# Experimental report

08/01/2021

**Proposal:** 9-10-1613 **Council:** 4/2019

**Title:** SANS studies of micellar structures from acyl-L-carnitines and their mixtures under different ratios pH

**Research area:** Soft condensed matter

**This proposal is a new proposal**


**Main proposer:** Jian Ren LU

**Experimental team:** Huayang LIU
Zongyi LI
Ke FA
Peter HOLLOWELL

**Local contacts:** Ralf SCHWEINS


**Samples:** H2O
D2O
L-carnitine esters (hydrogenated / deuterated)
(CH3)3N+-CH2-CHOCO-R-CH2-COO-R (R=C12-16)

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D11 | 2 | 1 | 16/02/2020 | 17/02/2020 |
| D33 | 2 | 0 | | |


**Abstract:**

Acyl L-Carnitines are a group of novel surfactants with attractive physical and biological properties associated with their head group charge features. This work proposes to use SANS as the unique technique to determine the size and shape of surfactant micelles as a function of surfactant concentration and the subsequent changes upon pH and ionic strength. The aim is to understand how changes in head group charge affects micellar size, shape and charge characteristics. We request 2 days of D11 or D33 beamtime to complete the proposed measurements.

# Experimental report

03/2020

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|---|---|
| **Proposal:** | **9-10-1613** |
| **Council:** | |
| **Title:** | SANS studies of micellar structures from acyl-L-carnitines and their mixtures under different ratios and pH |
| **Research area:** | Biosurfactants |


| | |
|---|---|
| **Main proposer:** | **Jian Ren LU** |
| **Experimental team:** | Huayang LIU |
| | Zongyi LI |
| | Ke FA |
| **Local contacts:** | Ralf SCHWEINS |


**Samples:** Acyl-L-carnitines

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| D11 | 2 | 1 | 16/02/2020 | 17/02/2020 |
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proposed measurements.

## Report of experiment – D11 9-10-1613

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## PhD Project: Study the fundamental properties of acyl-L-carnitines


## Huayang LIU


The purpose of this experiment is to study the aggregation of acyl-L-carnitines in water under different concentrations, ratios and pH. We use the D11 to measure the different contrasts and fit them to have the structural details of aggregation of acyl-L-carnitines.

Materials / samples:

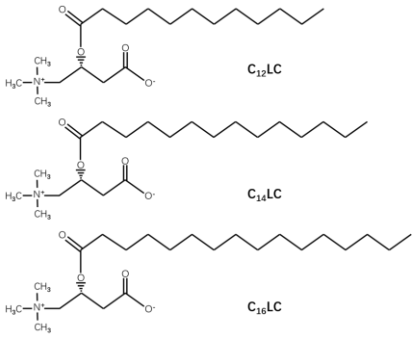


Figure 1 Chemical structure of acyl-L-carnitines

The structure of acyl-L-carnitines are shown in Figure 1. The surfactants have been purified by crystallization method to remove acid and salt, so that the samples have very high purity. The sample powder is dissolved in water with different concentrations, and the pH is adjusted with HCL solution. Then NaCl solution is added to control the ionic strength. Besides, three contrasts are measured in this experiment: h-acyl-L-carnitines are dissolved in $H_2O$, d-acyl-L-carnitines are dissolved in $D_2O$. The ready samples are injected to 1 or 2mm banjor cells and measured at 25℃.

Experiment and results

Firstly, 20mM $C_{12}LC$, $C_{14}LC$ and $C_{16}LC$ were measured, $C_{12}LC$ and $C_{14}LC$ were measured at 25℃ and $C_{16}LC$ was measured at 40℃ because of its solubility is low at 25℃. H sample in $D_2O$ was measured for 15mins, D sample in $H_2O$ is measured for 15mins and D sample in $D_2O$ was measured for 35mins.

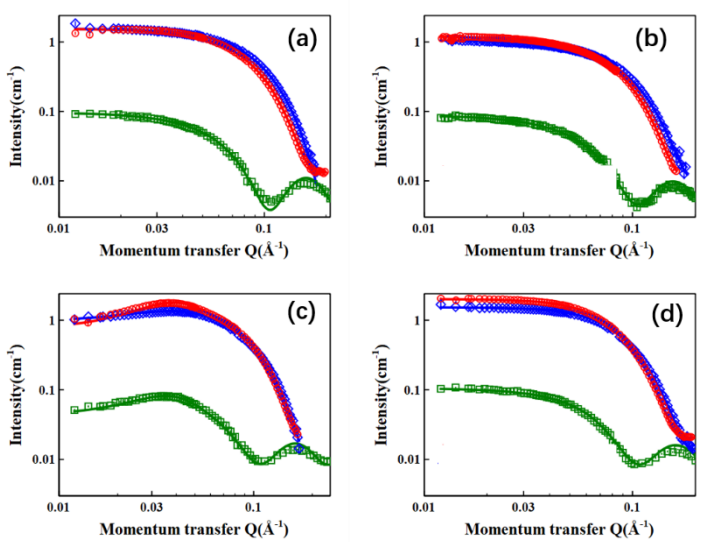


Figure 2SANS profiles of $C_{16}LC$ in water for 3 contrasts at pH7 with 10mM ionic strength (a), pH7 with 160mM ionic strength (b), pH2 with 10mM ionic strength (c), pH2 with 160mM ionic strength (d).

Figure 2 are some SANS data profiles for $C_{16}LC$(20mM, 40°C), and the red is h-$C_{16}LC$ in $D_2O$, blue is d-$C_{16}LC$ in $H_2O$ and green is d-$C_{16}LC$ in $D_2O$. SASVIEW 4.2 is used to fit the data. And the SLD values of different parts are shown in Table 1. Table 1 gives the protonated tails have a value of -0.4 ,while the deuterated tails have a value of 7. The best fitting model is core-shell spherical model. It shows that the micelles of $C_{16}LC$ have a core radius of 20 Å and thickness of 12 Å. At pH7, the ionic strength has no effect on the structure of micelles of acyl-L-carnitines. When the pH decreased to pH 2, as Figure (c) shown, there is a decrease of intensity in low q range. This means that there is a strong interaction between micelles under water. As the chemical structure shown in Figure 1, when pH equals to 2, strong acidic environment will lead to protonated carboxylic group. As a result, the zwitterionic acyl-L-carnitines will change into cationic surfactant. So the micelles will carry positive charges and have strong interaction between each other. The charge number is around 20. At this pH, when the ionic strength is increased to 160mM, as the Figure(d) shows, the interaction is weakened. However, pH and ionic strength have little effect on the size of micelles of acyl-L-carnitines.

| Segments | Scattering length density/ρ(Å$^{-2}$ ×10$^{-6}$) |
|---|---|
| Protonanted tail ($C_{11}H_{23}$) | -0.40 |
| Deuterated tail ($C_{11}D_{23}$, 98%D) | 7.01 |
| Protonanted tail ($C_{13}H_{27}$) | -0.39 |
| Deuterated tail ($C_{13}D_{27}$, 98%D) | 7.03 |
| Protonanted tail ($C_{15}H_{31}$) | -0.37 |
| Deuterated tail ($C_{15}D_{31}$, 98%D) | 7.00 |
| Head(hL-carnitine with -C=O) | 1.31 |
| $H_2O$ | -0.56 |
| $D_2O$ | 6.35 |

Table 1 The SLD of different segments used in fitting SANS data

Conclusion

The micelles of acyl-L-carnitines have a core-shell spherical structure. The core radius increase with the acyl chain length, the values are 16, 18 and 20 Å, respectively. The thickness is around 11 Å. The pH and ionic strength have little effect on the size of micelles, but the low pH environment will lead to strong interaction between cationic micelles, however, high ionic strength could screen and weaken this interaction.