

**Abstracts for posters:**

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**Fifth Nordic Workshop on Scattering from Soft Matter Trondheim Feb 6-7 2008**

**Auditorium R9 Realfagbygget NTNU**

# **POSTERS**

# **ABSTRACTS**

## Attractive interactions of PEO-containing block copolymer micelles in salt solutions.

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Molecular self-assembly is widely used in the industry. The properties of products like paints, inks and cosmetics can be contributed to this. As well as these commercial applications polymers are also interesting with regard to drug delivery.<sup>1</sup> Systems containing poly(ethylene oxide) (PEO) have been shown to be a good model system for self-assembly studies. PEO is chemically the simplest water soluble polymer as well as the simplest bio-compatible polymer, not recognizable by the human immune system. PEO is shown to form spherical micelles in water when linked to a small hydrophobic tail.<sup>2,3</sup> In this project the attractive interactions of the micelles formed by the block copolymer Brij 700 is studied. This polymer is composed of a PEO chain with 100 EO units linked to a hydrophobic tail of C<sub>18</sub>. Use of 0.9 M NaF as solvent has been shown to lower the cloud point sufficiently for studying both intermicellar repulsive and attractive interactions at manageable temperatures. Turbidity measurements have been carried out as a function of temperature on several concentrations in the range from 0.2 to 5 wt% to determine the cloud points. The zero shear viscosity of the system has been determined in the temperature range from 5 to 60 degrees at different weight concentrations. A SAXS study at concentrations of 1,2 and 5 wt% has been carried out in a broad temperature range and analysis of these data is in progress.

<sup>1</sup> Hamley, I.W.: 'Introduction to Soft Matter' (Wiley, 2000)

<sup>2</sup> Sommer, C., Pedersen, J.S., Temperature Dependence of the Structure and Interaction of Starlike PEG-Based Block Copolymer Micelles. (2004) *Macromolecules* 37, 1682-1685

<sup>3</sup> Sommer, C., Pedersen, J.S., Garamus, V.M., Structure and Interactions of Block Copolymer Micelles of Brij 700 Studied by Combining Small-Angle X-ray and Neutron Scattering. (2005) *Langmuir* 21, 2137-2149

## Scattering from mixed micelles analysed with a spherocylinder + sphere model

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We have studied mixed micelles formed by two non-ionic surfactants with very different headgroups, *n*-dodecyl-hexaethylene glycol ( $C_{12}E_6$ ) and *n*-dodecyl- $\beta$ -D-maltoside ( $C_{12}G_2$ ), as well as the pure amphiphiles, with small angle x-ray and neutron scattering (SAXS and SANS). Both scattering data sets were analysed simultaneously. For all samples containing  $C_{12}E_6$ , we used a new form factor for spherocylinders coexisting with spheres. Spherocylinders have end-caps with a radius that is larger than for the cylindrical part. The negative curvature induced by the radius difference results in a gap in the size distribution of the cylindrical micelles, where some short lengths have a very low probability. The 'shortest end' of this distribution is represented by spherical aggregates of the same size as the endcaps that will exist since they do not have this unfavourable curvature. Pure  $C_{12}G_2$  micelles were modelled as two-axial ellipsoids.

Results show that spherical micelles of all compositions are of the same size, whereas the cylindrical micelles are longer the more  $C_{12}E_6$ . Pure  $C_{12}G_2$  micelles are globular which means that this surfactant prefer curved surroundings. A small amount of  $C_{12}E_6$  induces micellar growth in mixtures with  $C_{12}G_2$ , but still a significant part of the surfactants are present in spherical aggregates; about 30 % of the surfactants in a pure 0.5% (w/w)  $C_{12}E_6$  sample is in spherical micelles and this figure increases with the amount  $C_{12}G_2$ .

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**Abstracts for posters:**

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**Fifth Nordic Workshop on Scattering from Soft Matter Trondheim Feb 6-7 2008**

**Auditorium R9 Realfagbygget NTNU**

**SAXS at MAX – Current status**

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The Small Angle X-ray Scattering (SAXS) set-up on beamline I711 has now been in operation for almost two years. Although already successfully used for a wide range of different (SAXS-) applications there is a continuously ongoing work in order to update the set-up. Extra attention has been on improving the experimental conditions when working with weakly scattering samples both to decrease to noise level but also to make as accurate background subtractions as possible. Also new type of sample environment cells have either already been added or will be in the near future. The SAXS set-up at I711 will continue to be open for users through proposals addressed to MAX-lab and the next deadline for submitting these proposals is in the end of February.

## **Kinetics of Oil Expulsion in a New Non-ionic Microemulsion Studied by Time-Resolved SAXS**

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Microemulsion systems containing chlorinated alkanes as oils using nonionic surfactants has hardly been studied. These systems might display interesting behaviors due to the different properties of the oil and is therefore a need for systematic investigations of such systems in order to better understand their phase behavior and the molecular transport/exchange in the various phases. We have formulated a new microemulsion system consisting of pentaethylene oxide surfactant ( $C_{12}E_5$ ), chlorinated alkane and water. Systematic phase behavior of this new system was studied by the well known Fish-plot methods and the kinetics of phase separation was followed both by time-resolved small angle scattering (SAXS) and turbidity techniques.

We followed the kinetics of phase separation in the  $L_1+O$  phase, and resolubilization of oil to the  $L_1$  phase of microemulsion of the type  $C_{12}E_5$ /water/1-chlorotetradecane. The stable microemulsion in the  $L_1$  droplet phase (24 °C) was temperature quenched to 13 °C. At this temperature which corresponds to a two-phase system, oil droplets nucleate and grow in size as function of time. The SAXS spectra were quantitatively analyzed on an absolute scale using a polydisperse hard-sphere model with molecular constraints for the microemulsion droplets and a polydisperse sphere model for the oil droplets.

The results indicate that the kinetics of oil expulsion in the present system follows a coarsening process similar to an Ostwald-like ripening process.

## Structural and Dynamical Aspects of the Cold Crystallization of a Flexible Polymer as seen by Time Resolved Neutron Scattering and Broadband Dielectric Spectroscopy

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Polymers that possess a certain molecular symmetry around its backbone have a natural tendency to crystallize into highly ordered domains. However, given the connectivity between segments, the dynamics of polymers is inherently slow which often kinetically counteracts the formation of crystals. As a result the structure of polymers is most often only partially or non-crystalline (amorphous).

In order to understand the mechanism governing the crystallization in polymers it is important to understand both the polymer dynamics and the structure and how they are interrelated. Polydimethylsiloxane (PDMS) belongs to a rather peculiar class of extremely flexible polymers which exhibits a very small energy barrier for rotations around their backbone. In view of this, PDMS represents a natural model system possessing very fast dynamics where the mobility of the chains can be directly related to the crystallization process.

Here we present a complete study of the structural and dynamical aspects of the crystal formation of PDMS starting from a fully amorphous glass state at low temperatures. Using time resolved wide angle neutron scattering (WANS) and small angle neutron scattering (SANS) we are studying the structural aspects of the crystallization over a wide range of length scales of approximately 5000 – 2 Å. This is combined with a real time analysis of the segmental dynamics by using broadband dielectric spectroscopy (BDS) and the overall thermodynamical properties employing differential scanning calorimetry (DSC). In this communication we will concentrate on the kinetic results obtained using the different techniques which will be compared and analyzed with particular emphasize on the interplay between the structural and dynamical properties. A particular emphasis is made on crystallization mechanism and the potential role of mesomorphic phases acting as precursors in the transition from the amorphous to crystalline state.

## The Role of Interfacial Fluctuations and Self-Concentration on the Segmental Dynamics of Block Copolymer Melts

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Block copolymers spontaneously self-assemble into well defined structures with variable geometry (spherical, cylindrical, lamellar etc) depending on the chemical details (molecular structure, composition etc) and external conditions such as temperature and pressure. It is of significant interest to understand how this structure formation, typical in the range of some tenths to hundreds of nanometer, affects the polymer chain dynamics.

For this purpose, poly(isoprene)-poly(dimethyl siloxane) block copolymer melts are ideal as a model system as the both polymers exhibit a low glass transition temperature, strong mutual repulsion ( $\chi \sim 0.11$ ) and well-separated dynamics. Consequently PI-PDMS spontaneously self-assemble to well defined equilibrium structures. Most importantly in the context of dynamics, cis-PI exhibits a net dipole both parallel and perpendicular to its backbone allowing a simultaneous observation of both the global normal mode relaxation as well as the local alpha relaxation using dielectric relaxation spectroscopy. By comparing the dynamics of the corresponding pure PI homopolymers, the effect of confinement on the dynamics can be directly deduced.

We will present first results of a study of various PI-PDMS block copolymer melts where the PI part varies between 5 000 to 10 000 g/mole. Using small angle x-ray scattering (SAXS), we deduce the detailed structure and the ordering behaviour (crystal structure) of the system. Dielectric spectroscopic measurements show that the confinement induces a faster and broadened normal as well as local relaxation process having an extra fast relaxing component in the high frequency side of the dielectric loss peak.

In this communication we show that by properly taking into account self-concentration and interfacial fluctuation effects, we show that both the broadening and fastening of the alpha-relaxation can be quantitatively accounted for. The extracted width parameters were found to be in the range of the estimated values based on thermodynamical and capillary wave dynamics arguments. These results not only demonstrate the importance of fluctuations effects on the local alpha-relaxation but also how BDS can complement scattering techniques to give highly detailed structural information.

## **Structural Ordering of Polystyrene Latex under Flow**

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Studies of the structure can provide important information about the rheological properties of complex fluids. Simple model systems are often useful starting points for such investigations. Polystyrene latex was prepared by emulsion polymerization. It was dialyzed and stored over ion exchange resin. The latex was characterized using atomic force microscopy, scanning electron microscopy and dynamic light scattering in order to establish its particle radius as well as small angle scattering. Initial flow experiments used a simple rectangular section pipe. This geometry has the advantage that domains can be studied in different orientations with respect to the incident beam. On the D11 SANS instrument at the ILL, Grenoble, the flow cell could rotate  $\pm 30$  degrees about a vertical axis and the resulting diffraction pattern was investigated. Preliminary results will be presented on the poster.



**SAXS and MAXS for soft matter**

Abstract

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The Bruker Microstar generator (2.7 kW Cu, 0.1 x 0.1 mm source size) has two ports for optics. This allows to build two beamlines (SAXS and MAXS) using a single generator.

**SAXS**

In the SAXS setup a Montel parallel optics with two Göbel mirrors make the beam parallel and monochromatized. The collimation of the beam is done with two defining slits and with two guard slits. The data is collected using a 2-dimensional position-sensitive gas detector (Hi-Star). The complete beam path from the first slit to the detector is evacuated. There is also an option to use helium gas in the sample chamber. The sample-to-detector distance can be varied from 0.5 meters to 5 meters. These configurations cover the magnitude of scattering vectors  $q$  from 0.0025 to 0.5  $\text{\AA}^{-1}$ .

**MAXS**

The beam is focused to the detector by Montel focusing optics. Three guard slits are used to minimize the parasitic scattering. The beam diameter at the sample is less than 1 mm. The complete beam path from the optics to the detector is evacuated. There is also an option to use helium gas in the sample chamber. The sample to detector distance can be changed by moving the sample inside the chamber by a motorized linear stage. Depending on the sample to detector distance the configuration covers the magnitude of scattering vectors  $q$  from 0.025 to 0.85  $\text{\AA}^{-1}$  or from 0.08 to 2.9  $\text{\AA}^{-1}$ . The flux is  $10^9$  photons  $\text{s}^{-1}$  for Cu  $K\alpha$  radiation at a generator power of 2.7 kW.

In both beamlines the intensity of the incoming and transmitted beam can be monitored with a semiconductor sensor. Motorized sample heaters allow automated measurements of 20 samples in temperatures from 25 °C to 250 °C.

## Adsorption of Mixtures of Sodium Dodecyl Sulfate and Polyethyleneimine at the Air-Water Interface

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The adsorption of mixtures of sodium dodecyl sulfate (SDS) and polyethyleneimine (PEI) at different pH-values (4, 7, 10) at the air-water interface was investigated by ellipsometry. First the effect of different mixing protocols on the surface composition was studied. Mezei et al. reported, that the method of mixing the components has a crucial effect on the size of the SDS-PEI-clusters in the bulk<sup>1</sup>. However it was found that not the mixing method but a freshly created surface is the determining factor in order to get a reproducible, homogeneous composition of the surface.

*For pH 4 and 7 comparable results are obtained, whereas for the mixtures at pH 10 a slightly different behaviour can be seen. This is in good agreement with the fact that at the lower pH-values PEI is a polycation whereas at the natural pH 10 PEI is nonionic. The oppositely charged polymer/surfactant system was investigated in detail.*

In the concentration region close to charge neutralization concentration of SDS for the PEI-SDS complex a rapid decrease in the ellipsometric response can be observed. But as the signal does not reach the value for the pure SDS-solution no complete displacement of the PEI occurs. At low SDS concentrations the thickness of the layer for the mixtures varies from that of the pure PEI-solution. It can be concluded that over the whole investigated range of SDS concentrations SDS-PEI complexes are adsorbed on the surface and do not desorb completely, but might only change their conformation.

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<sup>1</sup> A. Mezei, R. Mészáros, I. Varga, T. Gilányi, *Langmuir* **2007**, 23, 4237-4247.

## Structure of PEP-PEO Block Copolymer Micelles in Solvents of Varying Selectivity: A Combined SAXS and Light Scattering Study

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Micelles of the block copolymer poly(ethylenepropylene)-b-poly(ethylene oxide) (PEP-PEO) with varying PEO length have been studied by SAXS and static light scattering (SLS). The two methods cover different ranges of the scattering vector  $q$ , and the relative contrast of PEP and PEO are different for them and they are therefore complementary. The PEP block had a fixed molecular weight of 5 kDa whereas the molecular weight of the PEO block was varied from 2.5 to 50 kDa. The solvents were water, having the highest selectivity for the PEO block, ethanol and mixtures of the two with lower selectivity. A model of spherical core-corona micelles was used for analysing the SAXS data. The high aggregation number, favoured by a decrease in core surface area, is limited by the entropy cost of stretching the PEO chains in the corona due to chain-chain interactions, as well as by stretching of the PEP chains in the core in order to make them space filling. The more selective the solvent is to the PEO block, the higher aggregation numbers are observed since stretching is relatively less unfavourable in this case. Increased molecular weight of the PEO block also leads to smaller aggregation numbers to limit PEO chain stretching due to chain-chain interactions. At low molecular weights for PEO, the chain-chain interactions for a spherical micelle would be rather low, and at some point formation of cylindrical micelles with a lower curvature is expected. However, this is not observable in the high resolution (low  $q$ ) SAXS data due to unfavourable contrast conditions with negative contrast for PEP and positive for PEO. However, for SLS the contrast conditions are very different with positive contrasts for both blocks. The SLS data, for the samples with 2.5 kDa PEO have a slope in a log-log plot of scattering intensity vs scattering vector that differ clearly from the rest, and indicate a cylindrical shape of these micelles. Hydrodynamic radii obtained from dynamic light scattering show that these micelles are much larger than the rest, confirming that they are not spherical.

## SAXS Studies of Glucagon: From Monomers to Fibers

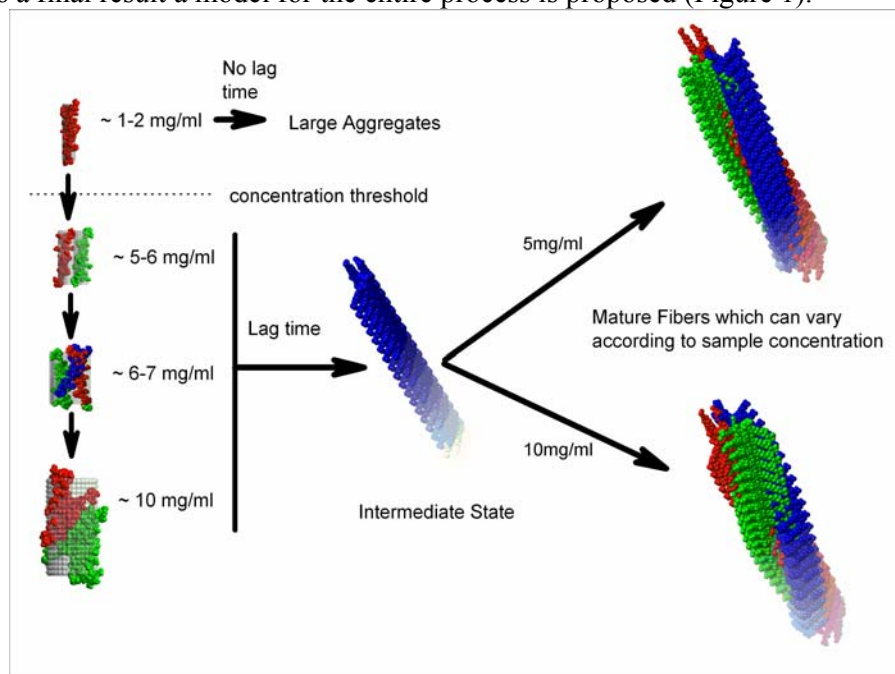
**Cristiano Luis Pinto Oliveira**<sup>1</sup>, Manja A. Behrens<sup>1</sup>, Jesper Søndergaard Pedersen<sup>2</sup>, Kurt Erlacher<sup>1</sup>, Daniel Otzen<sup>2,3</sup> and Jan Skov Pedersen<sup>1</sup>

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Glucagon fibrils have a high resemblance to the class of amyloid fibrils [1], which among other diseases cause Alzheimer's disease and Creutzfeldt-Jakobs syndrome [2]. Since it is very easy to make fibers of glucagon, it has been used as a model system for amyloid like fibrillation. The mature fibers of glucagon have been well characterized by electron microscopy studies [3,4], but very little is known about the intermediate steps where many tentative models have been proposed [4]. Using Small-Angle X-ray Scattering (SAXS), we performed a comprehensive study of the full fibrillation process, starting on the early oligomerization stages up to the formation of mature fibers. Several model strategies were applied and as a final result a model for the entire process is proposed (Figure 1).



**Figure 1 - Fibrillation model for Glucagon samples obtained from SAXS modeling.**

[1] Onoue, S. et al. (2004). *Pharmac. Res.* (21), 1274-1283

[2] Dobson, C. M. (2003). *Nature* (426), 884-890

[3] Pedersen, J.S. et al. (2006). *J. Mol. Biol* (355), 501-523

[4] De Jong, K.L et al (2006) *Bioph. Journal.* (91), 1905-1914

## **SAXS studies of protein-surfactant complexes**

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Protein-surfactants interactions has been studied extensively for decades due to their importance in many biological, pharmaceutical and industrial systems. Many different protein-surfactant systems have been characterized by thermodynamic and biochemical measurements but, very little structural information is available on the structure of the formed complexes. We use small-angle x-ray scattering and modeling methods for obtaining structural information on a protein-surfactant system. A relatively simple model based on cylinders and cylindrical shells was successfully applied to describe the complex formation between the anionic detergent sodium dodecyl sulfate (SDS) and the 86-residue  $\alpha$ -helical bovine Acyl-coenzyme A-binding protein (ACBP) [1].

[1] Andersen KK, Larsen KL, Poulsen F, Pedersen JS, Oliveira CLP, Callisen T, Westh P and Otzen DE. *Denaturation of ACBP by SDS hemi-micelles, in preparation*

## Size, shape and charge in cationic microemulsion droplets: a SANS study

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Cationic surfactants with significant asymmetry in chain length can be water-soluble at (or near) room temperature, presenting several unusual features in their self-assembly<sup>1,2</sup>. If there is a large difference in solubility between the individual cationic and anionic amphiphiles that compose the ion-paired amphiphile, the cationic aggregates in solution will be slightly charged (instead of neutral, as could be expected). Hence, the solubility in water of the cationic compound is greatly enhanced, as compared to a symmetric one.

Vesicles, micelles and coexisting lamellar phases can be found in the binary cationic surfactant-water system. Furthermore, upon the addition of oil, the formation of oil-in-water (o/w) microemulsions has been observed. In the present work, we have investigated by SANS o/w microemulsion droplets, forming along the emulsification failure (EF) boundary, by means of two different contrasts, bulk and shell<sup>3</sup>. It is found that the droplets are spherical with almost invariant size in the studied region, as expected, and with good agreement between both contrast methods.

Further away from the EF boundary, when the oil-to-surfactant ratio is lowered, the droplets remain spherical but their size decreases. After some o/s ratio, more unusual shapes are found. Analysis of the structure factors also suggests that the droplets have charge, the magnitude of which is dependent on the total surfactant concentration.

- (1) Silva, B. F. B.; Marques, E. F. *J. Colloid Interface Sci.* **2005**, *290*, 267.
- (2) Silva, B. F. B.; Marques, E. F.; Olsson, U. *J. Phys. Chem. B* **2007**, *111*, 13520.
- (3) Bagger-Jørgensen, H.; Olsson, U.; Mortensen, K. *Langmuir* **1997**, *13*, 1413.

## Wide Angle Scattering Study of Organic Modified Fluorohectorite Electrorheological Suspension

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**Abstract:** Organic modified Na-fluorohectorite electrorheological suspension was synthesized by the combination of ion exchange and liquid-liquid phase transfer method. Firstly, Cethyl-Trimethyl-Ammonium Bromide (CTAB) modified Na-Fluorohectorite is synthesized by ion exchange method. Through the formation of the organoclay, the properties of clay change from hydrophilic to hydrophobic and that is suitable for the transfer from aqueous phase to organic phase. Then a liquid-liquid phase transfer method was employed to prepare novel organic modified Na-fluorohectorite/silicone oil suspension. The transfer process was certified by the rapid change of color for the aqueous and organic phase. The three-dimensional WAXS images from chains of CTAB modified Na-Fluorohectorite under a DC external electric field exhibit a marked anisotropy patterns, which is analyzed close with its electric polarisation and chain formation. The dynamic formation of the chain for CTAB modified Na-fluorohectorite ER suspension is also investigated.

### References:

- [1] Fossum JO, Meheust Y, Parmar KPS, et al. EUROPHYSICS LETTERS 2006, 74 (3), 438-444.
- [2] Parmar KPS, Meheust Y, Schjelderupsen, B.; Fossum JO, Langmuir; **2008**; ASAP Article; DOI: [10.1021/la702989u](https://doi.org/10.1021/la702989u).

## Wide Angle Scattering Preliminary Studies of Dynamic Chain Formation in Na-FLHC

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**Abstract:** The most common electrorheological fluids (ERFs) are suspensions of polarizable particles dispersed in an inert matrix insulating liquid. Under external electric fields ER fluids experience dramatic changes in rheological properties: a viscoelastic solid is created that yields when the applied stress exceeds a limit value. This phenomenon is also reversible when external electric field is removed. Because of their controllable viscosity and short response time, ERF materials are regarded as smart materials for active devices, brakes, clutches shock absorbers and actuators.

In the present study, Na-Fluorohectrite (Na-FLHC) clay particles suspended in insulating fluid (silicon oil) form with time chain-like structures when subjected to an external electric field. The time needed to create columns of aggregated particles varies as the electric field (0.35, 0.50 and 0.75 kV/mm) increases. Wide Angle X-Ray Scattering diffraction patterns reveal changes of the direction of the dipolar moment induced in clay particles when the electric field is applied.

### References:

1. J.O. Fossum, et al. "*Intercalation-enhanced electric polarization and chain formation of nano-layered particles*", Europhys.Lett. 74 (3), pp.438-444 (2006).
2. J.O. Fossum, et al. "*Electric polarisation and chain formation of nanolayered particles*", European Synchrotron Radiation Facility, ERSF Scientific Highlights 2006, Published February 2007.
3. Parmar KPS, Meheust Y, Schjelderupsen, B.; Fossum JO, Langmuir; **2008**; ASAP Article; DOI: [10.1021/la702989u](https://doi.org/10.1021/la702989u).



## Diffraction to study the alignments of particles in liquid dispersions.

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Previous studies of flow alignment [1, 2, 3, 4] have been extended to use angle dispersive diffraction of high energy (50 keV) x-rays to map alignment distribution in complex flows of concentrated clay dispersions. The poster presents methods to evaluate data and initial results for flow through a uniform pipe and a constricting nozzle. Typical raw data from a single  $50 \times 50 \mu\text{m}^2$  spot is shown in figure 1. Simple models are used to remove background and provide a parameterization of intensity. Figure 2 shows the results of a fit to the data in figure 1. The average intensity in the plane normal to the incident beam provides a measure of alignment parallel to the flow. This is shown in figure 3.

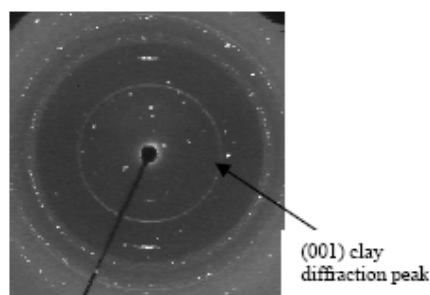


Fig.1. Shows the raw data diffraction patterns from the sample while flowing through the aluminum pipe.

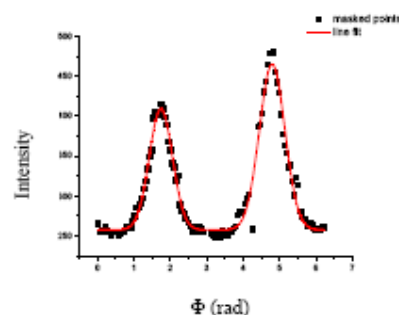


Fig-2. Diffraction intensity for the (001) peak is fitted as a function of azimuthal angle  $\phi$  with two Gaussians. Solid squares are the actual data points after masking while continuous line is a fit to data.

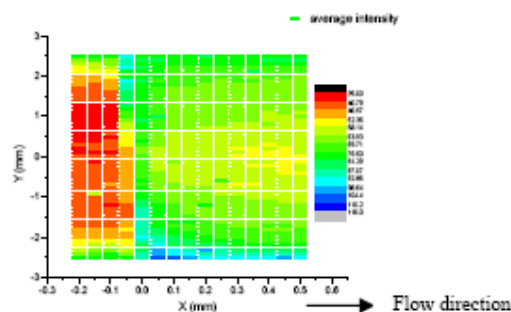


Fig-3. Shows the average intensity for (001) peak in the plane normal to the incident beam.

### Reference:

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