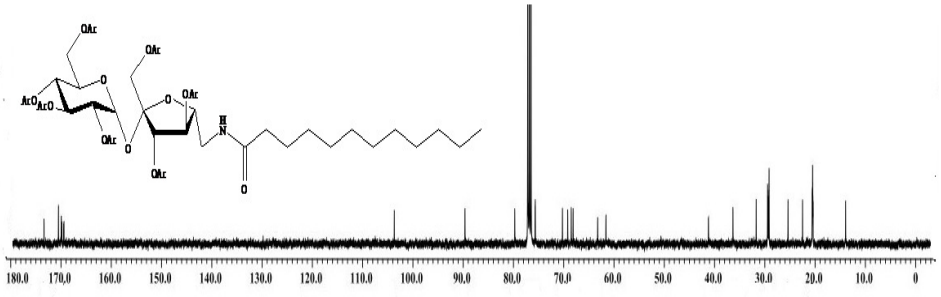

Figure 5. ^{13}C NMR of the acetylated surfactant (**7a**)

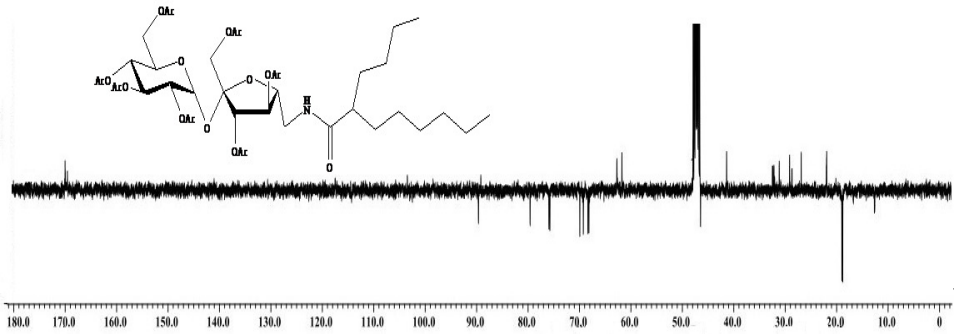

Figure 6. ^{13}C NMR of the acetylated surfactant (**8a**)

The ^{1}H NMR of deacetylated surfactants are shown in Figures 7 and 8, the signal of the amide proton in about δ 7.80, the protons of the sugar ring in between δ 4.00 and δ 3.30, while the protons of the alkyl chain appear between δ 2.21 (α-CH_2) and δ 0.91 (terminal CH_3). The ^{13}C NMR spectra (Figures 9 & 10) shows the signal of the amide carbon at about δ 175.5, the signals of sugar carbons in between δ 92.16 (C-1) and δ 61.4 (C-1'), The primary carbon of the fructose found at δ 42.29 (C-6') reflecting the replacement of the hydroxyl group with amide, and the signal of the alkyl chain carbons in between δ 35.75 (α-CH_2) and δ 13.08 (terminal-CH_3).

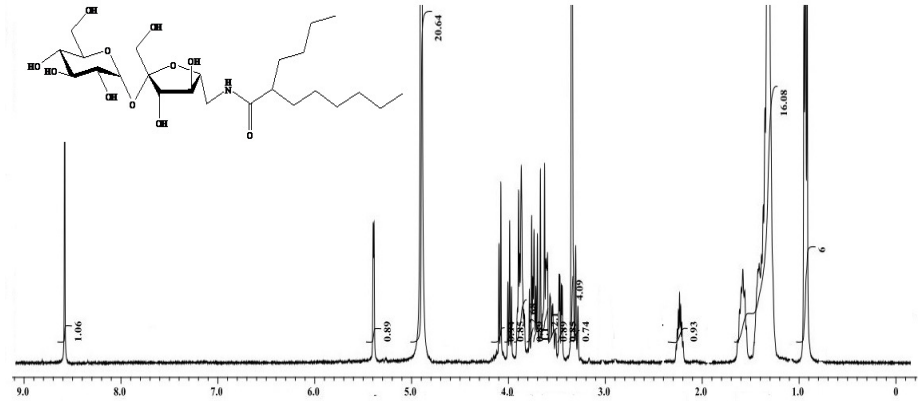

Figure 7. ^{1}H NMR of the deacetylated surfactant (**7b**)

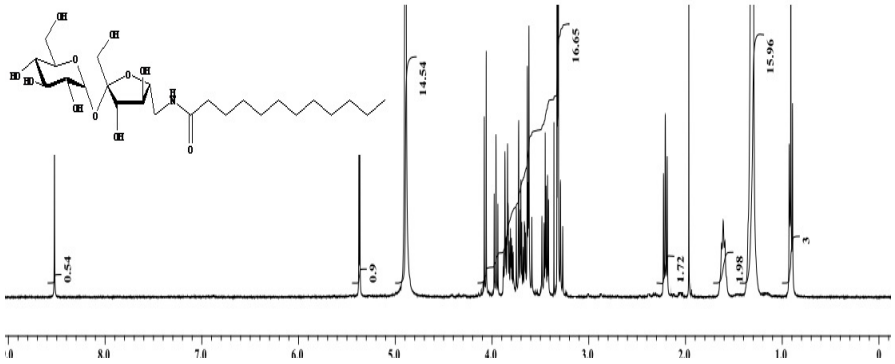

Figure 8. ^{1}H NMR of the deacetylated surfactant (**8b**)

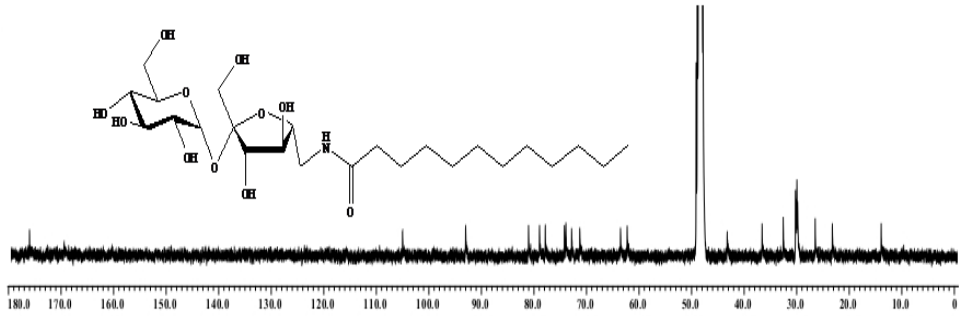

Figure 9. ^{13}C NMR of the deacetylated surfactant (**7b**)

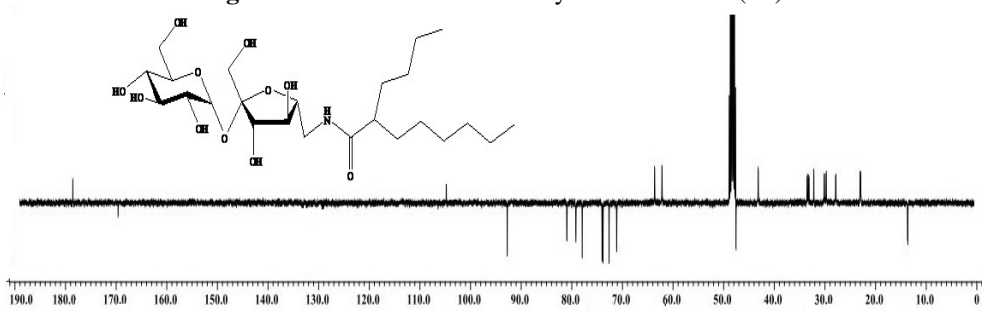

Figure 10. ^{13}C NMR dept. of the deacetylated surfactant (**8b**)

Physical properties

Both sucrose amide surfactants C_{12} straight and corresponding branched are soluble in water at room temperature. The surfactants only exhibit crystalline as a thermotropic phase. The straight surfactant melts at 158 °C with an enthalpy of 66 kJ/mol, as shown in Figure 11. The compound does not crystalline upon cooling. OPM investigations suggest a (partial) decompose of the compound at about 190 °C. In contact with water the micellar phase, L_1 is followed by strong birefringent phase refers to hexagonal phase, H_1 (Figure 12).

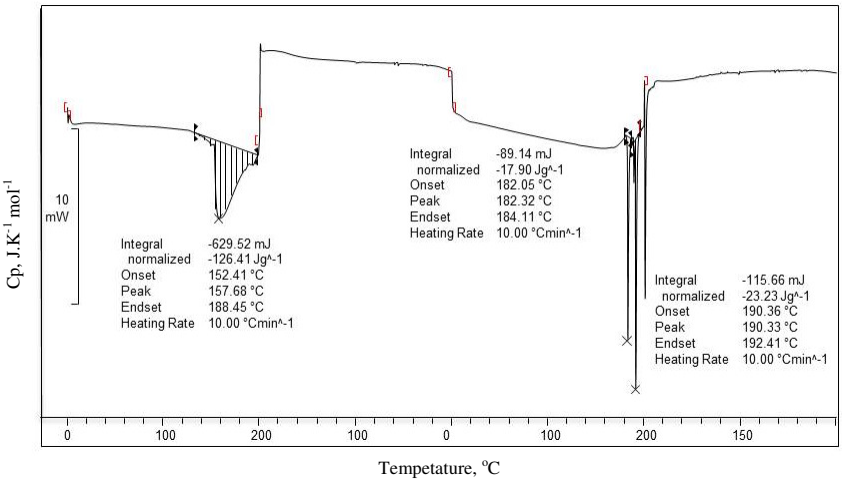

Figure 11. DSC spectrum of the surfactant 6'-dodecanamido-sucrose (**7b**)

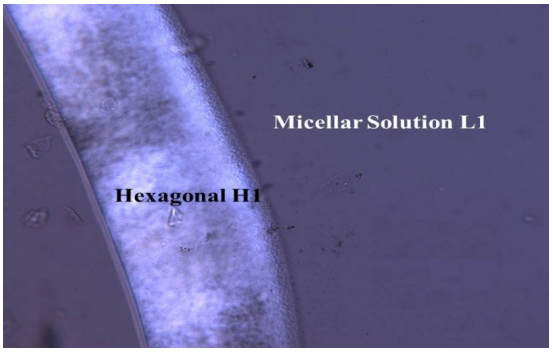

Figure 12. Lyotropic phase ofthe surfactant (**7b**)

The CMC of the C_{12} straight sucrose mono amide surfactants (**7b**) was determined at room temperature as 0.7 mmol/L (Figure 13). This value is in good agreement with other di-saccharide surfactants of similar chain length[24,25]. The correlated surface tension of 36 mN/m suggested good emulsifying ability. Although less than corresponding glycosides.

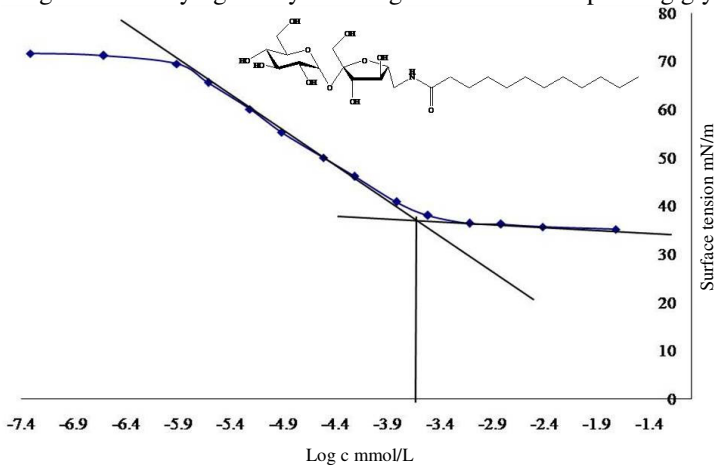

Figure 13. CMS investigation of the surfactant 6'-dodecanamido-sucrose **(7b)**

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