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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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	Effect of particle shape on the structure and rheology of colloidal systems with attractive interactions	Experiment number: SC2201
Beamline:	Date of experiment:	Date of report:
ID02A	from: 30-04-2007 to: 01-05-2007	
Shifts:	Local contact(s):	Received at ESRF:
3	T. Narayanan	
Names and affiliations of applicants (* indicates experimentalists):		
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Report:

The objective of these experiments was to investigate the effect of particle shape on quiescent and flowinduced microstructure of model colloids interacting through short-range attractive potential. Hematite particles with variable aspect ratio can be made using forced hydrolysis of iron chloride using the method of Ozaki et al. [1], originally proposed by Matijevic et al., [2-4]. A silica coating was applied using the method of Thies-Wiessie et al. [5], which were subsequently coated with octadeconal [6]. Using a solvent where the octadecanol brush collapses as a function of temperature, in this case n-tetradecane, yielded a thermoreversible system of ellipsoids. Hence, the results can be compared with sticky sphere system to elucidate the effect of particle shape, synthesized by identical coating procedure. S-coated Hematite particles had an aspect ration of 3.1 (major axis – 340nm; minor axis – 110nm), silica coating was confirmed by EDAX measurements and shell thickness was determined to be 25 nm by SEM characterization.



Figure 1 – Scanning Electron Microscopy (SEM) images of a) Hematite b) Silica coated Hematite particles

Static and Flow-induced structure:

Preliminary results were performed on sticky ellipsoid suspensions. As expected, the experiments revealed the formation of aggregate networks of ellipsoids as shown in fig.1.b, the basic aggregates formed where rather dense. We where plagued by the presence of some irreversibility in the aggregation and could within the experimental time frame not investigate if this was a kinetic effect or due to problems during the synthesis. Data for a few volume fractions was obtained using SAXS and USAX, an example is shown in figure 1.a The resulting microstructure under flow was, somewhat counter intuitively, less affected by shear flow compared to the case of sticky spheres (see SC2200). A volume fraction of 0.20 was studied using 2D-SAXS for a range of shear rates. An isotropic pattern was observed, as in figure 1.b, with at most a slight densification of the structure taking place. This weaker dependence to shear flow is probably related to the more dense local structure of the ellipsoid clusters as compared to the spheres, as suggested by recent experiments by Solomon and coworkers [10]. The results are consistent with data of the linear rheology obtained off-site.



Figure 2 – a) I vs q – static scan for 0.06 volume fraction and b) 2D pattern obszerved during flow of a suspension at volume fraction of 0.20 at 10° C.

As the allocated beam time (3 shifts) was less than what we asked for (9 shifts), we could not complete our experimental program. We therefore will submit a request to continue this proposal, with some changes to the suspension chemistry.

References:

- [1] M. Ozaki, S. Kratohvil and E. Matijevic; J. Colloid Interface Sci. 1984. 102 146-151.
- [2]. Matijevic, E., *Langmuir* 2, 12 (1986).
- [3]. Matijevic, E., Chem. Mater. 5, 412 (1993).
- [4]. Matijevic, E., and Scheiner, P., J. Colloid Interface Sci. 63, 509 (1978).
- [5] D.M.E. Thies-Weesie, A.P. Philipse and S.G.J.M. Kluytmans, J. Colloid Interface Sci. 174, 211(1995).
- [6] van Helden, A. K.; Jansen, J. W.; Vrij, A. J. Colloid Interface Sci. 1981, 81, 354-368.
- [7] S. Sacanna et al., Langmuir 2006, 22, 1822-1827
- [8] Ocan[~]a, M.; Morales, M. P.; Serna, C. J. J. Colloid Interface Sci. 1999, 212, 317-323.
- [9] Graf, C.; Vossen, D. L. J.; Imhof, A.; van Blaaderen, A. Langmuir 2003, 19, 6693-6700.
- [10] Mohraz A, Moler DB, Ziff RM, Solomon MJ, Effect of monomer geometry on the fractal structure of colloidal rod aggregates PRL 92 (15): Art. No. 155503 (2004).