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:** Lignin nanocapsules for delivering natural antimicrobial agents	**Experiment number**: SC 5179
Beamline: ID 02	**Date of experiment**: from: 07/12/21 to: 10/12/21	**Date of report**: 22/09/23
Shifts: 6	**Local contact(s)**: Michael Sztucki	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Sara Falsini, Dept. of Biology, University of Florence

Dr Sandra Ristori, Dept. Of Chemistry "Ugo Schiff" and CSGI, University of Florence

Report:

Polymers from natural biomasses, and particularly lignin, have emerged as a green alternative with diverse applications, including large-scale delivery of bioactives for agricultural purposes. Lignin, a renewable biopolymer abundant in plant cell walls and represents a byproduct of paper industry, can be harnessed to create nanocapsules suitable for this goal. Lignin-based nanocapsules (NCs) offer a sustainable and environmentally friendly way to encapsulate bioactive compounds, particularly molecules endowed with antimicrobial properties such as capsaicin.

We prepared lignin NCs at two different starting concentrations (1% and 5% w/w lignin), filled with either olive oil or neem oil, both known for their antioxidant properties, and we loaded them with capsaicin at three different concentrations (1, 3, 6 mg/mL). Two pH values were tested (11 and 13) and different sonication conditions to assess the impact of these parameters on the supramolecular structure and stability of the obtained empty and loaded NCs. We performed advanced structural characterization of the supramolecular arrangement by high-resolution techniques, such as synchrotron SAXS and USAXS, covering a wide q-range, i.e. $3x10^{-4}$ Å $^{-1}$- 0.7 Å $^{-1}$, which allowed to explore the different size populations in our samples. Particularly, two populations were evidenced from complementary techniques (DLS, SEM) in the range 50-100 nm and 500-600 nm. Data analysis showed that the pH influenced the overall size and stability of the samples, giving a bimodal distribution of sizes and a form factore typical of spherical objects for samples at higher pH; moreover, NCs showed to be quite flexible and to be able to encapsulate different amounts of capsaicin at high encapsulation efficiency without changing dramatically their overall structure.

These data complemented by other techniques, allowed us to finely investigate such complex samples and, we believe, to meaningfully contribute on the study of natural-derived unconventional nanovectors for sustainable delivery in agriculture.