EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Structural study of nonlamellar nanostructures from algal biomass to encapsulate natural antioxidants	**Experiment number**: SC/4992
Beamline: ID02	**Date of experiment**: from: 18-09-2020 to: 20-09-2020	**Date of report**: 12-03-2021
Shifts: 6	**Local contact(s)**: Michael Sztucki, Narayanan Theyencheri	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Ilaria Clemente, University of Siena and University of Florence

Dr Sandra Ristori, University of Florence

Report:

The experiment was carried out remotely in continuos collaboration with the beamline staff, that we thankfully acknowledge. All the procedures carried out to send samples, obtain data and receive back the samples went by smoothly. Our investigation included a total number of 104 samples, measured at two or three sample-to-detector distances either in flow-through or regular capillaries. This beamtime allowed us to investigate novel nanovectors obtained by extracting lipids from dry algae biomass and loaded with natural hydrophobic antioxidants of widespread commercial interest, i.e. curcumin, piperine and tocopherol (Vitamin E). Such nanosystems thus obtained usually have complex supramolecular arrangements,so that in-depth structural and morphological studies are needed to formulate robust models. Indeed, we firstly measured the complex nonlamellar systems obtained by this route, then we measured a lamellar systems series obtained form algal material treated with different growth conditions and compared the two series of nanosystems in a complex-to-simple progression. Lastly, our interest lied in the de-structuring and eventual structural rearrangement of the nanocarriers measured at various times during a controlled disruption and release experiment, that we conducted previously in our lab. The aliquots taken from the samples at different time points during such experiment were then sent together with the other samples in their regular state. Such measurement allowed us to assess the structural stability of the nanovectors in hostile pH and temperature conditions, and to complement previous information obtained through spectroscopic techniques.

Moreover, the comparison of such data among the two series of nanostructures evidenced which types of lipids in the mixture mainly contribute to the assembly in certain phases. This information was then also combined with the role played by type, concentration and localization of cargo bioactive compounds in the structuring. Two exemplificative 1D intensity plots obtained are shown below for complex nonlamellar samples loaded with different cargos (Figure 1) and lamellar samples loaded with the same molecules and concentrations (Figure 2). A paper is in progress using the data collected in this SAXS experiments.

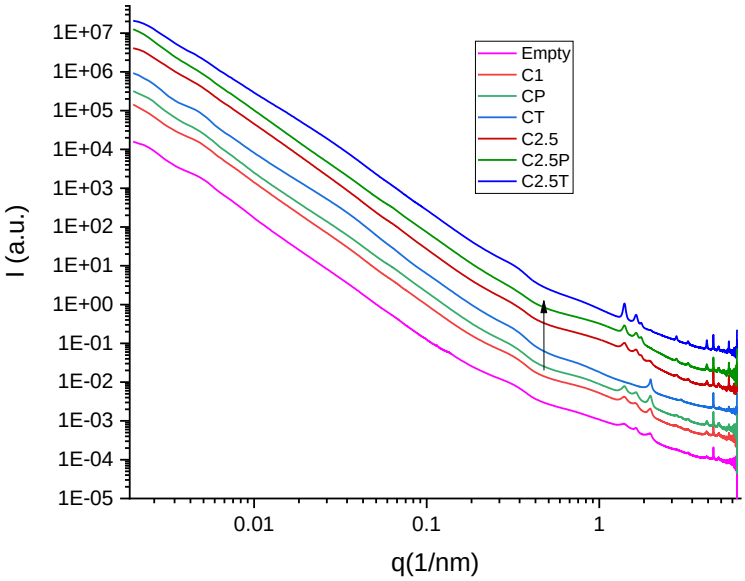

Figure 1. Nonlamellar nanocarriers loaded with the three adjuvants at various concentrations

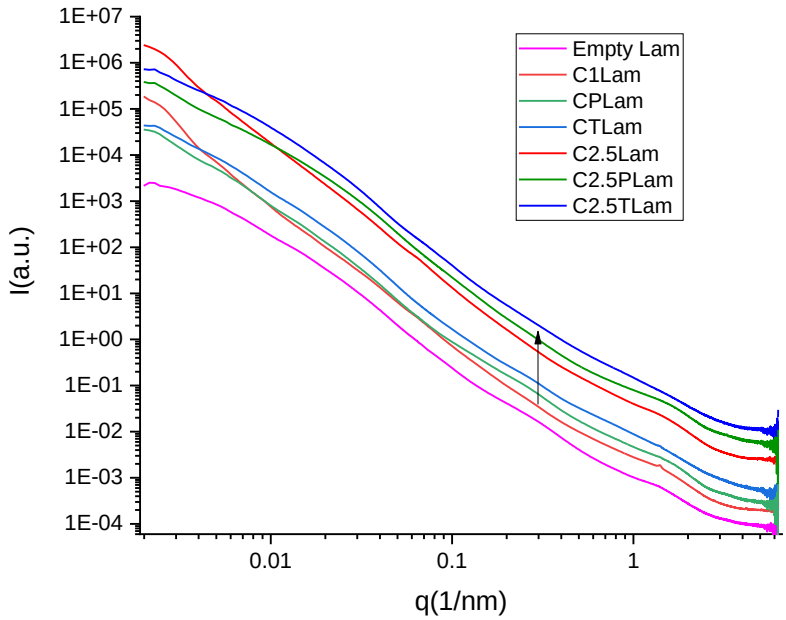

Figure 2. Lamellar nanocarriers loaded with the three adjuvants at various concentrations