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 investigation of the double network formation of a modified polypeptide derivatives"**	**Experiment number**: 26-02-900
Beamline: BM26B	**Date of experiment**: from: September 3rd 2018 to: September 5th 2018	**Date of report**: June 24th 2019
Shifts: 6	**Local contact(s)**: Dr. Michela Brunelli	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Daniel Hermida Merino – Dubble/ NWO

Bing Wu* – Dubble/Royal College of Surgeons in Ireland

Report:

Consisting of three-dimensional hydrophilic polymeric network, hydrogel has been the focus of recent biomaterial innovation due to its great potential in various biomedical applications. However, since most hydrogels contain a large amount of the water, their mechanical properties are often inferior to other traditional polymeric materials, which unavoidably limit their applicabilities in the field like tissue engineering. One way to circumvent this problem is to use DN systems, which significantly improve the mechanical performance of these hydrogels. Since the invention of DN in 2003, several DN systems has been designed and tested, however, majority of them either has rather lower biocompatibility or is completely based on natural polymers which is difficult to be modified. Hence it is necessary to design a new synthetic DN with modifiable chemical properties. In this study, a novel polypeptide-based bio-compatible DN hydrogels with enhanced toughness. The first network is based on a polypeptide (PP) synthesized from NCA polymerization. Through modifying the Glu units in this polypeptide and using alkyne-azide click chemistry to crosslink the system, a transparent polypeptide single network gel was generated. After sufficient dialysis to remove the copper catalyst, the whole network was immersed into a modified di-acrylate solution mixed with photo initiator, after the gel was fully swollen inside this solution, the whole system was exposed to UV light to form the second network (PA).

The DN network shows several promising properties in relation to its tissue engineering application. The great cell biocompatibility can be found even after 1-week-long cell exposure, while the strength of DN is around 1.5 times the SN from the preliminary mechanical analyses.

Due to the difficulty on adjusting the samples size to fit into the shearing cell, two measurements were carried out. One is SAXS analyses of DN in its swollen state over time. In this case, the sample was cut into small pieces and put into a 2mm

capillary filled with water. As shown in Figue 1, a clear shifting of the seond domain size can be observed toward to smaller value, which is the direct resutl of polymer chain stretching in PP netwok.

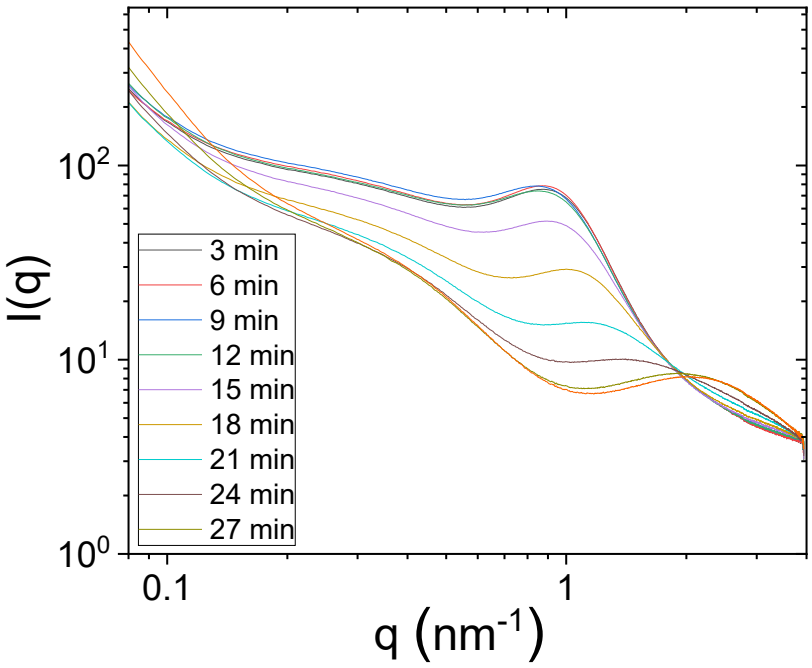

Figure 1: SAXS profile of double network (M(PP):M(PA)=1:2) after swelling in water solution over time.

The second test is ex-situ shearing test. The swelling sample underwent a constant shearing for 5mins, and then mounted on a sample stage and tested. As shown in Figure 2

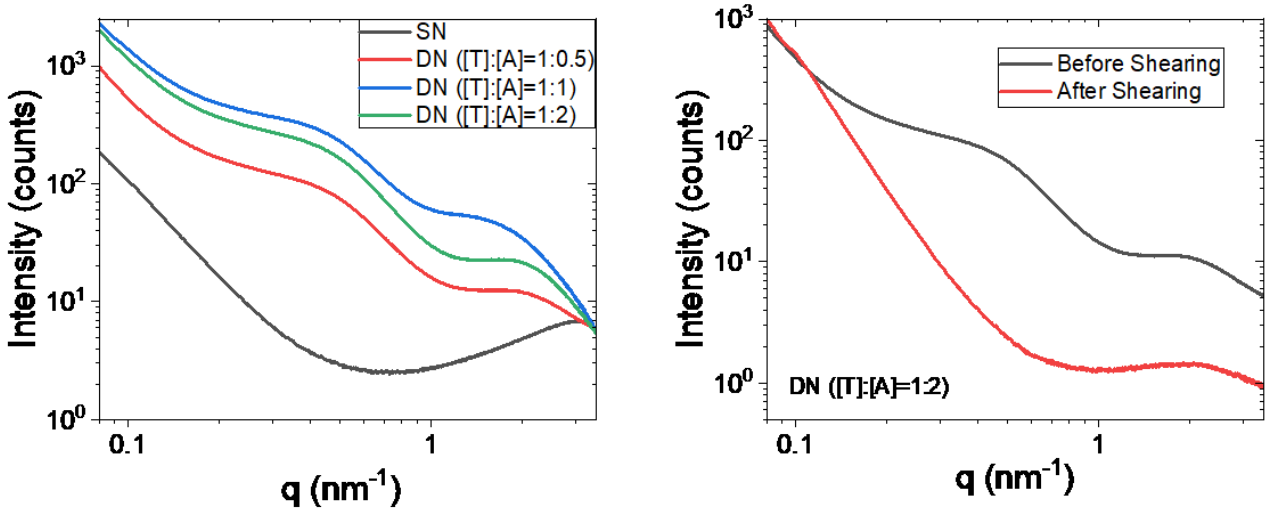

Figure 2. SAXS profile of : (a) swelling networks; (b) the comparison between swelling network before and after applying forces;

The whole experiment helps us to further advance our knowledge on the impact of swelling and individual network ratio on the DN domain distributions, and the manuscript is under preparation currently.