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## Synthesis and interfacial properties of new 6-sulfate sugar-based anionic surfactants

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### ABSTRACT

Three families of anionic sugar-based surfactants bearing a sulfate functional group on the primary position of a monosaccharide were synthesized and their physicochemical properties were compared. The first family corresponds to 6-sulfate derivatives of commercially available octa- and dodecyl  $\beta$ -D-gluco- and galactopyranosides. The second and the third families contain an amide linker between the sulfated monosaccharide (galactose, glucose or xylose) and the hydrophobic alkyl chain. Twelve of the as-synthesized anionic glycolipids, including nine novel sulfated compounds, were investigated for their surface activity at the air/liquid interface and for their self-assembling properties. These sugar-based surfactants show surface properties similar to those of commercial anionic surfactants (SDS and SLES) with good ability to reduce surface tension. The obtained results confirm the interest in these new bio-based molecules for potential substitution of anionic surfactants in various formulations.

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### 1. Introduction

Sugar-based surfactants are surface-active agents that are gaining more and more attention due to their valuable properties, making them interesting for a wide range of applications in industrial fields such as food, detergents and cosmetics [1]. They exhibit major advantages regarding their performance, relatively low cytotoxicity, higher biodegradability [2-4] and environmental compatibility [5] making them promising alternatives to petroleum-based surfactants [6].

Compared to other types of surfactants, anionic molecules are the most consumed on a worldwide scale because of their interesting properties and low cost [7]. Nevertheless, the use of these surfactants in cosmetics, detergents and toiletries is limited by their high skin and eye irritation potential [8]. The development of new anionic surfactants is an important priority in the field of colloid science. To the best of our knowledge, only a limited number of studies have reported the production of anionic surfactants from sugars and the characterization of their physicochemical properties. Among them, several articles have reported the synthesis and properties characterization of anionic sugar derivatives; these molecules showed good adsorption properties similar to other ionic surfactants [9].

For example, *N*-dodecyl-*N*-(3-propanoate)- $\beta$ -D-glucosylamine showed a synergetic effect with Ag-TiO<sub>2</sub> nanoparticles for enhanced oil recovery [10], as did the derivative with an

ethylsulfate group on the nitrogen atom of *N*-dodecyl- $\beta$ -D-glucosylamine [11].

The synthesis of sugar-based surfactants with phosphate groups as hydrophilic moieties, exhibiting surface activities superior to those of classical surfactants containing only one non-polar or hydrophobic moiety and one polar head unit, was also described [12]. As a representative example, dodecyl  $\beta$ -D-glucopyranoside-6-phosphate has been successfully used as a chiral selector for the enantiomeric separation of analytes and enantiomers in micellar electrokinetic capillary chromatography [13,14].

Surfactant synthesis involves the combination of a polar head and a hydrophilic part. A large number of studies have been focused on the understanding of relationships between the chemical structure of surfactants and their surface properties [15-17]. The main challenge remains their synthesis, in particular the connection of the hydrophilic group (carbohydrate) to the alkyl chain by a linker. Another challenge is to anticipate the trends between the sugar-based structure and their properties, to produce greener surfactants for specific applications. Sugar-based surfactants, comprising of glyco-amides, glyco-esters, glycosides and other derivatives can exhibit various physicochemical properties depending on their molecular structure.

In previous work [18], we synthesized *N*-octyl-glycosylamines (sugar-NH-CO-R) derived from glucose and maltose, 6-amino-deoxy-*N*-octanoyl-D-glucose (sugar-NH-CO-R) and *N*-octyl-D-

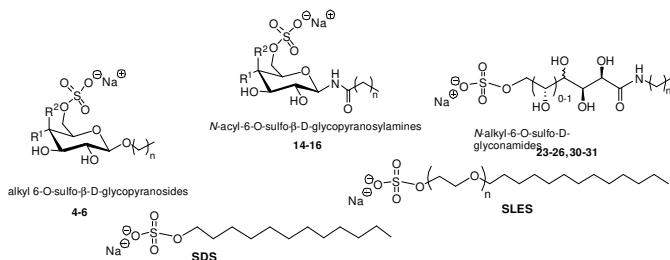
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glucuronamide (sugar-CO-NH-R). The surface-active properties of these nonionic glycolipids (bearing an amide linker) were compared to those of Tween®20 and Hecamed. We observed differences in their solubility and ability to form micelles as a function of temperature. They were also evaluated for their cytotoxicity on mouse fibroblasts with *N*-acyl-glycosylamines presenting the lowest cytotoxic effect. The results revealed significant differences in both the physicochemical properties and cytotoxic effects depending on structural features, such as the position of the linkage on the sugar head-group, or the orientation of the amide linkage.

According to the literature, introduction of an anionic site should enhance the water-solubility and detergent performance of the surfactant [19]. Herein, we have introduced a sulfate group at the primary position of the monosaccharides. Thanks to the enhanced solubility, we can extend the study to glyco-amide derivatives bearing a longer (dodecyl) alkyl chain in order to identify sugar-based surfactants which could be potential candidates for the replacement of widely used petroleum-based molecules such as sodium dodecyl sulfate (SDS), sodium laureth sulfate (SLES) or sodium dodecylbenzene sulfonate (SDBS).

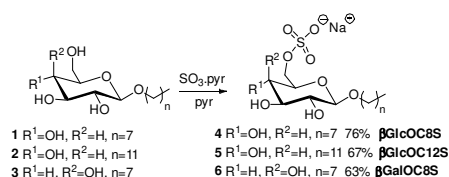
## 2. Results and Discussion

A library of sulfated glycolipids was prepared following three main structures: alkyl 6-*O*-sulfo-β-D-glycopyranosides, *N*-acyl-6-*O*-sulfo-β-D-glycopyranosylamines and *N*-alkyl-6-*O*-sulfo-D-glyconamides. The physicochemical properties of these anionic glycolipids were compared to those of two commercially available standard anionic surfactants, sodium laureth sulfate (SLES) and sodium dodecylsulfate (SDS) (Fig. 1).



**Figure 1.** General structures of sulfated glycolipids and commercially available surfactants studied.

First, sulfation of commercially available octyl β-D-glycopyranoside **1**, and octyl and dodecyl β-D-galactopyranoside **2** and **3** was carried out using 2 equivalents of SO<sub>3</sub>.pyr according to literature procedures [20]. After purification by reverse flash chromatography, the corresponding sulfated glycolipids **4-6** were obtained in 76%, 67% and 63% yield, respectively (Scheme 1). In each case, small amounts of disulfated glycolipids were observed by reverse phase TLC (disulfated compound R<sub>f</sub>=0.7, monosulfated compound R<sub>f</sub>= 0.3, H<sub>2</sub>O/CH<sub>3</sub>CN 6/4) and isolated as mixtures of inseparable isomers. In the <sup>1</sup>H NMR spectrum, for monosulfated compounds **4-6**, a downfield shift for the protons of the primary alcohol (4.15 ppm) was observed.

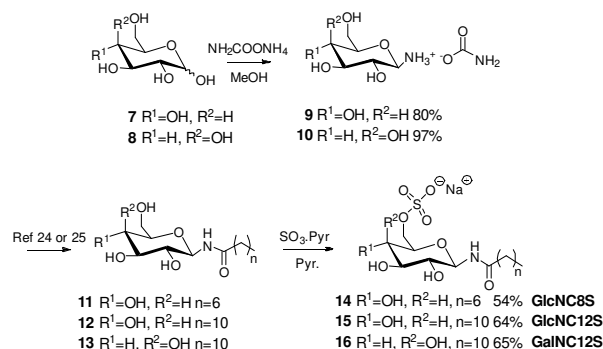


**Scheme 1.** Sulfation of alkyl D-glycopyranosides.

Some alkyl 6-*O*-sulfo-β-D-glycopyranosides have previously been reported in the literature. Compound **4** was studied as a potential inhibitor of trehalose 6-phosphate phosphatase, which is involved in microbial and nematodal human pathogens [21]. Compound **5** was reported as an effective chiral selector for micellar electrokinetic capillary chromatography resolution of a variety of enantiomeric mixtures [22]. Its low CMC (1 mM) and its high hydrosolubility are given as advantages for this application.

The second and third families of sulfated glycolipids involve an amide linker between the sugar and lipid moieties. Depending on the direction of this linker, cyclic (*N*-acyl-6-*O*-sulfo-β-D-glycopyranosylamines) or acyclic (*N*-alkyl-6-*O*-sulfo-D-glyconamides) structures were synthesized.

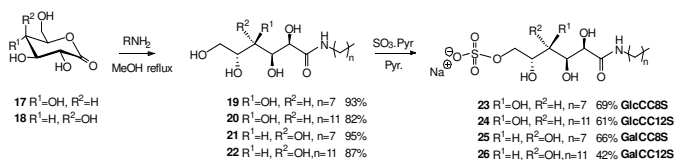
The preparation of the second family followed a three-step strategy, with the preparation of a glycosylamine carbamate salt intermediate [18,23] which was then treated with an acyl chloride and then sulfated at the primary position (Scheme 2).



**Scheme 2.** Synthesis of sulfated *N*-alkyl β-D-glycopyranosylamine compounds **14-16**.

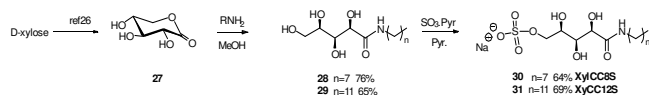
The carbamate salts of gluco- and galactosylamine were obtained in good to excellent yields (80-97%). According to the Plusquellec [24] or Lubineau [25] protocols, subsequent acylation gave the corresponding *N*-acyl β-D-glycopyranosylamines derivatives **11-13** which were directly engaged in the next reaction. As previously observed, the sulfation led to monosulfated compounds **14-16** as the major products in 54-65% yield. The formation of disulfated derivatives was also observed as mixture of isomers which have not been identified. The desired compounds **14-16** were easily purified by reverse phase flash chromatography. These compounds correspond to cyclic sugar derivatives in which the sugar is linked by an NH-CO bond to the alkyl chain.

The third family is designated as an acyclic sugar with a CO-NH-linkage between the sugar and the alkyl chain, in which the amide carbonyl is the anomeric carbon of the sugar. First, nucleophilic addition of the alkylamine was carried out on commercially available glucono- and galactono-1,5-lactone. The glyconolactone solubilized in methanol (0.42 M) was stirred in the presence of 1.1 equivalents of octyl- or decylamine until disappearance of the starting material, giving the corresponding *N*-(alkyl)-D-glyconamide that precipitated from solution and was separated by filtration. These acyclic glyconamides **19-22**, obtained in 82-95% yield, were then sulfated following the same protocol used for the glycoside and glycosylamine derivatives. The corresponding 6-*O*-sulfate compounds were obtained in 42-69% isolated yield. As previously observed, the disulfate mixtures were separated from the monosulfated compounds by reverse flash chromatography.



**Scheme 3.** Synthesis of sulfated *N*-octyl and *N*-dodecyl-D-gluco- and D-galactonamides derivatives **23-26**.

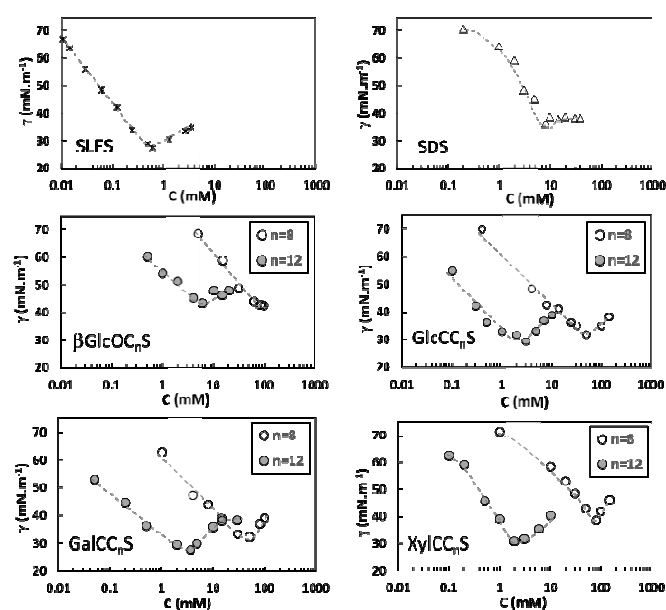
For the synthesis of xylonamide derivatives **30** and **31**, a prior oxidation of xylose was carried out in the presence of hydrogen peroxide catalyzed by gold nanoparticles supported on alumina under microwave irradiation, as previously described for the synthesis of glycobionate sodium salts [26]. These conditions gave a good conversion of xylose into sodium xylonate (up to 85%), and a moderate yield for isolated xylonolactone (51%). The basic conditions used for the oxidation step catalyzed by gold nanoparticles require subsequent treatment with an acidic resin (Amberlite IR120 H<sup>+</sup>). Complete conversion of sodium xylonate into its lactone form is difficult to accomplish, and a mixture of xylonic acid and xylonolactone was obtained, thus giving a moderate yield for lactone **27** [27].



**Scheme 4.** Synthesis of sulfated *N*-octyl and *N*-dodecyl-D-xylonamides derivatives **30** and **31**.

The aminolysis of **27** with octylamine and dodecylamine in methanol proceeded at reflux to afford xylonamides **28** and **29** in 76% and 65% yield, respectively, which were sulfated following the general sulfation procedure to afford acyclic 6-sulfate xylonamides **30** and **31** in 64% and 69% yield, respectively (Scheme 4).

All of the sulfated compounds were evaluated for their interfacial properties (described in the ESI). The surface-active data are summarized in Table 1.



**Figure 2.** Representative curves of surface tension vs. concentration for selected anionic glycolipids. Dotted lines are drawn as a visual guide only.

Each point represents the mean of three measurements. Error bars are smaller than the plot symbols.

The most important applications of surfactants are related to their surface-activity and self-organization in solution. Knowledge of the adsorption properties allows evaluation of the efficiency and performance of the surfactant system. The higher the level of surfactant performance, the lower the amount that is required and, consequently, the lower the environmental and economic impact produced. Detergents with low CMC, low  $\gamma_{\text{CMC}}$  and strong efficacy in decreasing surface tension ( $pC_{20}$ ) are often considered as suitable for different applications.

The evolution of equilibrium air/liquid surface tension with concentration of the studied anionic glycolipids is presented in Figure 2. A minimum was observed at the break point of surface tension-concentration curve for all anionic glycolipids. Usually, the minimum in surface tension near CMC is related to the presence of a small amount of residual impurities with amphiphilic properties participating in the surface tension reduction. For example, this particularity is often noticed for SDS because of the presence of residual dodecanol [28,29]. However, some authors claim that the local minimum of the surface tension is mainly related to the local maximum of the chemical potential, and does not necessarily indicate the presence or absence of impurities [30].

By comparing the obtained curves for four families of surfactants (sugar-based surfactants and SDS/SLES), it was observed that the minimum at the break point, corresponding to self-assembling of molecules, for surfactants with a shorter alkyl chain (C8) occurs at significantly higher concentration compared to surfactants with a long chain (C12). This observation can be explained by the stronger contribution of the hydrophobic forces occurring during micellization. Surfactants with a longer hydrophobic chain can self-assemble to form micelles easier and at lower concentration. This behavior is in agreement with results usually observed for conventional surfactants [31-34]. Overall, the CMC values of glycolipids with a C8 chain were between 25.3 and 75.5 mM, whereas for glycolipids with a C12 chain, the CMC varied from 2.1 to 10.2 mM. The CMC of **14** GlcNC8S could not be determined because of its high value and its limited quantity (Table 1). It can also be noted that the ratio of  $\text{CMC}(\text{C8})/\text{CMC}(\text{C12})$  for different families of surfactants, except XylCCnS, were of the same order of magnitude, between compounds **11** and **16**. This constant ratio indicates the systematic and significant effect of the linear alkyl chain length on the surface-active properties of the synthesized surfactants, regardless of the structure of the hydrophilic sugar part. This is comparable to the values of commercially available anionic surfactants: CMC (sodium octyl sulfate, SOS)/CMC (sodium dodecyl sulfate, SDS) is 15-16 [35].

The data related to the surface tension at the CMC ( $\gamma_{\text{CMC}}$ ) and the efficiency of surfactant adsorption ( $pC_{20}$ ) indicate that anionic glycolipids have high efficiency in lowering the surface tension of water comparable to commercially available molecules (Table 1). Surfactants with a long alkyl chain possess higher ability for the reduction of surface tension of aqueous solutions (higher  $pC_{20}$  values) compared to those with a short alkyl chain. This assumes that the energy needed to bring C12 surfactant molecule from the bulk to the air/water interface is lower. As can be seen, for glycolipids with the same hydrophobic chain length, the  $\gamma_{\text{CMC}}$  varied noticeably. For example, this minimum surface tension changes from 28  $\text{mN}\cdot\text{m}^{-1}$  (**16**: GalNC12S) to 43  $\text{mN}\cdot\text{m}^{-1}$  (**5**: GlcOC12S) for C12 surfactants. This variation is undoubtedly related to the structure of the polar part of the glycolipid (cyclic or open chain sugar) and the nature of the linker between the hydrophilic and hydrophobic parts. In fact, the change in

flexibility of the hydrophilic part can lead to various molecular configurations of the surfactant. The structure of the surfactant layer adsorbed at the air/water interface as well as the surface properties is also influenced [36].

If we compare the properties of glycolipids with C12 anionic commercial surfactants, SLES shows the highest efficacy characterized by the lowest CMC. However, several glycolipids bearing a C12 chain (**5**:  $\beta$ GlcOC12S, **24**: GlcCC12S, **26**: GalCC12S, **31**: XylCC12S) possess lower CMC values than SDS. Furthermore, according to the literature, the CMC of sodium octyl sulfate (SOS) is equal to 120 mM [37] which is significantly higher than the CMC values of anionic glycolipids bearing a C8 chain synthesized in this study. Regarding the surface tension reduction ( $\gamma_{\text{CMC}}$ ) surfactants **15** (GlcNC12S), **24** (GlcCC12S) and **31** (XylCC12S) have similar performance to SDS, whereas **15** (GlcNC12S) and **31** (XylCC12S) possess equivalent performance to SLES. The data show that for studied sugar-based anionic surfactants with a C12 alkyl chain (**5**: GlcOC12S, **15**: GlcNC12S, **16**: GalNC12S, **24**: GlcCC12S, **26**: GalCC12S, **31**: XylCC12S), the  $pC_{20}$  values are between those of commercial anionic molecules with the same chain length (SLES and SDS).

In conclusion, three sugar-based surfactants families, including nine new sulfated compounds, were synthesized in one or three steps and then compared with commercially available surfactants. The synthesized sugar-based anionic surfactants were found to exhibit interesting surface adsorption properties comparable to those of commercially available anionic surfactants. As expected, the CMC values decreased with the increase in alkyl chain length. Glycolipids with a longer chain (C12) were characterized by their higher ability to reduce the surface tension. In addition, the functional properties of three anionic glycolipids (**15**: GlcNC12S, **24**: GlcCC12S and **31**: XylCC12S) were described in our recent study [38]. These surfactants have good foaming properties comparable with commercial anionic molecules, and low irritant effect on a human skin model. These promising results highlight the interest in the development and characterization of anionic sugar-based amphiphiles as potential candidates for the substitution of petroleum based anionic surfactants in cosmetic and personal care formulations. Finally, these findings indicate a direction for future studies, which will focus on the characterization of mono- and disulfate mixtures, avoiding the separation step during synthesis.

**Table 1.** Physical parameters of surface activity for the studied sugar-based and commercially available reference surfactants SDS and SLES at 25 °C (except for **25** GalCC8S and **26** GalCC12S which were measured at 50 °C).

Compounds	Mw (g.mol <sup>-1</sup> )	CMC (mM) <sup>*</sup>	$\gamma_{\text{CMC}}$ (mN.m <sup>-1</sup> ) <sup>*</sup>	$pC_{20}$ <sup>*</sup>
SDS	288	8.0 ± 0.5	35 ± 1.3	2.52 ± 0.02
SLES	370	0.5 ± 0.15	27 ± 1.8	4.40 ± 0.01
<b>4</b> GlcOC8S	396.4	45.2 ± 2.5	42 ± 1.5	1.73 ± 0.1
<b>5</b> GlcOC12S**	452.5	4.1 ± 0.4	43 ± 2.5	2.75 ± 0.03
<b>6</b> GalOC8S	396.4	25.3 ± 1.8	37 ± 1.7	2.27 ± 0.05
<b>14</b> GlcNC8S	407.4	>70	nd	nd
<b>15</b> GlcNC12S	463.5	10.0 ± 0.8	29 ± 1.2	2.89 ± 0.02
<b>16</b> GalNC12S	463.5	9.0 ± 0.6	28 ± 1.2	3.21 ± 0.02
<b>23</b> GlcCC8S	409.4	50.4 ± 2.3	40 ± 1.9	2.52 ± 0.05
<b>24</b> GlcCC12S	465.5	3.0 ± 0.4	32 ± 2.8	3.92 ± 0.05
<b>25</b> GalCC8S	409.4	43.2 ± 3.0	40 ± 2.1	2.55 ± 0.03
<b>26</b> GalCC12S	465.5	3.5 ± 0.3	38 ± 1.7	4.3 ± 0.04
<b>30</b> XylCC8S	379.4	75.5 ± 2.8	39 ± 1.7	1.82 ± 0.07
<b>31</b> XylCC12S	436.5	2.0 ± 0.3	31 ± 2.2	3.43 ± 0.06

\* standard deviation of each value corresponds to the precision of the graphical determination of the physicochemical parameter

\*\* values in reference [20] are 11.1 mM for CMC, and 34.5 mN.m<sup>-1</sup> for  $\gamma_{\text{CMC}}$

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## Graphical Abstract

### Synthesis and interfacial properties of new 6-sulfate sugar-based anionic surfactants

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