EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** Dynamics of structure formation during gelation of resorcinol-formaldehyde hydrogels	**Experiment number**: SC-3157
Beamline:	**Date of experiment**: from: 29.06.2011 to: 04.07.2011	**Date of report**: 22.02.2012
Shifts: 18	**Local contact(s)**: Orsolya Czakkel	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Krisztina László (Budapest University of Technology and Economics, Budapest, Hungary)

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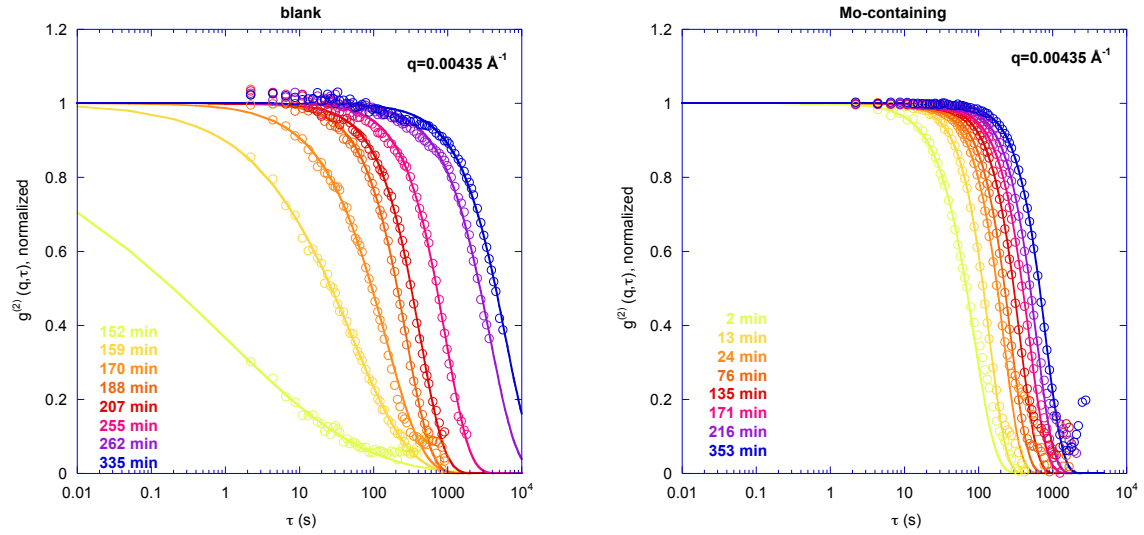
Report:

The aim of the experiment was to investigate the effect of molybdenum on the synthesis of resorcinol-formaldehyde (RF) polymer gels. To introduce the molybdenum to the RF gel system the standard preparation process was modified. A pre-polymerization period was applied, whereby the aqueous sol containing the resorcinol, formaldehyde, and sodium-carbonate was placed in an oven for incubation at 70°C, in order to promote the polycondensation reaction. This period is referred to as pre-polymerization time. After the given period, an appropriate amount of ammonium-heptamolybdate (AM) solution was added to the RF sol. Seven samples, with pre-polymerization times varying between 70-120 min were measured. One additional sample, containing only sodium-carbonate as a blank sample was also prepared and investigated.

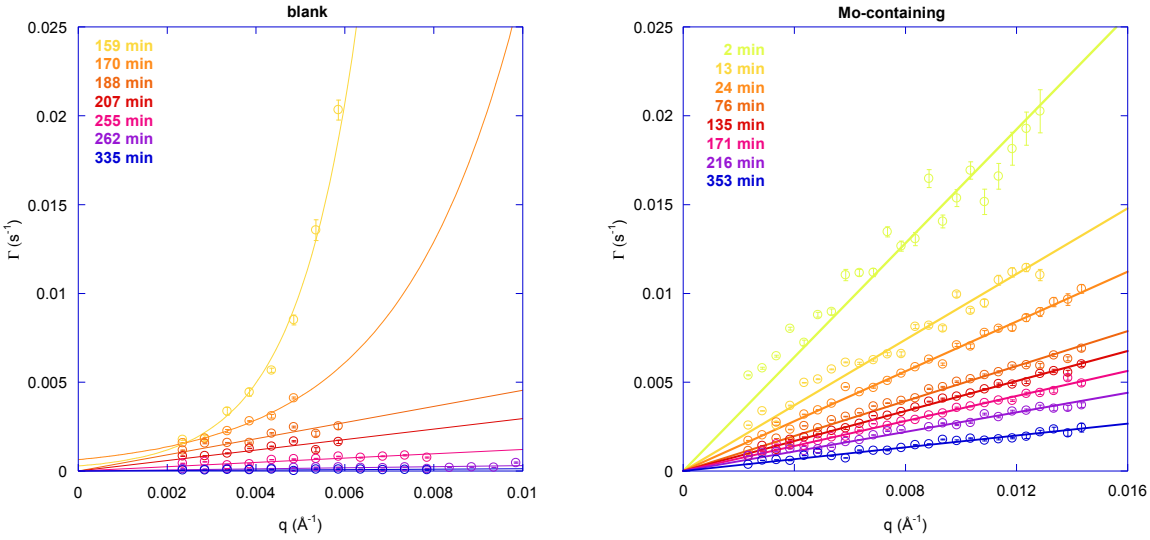
The samples after addition of the AM solution were inserted into borosilicate capillaries of 1.5 mm in diameter and placed in the thermostated (70°C) measurement chamber. Owing to the relatively slow gelation procedure of these systems, the samples were measured over several hours to follow the structural and dynamical changes. Prelilminary results from the data analysis show that the presence of molybdenum affects the kinetics of the process significantly. Figure 1. presents a set of normalized, time averaged correlation functions ($g^{(2)}(q,t)$) measured in samples after 100 min of pre-polymerization, with and without the presence of molybdenum. In the case of the blank sample no relaxation of the correlation functions was observed before 152 min after the addition of the catalyst (Na_2CO_3) solution at the end of the pre-polymerization period, while with molybdenum the process is immediately slow enough to fit into the time window of the measurement.



(a) (b)

Figure 1. Normalized, time averaged correlation functions at q=0.00435Å^{-1} of (a) the blank sample (b) Mo-catalysed sample. Both prepared with 100 min pre-polymerization time. Solid lines represent the corresponding Kolrausch-Wiliam-Watts fits.

The result of the fits obtained by the Kolrausch-Wiliam-Watts formula are shown in Figure 2. In the case of the blank sample at early stages the dominant processes that govern the dynamics are related to the diffusion of the primary clusters, as the exponential behaviour of Γ with q suggests (Figure 2a). At later stages the relaxation rate exhibits a linear dependence on q. In the case of the Mo-catalysis the system is always in the hyperdiffusive state, Γ being propotional to q (Figure 2b).



(a) (b)

Figure 2. Variation of Γ with q and time in the (a) blank and (b) Mo-catalyzed samples. Both samples were prepared with 100 min pre-polymerization.

Further analysis of the data to discover the effect of the applied prepolymerization time is in progress.