Synthesis and Properties of *N*-(3-Oxapropanoxyl)dodecanamide

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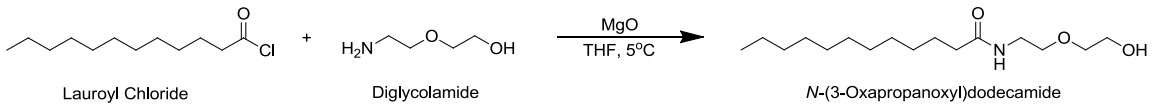
Introduction

Materials and Methods

The novel detergent, *N*-(3-Oxapropanoxyl)dodecanamide (NOPD), is synthesized via the reaction of lauroyl chloride and diglycolamide (DGA) in the presence of magnesium oxide. The solvent used is tetrahydrofuran (THF) and the mixture is allowed to react at low temperature for several hours (Scheme 1). A more detailed description of the synthesis as well as spectroscopic characterization of the pure product are provided in supporting information.

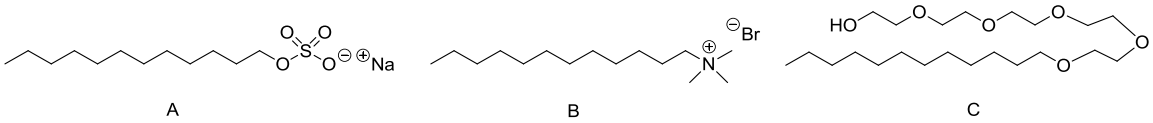


Scheme 1. Outline of the synthesis of *N*-(3-Oxapropanoxyl)dodecanamide, NOPD.

The usefulness of NOPD in regards to surfactant-polymer flooding is based on the surfactant's ability to form micelles. The critical micelle concentration (CMC) is found as the concentration of surfactant above which micelles form. The molecule's effectiveness in reducing the surface tension of water is characterized by the value of the surface tension of the solution at the CMC. This surface tension γ_{CMC} is measured using the du Noüy ring method. With this technique a platinum ring is suspended in solution and the force is measured which is required for the raised ring to break the surface of the liquid.[1] Using the Szyszkowski equation, , and the surface

tension measurements the saturated adsorption ( ) and adsorption coefficient ( ) can be calculated. Simple multiplication gives the cross section area of the air-water interface (a^{∞}) corresponding to each value of .

To better understand the relative effectiveness of NOPD, these tests were performed on additional, common surfactants with 12-carbon non-polar tails analogous to that of NOPD. The comparative surfactants used were anionic sodium dodecyl sulfate (SDS), cationic dodecyl-trimethyl-ammonium bromide (DTAB), and nonionic dodecylpentaglycol ($C_{12}E_5$). The structures of these three surfactants are shown in Scheme 2 and the inclusive results from the performance tests and calculations are reported in Table 1.



Scheme 2. Surfactants Used for Comparison. A: SDS; B: DTAB; and C: $C_{12}E_5$

Table 1. Surface Properties of NOPD and Three Comparable Detergents

	CMC (M)	$\boldsymbol{\gamma_{CMC}}$ (nM/m)	$\boldsymbol{\Gamma^{\infty}}$ (mol/cm^2)	$\mathbf{a}^{\infty}$ (nm^2/mol)	**K** (M^{-1})
NOPD	1.5×10^{-4}	25.0	4.79×10^{-10}	0.35	2.21×10^{-5}
SDS	6.0×10^{-3}	36.6	3.18×10^{-10}	0.52	1.24×10^{-6}
DTAB	1.6×10^{-2}	38.7	3.20×10^{-10}	0.52	6.57×10^{-2}
$C_{12}E_5$	6.0×10^{-5}	30.4	3.20×10^{-10}	0.52	3.65×10^{-6}

Results and Discussion

Conclusion

Supplemental Material Available: A detailed description of the synthesis of *N*-(3-oxapropanoxyl)dodecanamide and details of its spectroscopic characterization (MS, IR, NMR).

References

(1) Cui, Z.; Yang, L.; Cui, Y.; Binks, B. P. Effects of Surfactant Structure on the Phase Inversion of Emulsions Stabilized by Mixtures of Silica Nanoparticles and Cationic Surfactant. *Langmuir* **2010,** *26,* 4717-4724.

Supporting Information

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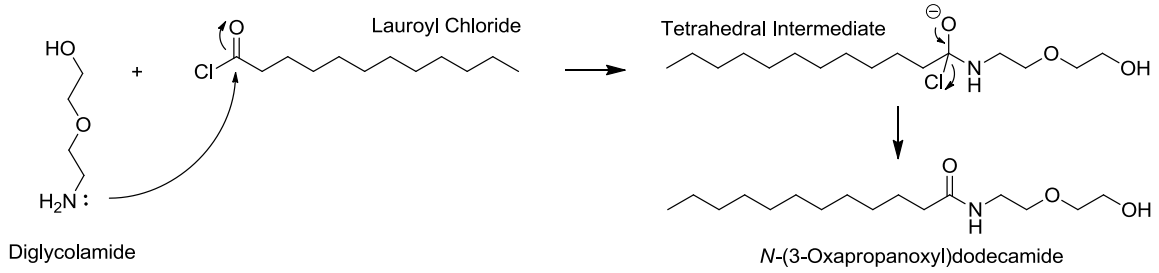
Synthesis of *N*-(3-Oxapropanoxyl)dodecanamide

The I-(3-Oxapropanoxyl)dodecanamide was produced by reacting lauroyl chloride with diglycolamine (DGA) while magnesium oxide (MgO) was present. The exact synthesis consisted of the following: 4.41g (42 mmol) of DGA was placed into a 250ml 3-neck flask. Then, 35 ml of deionized water, 8.00g (200 mmol) MgO, and 105 mL of THF at 5°C was added to the flask. The mixture was allowed to stir for 30 min while cooling in an ice bath of 5 °C. Next, a solution of 8.74 g (40 mmol) lauroyl chloride in 35 mL THF was added dropwise over 1 hour with the use of a constant pressure funnel all while stirring at 5 °C for a total of 2-2.5 hours. The MgO was then removed with filtration and the purified product was obtained by evaporating the filtrate in a vacuum to remove the THF. Pure water was then used to wash the product and it was dried at 50 °C in a vacuum. This yielded a pure product in the form of a white powder. The mechanism for this synthesis is the nucleophilic acyl substitution of an acyl halide with a primary amine (Scheme S3).



Scheme S3. Mechanism of Formation of *N*-(3-Oxapropanoxyl)dodecanamide

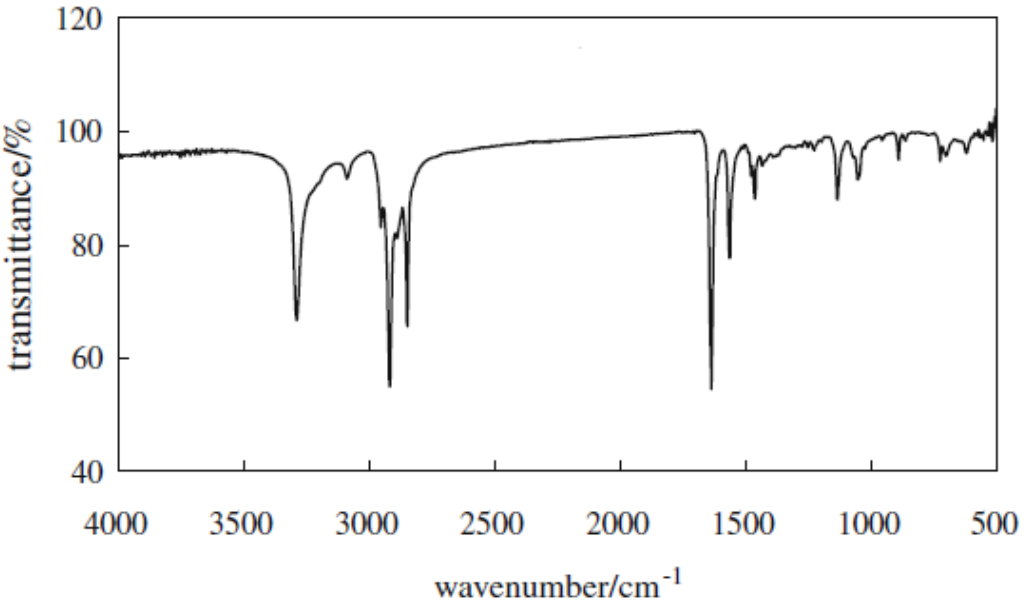


Figure S1. IR spectrum of *N*-(3-oxapropanoxyl)dodecanamide.

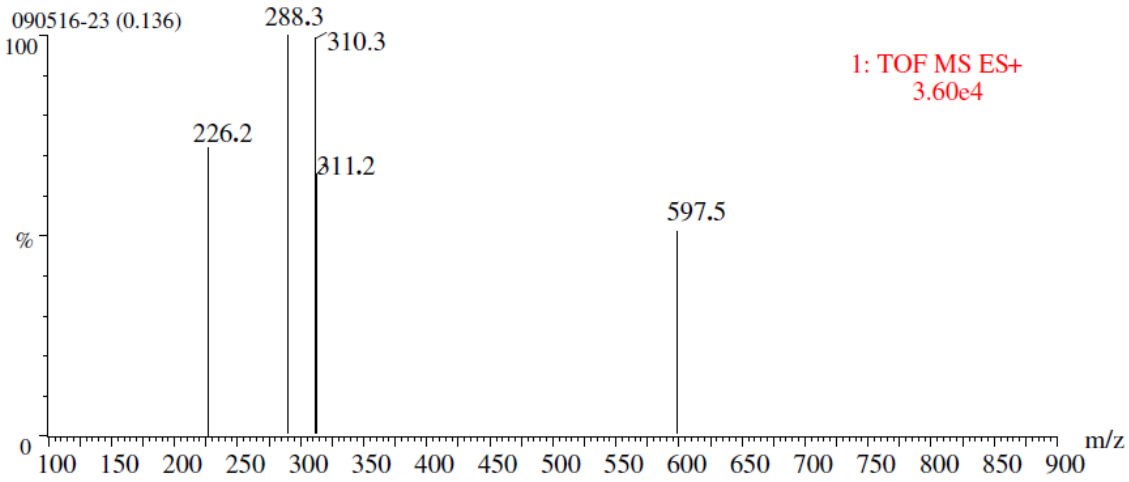


Figure S2. ESI-MS spectrum of *N*-(3-oxapropanoxyl)dodecanamide.

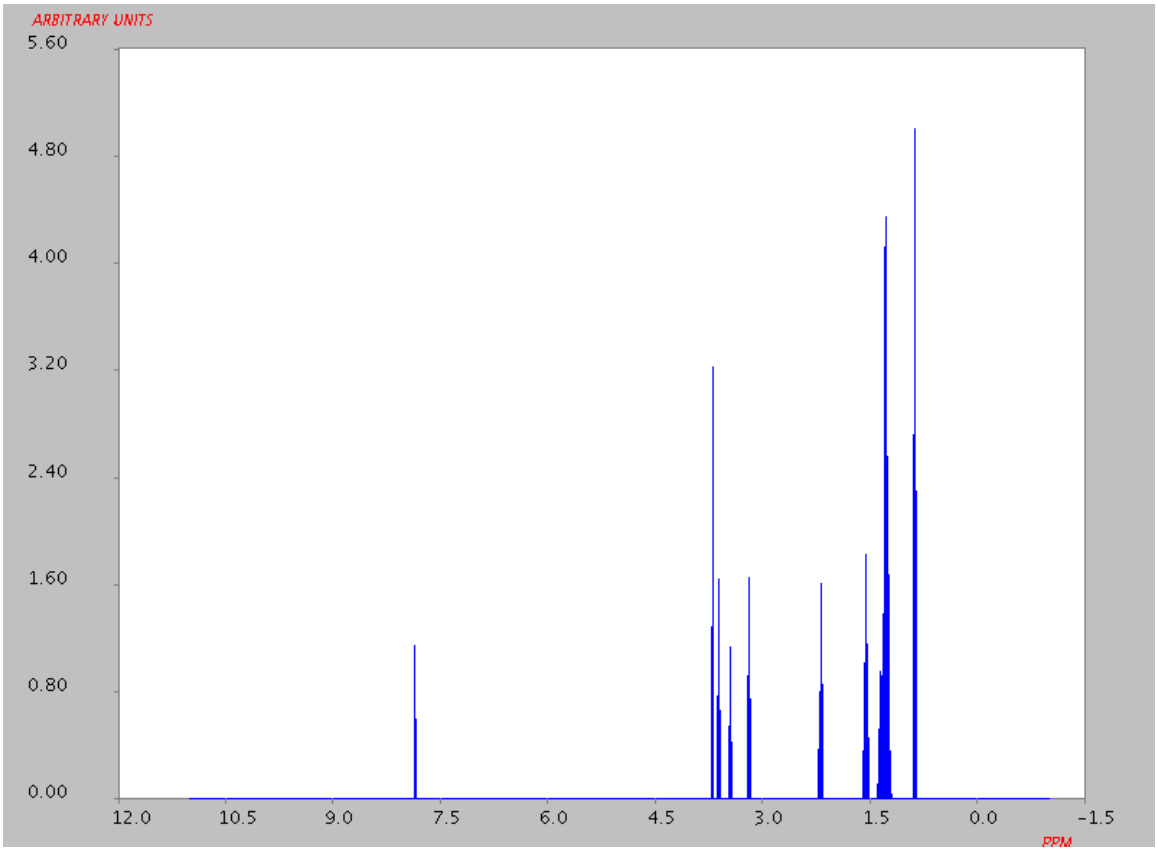


Figure S3. Proton NMR spectrum of *N*-(3-oxapropanoxyl)dodecanamide at 400MHz.

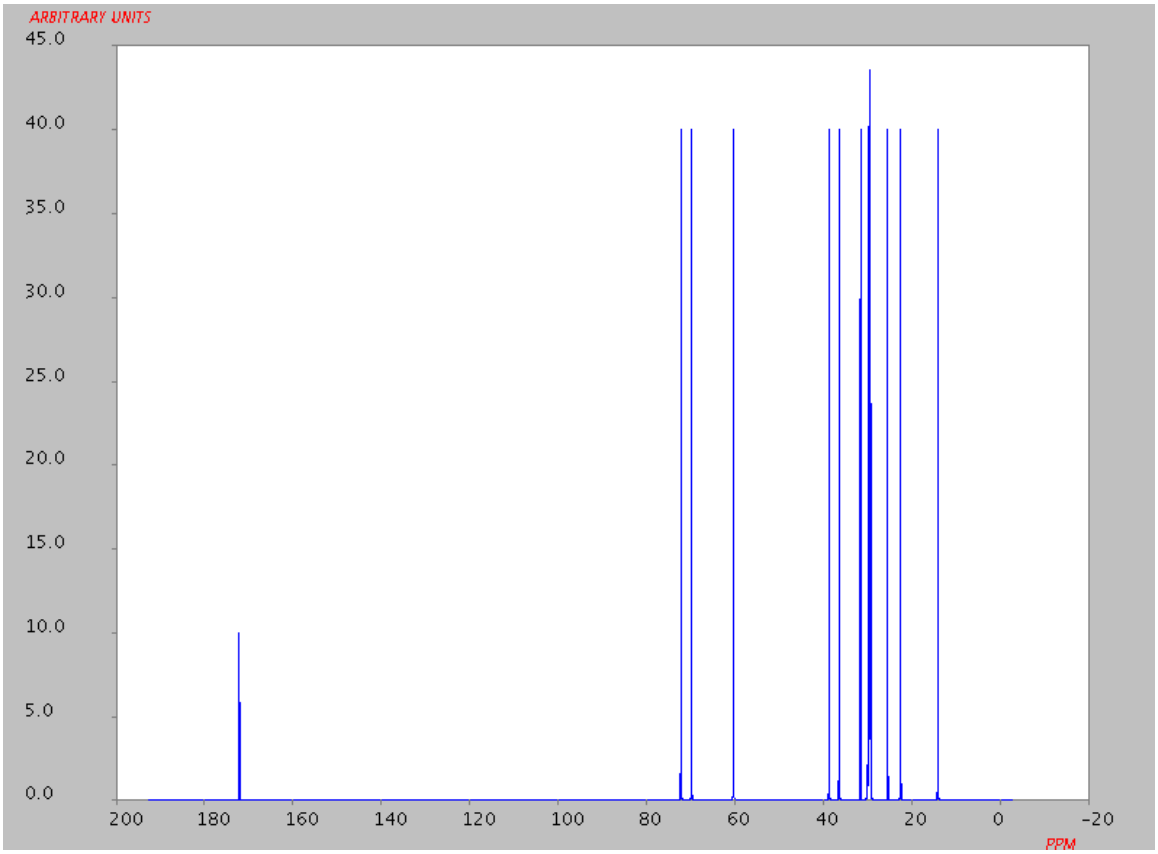


Figure S4. ^{13}Carbon NMR spectrum of *N*-(3-oxapropanoxyl)dodecanamide in Tetramethylsilane.

Bibliography

1 Cui, Z.; Song, H.; Yu, J.; Wang, F. Synthesis of *N*-(3-Oxapropanoxyl)dodecnamide and its Application in Surfactant-Polymer Flooding. *J. Surfact Deterg* **2011**, *14*, 317-324.

2 *SciFinder*; Chemical Abstracts Service: Columbus, OH; Proton NMR Spectrum; spectrum ID 20138287-HNMR; RN 20138-28-7; https://scifinder.cas.org (accessed April 2, 2012).

3 *SciFinder*; Chemical Abstracts Service: Columbus, OH; Carbon-13 NMR Spectrum; spectrum ID 20138287-CNMR; RN 20138-28-7; https://scifinder.cas.org (accessed April 2, 2012).