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.

	Experiment title: Time-Resolved SAXS Study of Colloidal Mg-stabilized Amorphous CaCO3 Precursor for Additive Manufacturing	**Experiment number**: CH-6486
Beamline:	**Date of experiment**: from: 13/09/2022 to: 16/09/2022	**Date of report**: 23/10/2022
Shifts:	**Local contact(s)**: SZTUCKI Michael	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Prof. POKROY Boaz, Technion - Israel Institute of Technology Department of Materials Engineering
Dr. POLISHCHUK Iryna, Technion - Israel Institute of Technology Department of Materials Engineering
Prof. FALINI Giuseppe, Laboratory University of Bologna Department of Chemistry "Giacomo Ciamician"

Report:

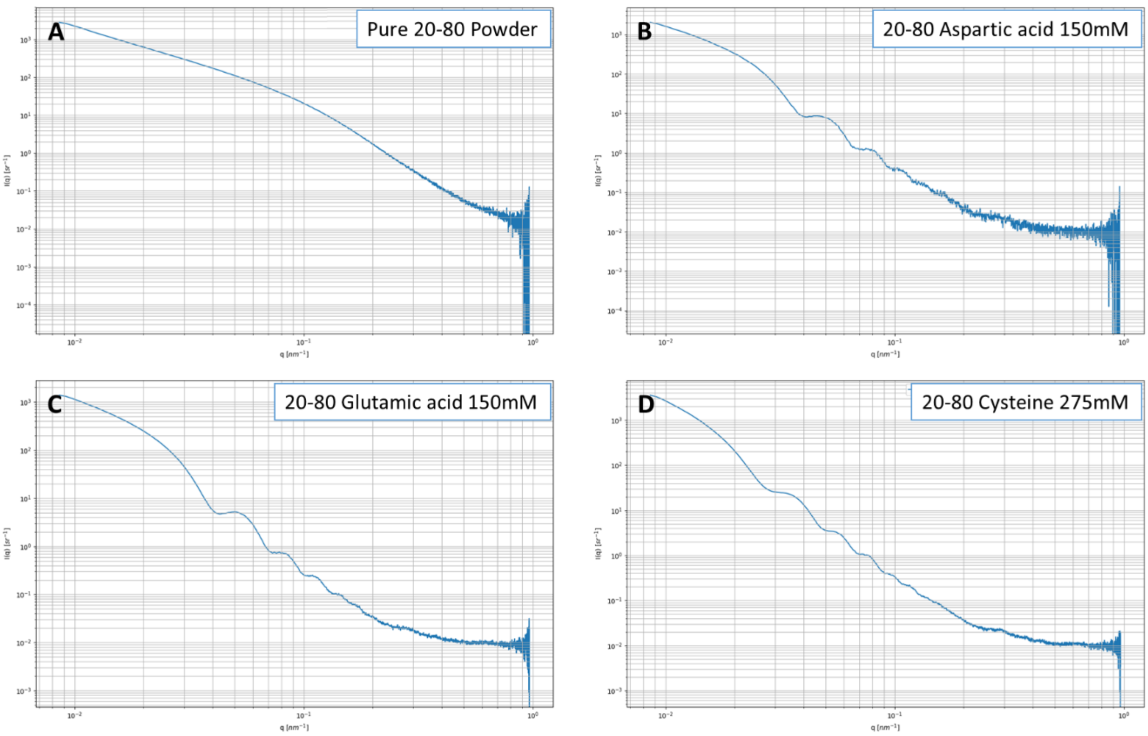

Figure 1 - Small-angle X-ray scattering difractograms of ACC powders formed from 20% Ca and 80% Mg solutions incorporated with various amino acids. A) pure 20-80 powder, B) with 150mM of Aspartic acid, C) with 150mM of Glutamic acid and D) with 275 mM of Cysteine

The influence of amino acids incorporation in amorphous calcium-carbonate (ACC) synthesis on the formation rate and the size\shape of the formed particles has been examined using small-angle X-ray scattering. *Figure 1* depicts the changes in grain distribution resulting from amino acid incorporation. Mixing of Ca^{+2}+Mg^{+2} mixture solution with CO_3^{-2} solution results with instant precipitation of ACC particles in the liquid, yet as can be seen in *Figure 1 A*, the received particles are not uniform in size or shape. Amino acids incorporation in ACC synthesis is known to inhibit the formation of particles, and as can be seen in *Figure 1 B-D* they play an important role in the formation of particles with uniform size and shape. This result was yet to be seen prior to this experiment and may offer another explanation to the found amino acids in organisms who use the amorphous-to-crystalline transformation in nature. The varying concentration of amino acid is also an intriguing point, possibly resulting from their chemical structure.

The dependency of the Mg-to-amino acid ratio in the inhabitance of ACC particles formation and size\shape of the particles was also examined as depicted in *figure 2*.

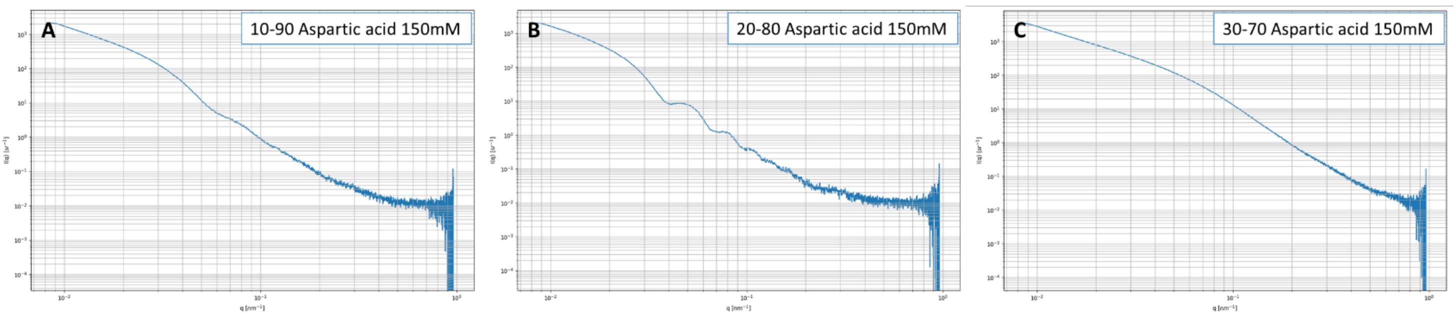

Figure 2 - Small-angle X-ray scattering difractograms of ACC powders formed with 150 mM of Aspartic acid, varying in Ca:Mg ratio, where A)10% Ca, 90% Mg, B)20% Ca and 80% Mg and C) 30% Ca and 70% Mg

As can be seen in *figure 2 A*, high Mg percentage results in small periodicity indicating uniformity in size and shape of the formed ACC particles. When the Mg to aspartic acid ratio is lowered in *figure 2 B*, the periodicity increases and a more uniform size distribution is formed. At lower ratios the particles formation is no longer inhibited and the periodicity cannot be observed. These results indicate that there is a specific ratio allowing both inhabitance and uniformity in grain size of the formed ACC particles.

The aforementioned results may shed light to an interesting phenomenon seen in nature where Mg-rich $CaCO_3$ crystals with amino acid surrounding is used to form uniquely shaped and mechanically enhanced crystals through an amorphous precursor.