Colloquium on Rheo-NMR

Friday 18th August , 2017
Kenneth C. Rowe Management Building, Room 1009
Dalhousie University, Halifax, Nova Scotia, Canada

8:30 am **Welcome** - *Petrik Galvosas*

8:45 am **Invited Speakers** - *Chair: Petrik Galvosas*
8:45 - Claudia Schmidt - *In situ 2H NMR spectroscopy of complex fluids under flow*
9:30 - Joseph Seymour - *Probing soft matter physics using NMR*

10:15 am **Morning Break**

10:45 am **Contributed Oral Presentations** - *Chair: Hilary Fabich*
10:45 - Karl-Friedrich Ratzsch - *Benchtop Rheo-NMR*
11:05 - Daniel Holland - *The need for rheoNMR measurements of granular flow*
11:25 - Tatiana Nikolaeva - *Constructing local flow curves of complex yield stress fluids based on Rheo-MRI velocity profiles*
11:45 - Nicolas Schork - *Investigation of the influence of fluid dynamics on polymerization kinetics via Rheo-NMR spectroscopy*

12:05 pm **Lunch**

1:00 pm **Invited Speaker** - *Chair: Petrik Galvosas*
1:00 - Simon Rogers - *Transient rheology from a structural perspective*

1:45 pm **Round Table Discussion** - *Chair: Claudia Schmidt*

2:30 pm **Afternoon Break**

3:00 pm **Contributed Oral Presentations** - *Chair: Gisela Guthausen*
3:00 - Andy Sederman - *q-space PFG-NMR methods to characterise non-Newtonian fluids*
3:20 - Melanie Britton - *Using MRI to understand the interplay between molecular interactions and flow in complex molecular systems*
3:40 - Gianfranco Mazzanti - *Dalhousie's Rheo-NMR contributions to soft matter knowledge in fats and oils*
4:00 - Michael Johns - *(Reacting) Hydrate Slurry Rheology*

4:20 pm **Poster Presentations and Reception**

14th ICMRM 2017

TE WHARE WĀNANGA O TE ŪPOKO O TE IKA A MĀUI
VICTORIA
UNIVERSITY OF WELLINGTON

BRUKER

In situ ^{2}H NMR spectroscopy of complex fluids under flow

Claudia Schmidt

Department of Chemistry, Paderborn University, Warburger Str. 100, 33098 Paderborn, Germany

Flow can have pronounced effects on the structure of complex fluids, leading to rheological phenomena such as shear-thinning, shear-thickening or even transient oscillations of the viscosity. In order to better understand the rheological properties of complex fluids rheological measurements have been combined with structural investigations in socalled hyphenated techniques, such as rheo-light scattering, rheo-small angle x-ray scattering (rheo-SAXS), rheo-small angle neutron scattering (rheo-SANS) or rheo-NMR [1–3]. NMR has the advantages of being very versatile (use of different nuclei and different methods like spectroscopy, relaxometry, and diffusometry) and being applicable to non-transparent samples.

In this contribution some examples of rheo-NMR will be reviewed. The focus will be on ^{2}H rheo-NMR spectroscopy of anisotropic fluids where the quadrupole interaction provides a convenient probe to study orientations. In order to illustrate the potential of this technique, examples for the effect of shear on liquid crystalline polymers, different lyotropic phases, and phase transitions will be provided.

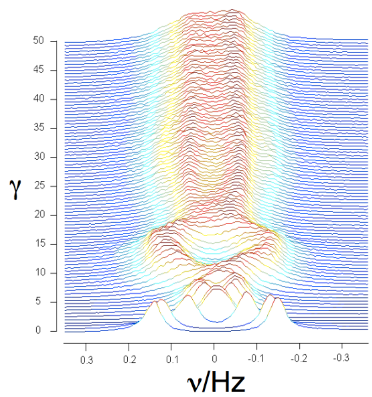

Fig. 1: ^{2}H spectra of a tumbling lyotropic nematic phase reflecting the transient director orientations as a function of strain γ at start-up of shear at a rate of c. 0.03 s^{-1}.

Literature

1. P. T. Callaghan, *Rep. Prog. Phys.* **1999**, *652*, 599–670.
2. P. T. Callaghan, *Current Opinion in Colloid Interface Sci.* **2006**, *11*, 13–18.
3. C. Schmidt, in *Modern Magnetic Resonance, Part 3*, 1515–1521, Springer, New York, **2008**.

ICMRM Halifax Rheo-NMR Meeting:

Title: Probing soft matter physics using NMR

Joseph D. Seymour, Chemical and Biological Engineering, Montana State University, Bozeman MT U.S.A.

Soft matter physics deals with systems that are complex due to the scales of the polymers, colloids, particulates or multiple phases of which they are composed (deGennes, 1992; Nagel, 2017). Soft matter materials underlie many systems encountered in daily life from foods to health and personal care products. The dissipative, heterogeneous and non-equilibrium nature of the microscale dynamics in most soft matter systems impact the macroscale material response. This has lead to microrheology (Mason and Weitz, 1995), *i.e*. probing the response of a material to the dynamics of tracer particles at a microscopic scale, becoming a significant method for connecting mesoscale structure to macroscale material properties. The role of NMR relaxation, diffusion and imaging in the study of soft matter systems will be overviewed with emphasis on colloid dynamics under shear, pattern formation in heterogeneous diffusive gelation and solvent dynamics in a weak gel of a concentrated polymer-solvent system.

References:

Pierre-Gilles de Gennes, “Soft matter–Nobel Lecture”, Reviews of Modern Physics **64**(3) 645-648 (1992).

Thomas G. Mason and David A. Weitz, “Optical measurements of frequency-dependent linear viscoelastic moduli of complex fluids”, Physical Review Letters **74**, 1250-1253 (1995).

Sidney R. Nagel, “Experimental soft-matter science”, Reviews of Modern Physics **89** 025002 (2017).

BENCHTOP RHEO-NMR

Karl-Friedrich Ratzsch, Manfred Wilhelm

Institut für Technische Chemie und Polymerchemie / Karlsruher Institut für Technologie

The development of compact Halbach-type permanent magnets for NMR [1] has made it feasible to integrate time-domain NMR equipment into laboratory rheometers. The *in-situ* combination of NMR relaxometry and advanced rheometry allows investigating the interplay of macroscopic mechanical behavior and molecular mobility under precisely controlled sample flow and deformation.

We would like to present the setup [2] developed in our group, using a strain controlled rheometer operating plate-plate and cone-plate sample geometries inside a Halbach magnet with B_0 = 0.7 T (30 MHz proton resonance). Experiments conducted with it include the investigation of crystallization and solidification of polyolefins under various shear protocols.

[1] H. Raich, P. Blümler, *Conc. Mag. Res. B,* **2004**, *23,* 16-25.

[2] K.-F. Ratzsch, C. Friedrich, M. Wilhelm; *Journal of Rheology*, **2017**, *61*, 905-917

The need for rheoNMR measurements of granular flow

D.J. Holland[1], D. Clarke[1], H.T. Fabich[2], T.I. Brox[3], P. Galvosas[3], J.R. Brown[4],S.L. Codd[4], J.D. Seymour[4]

1. University of Canterbury, New Zealand; 2. ABQMR, Albuquerque, New Mexico; 3. Victoria University of Wellington, New Zealand; 4. Montana State University, Montana

A complete rheological characterisation of granular materials does not yet exist. Granular materials have a yield stress as in solids, flow under gravity, akin to liquids, and exhibit compressibility similar to gases. Many of these features can be captured using a rheological model where the confining pressure determines the shear stress according to $\tau = \mu P$. However, there is significant debate over what defines the parameter μ. It is known that μ varies with pressure, shear rate, solid packing fraction and particle size [1]. On the basis of these observations, the so called $\mu(I)$ model was developed which captures many of the unusual features of granular flows, including a limiting yield stress when $\mu < \mu_s$ and steady flow on inclined planes. However, it does not describe, e.g., the slow flow that occurs when $\mu < \mu_s$ if the region is adjacent to a flowing region. New models of granular rheology are now being developed which incorporate, e.g., the effect of rotation of the particles or non-local affects linked to fluctuations in the velocity of particles about the local mean velocity [2,3]. To date these models have been developed based on macroscopic observations of flow and from numerical simulations; experimental measurements in dense, three-dimensional (3D) granular flows are not readily available.

Here, we use pulsed field gradient NMR to measure the granular dynamics in a 3D granular couette flow. From these measurements (1) the average local velocity is calculated from the phase of the PFG signal, from which we can determine the local shear rate, and (2) the velocity fluctuations about the local average velocity are calculated from the attenuation of the PFG signal, analogous to measurements of diffusion. Figure 1 presents a preliminary comparison of these measurements with the recently proposed granular fluidity model [2]. The agreement is striking. Unfortunately, we were not able to measure the torque or the solid fraction here. Instead the comparison here requires the solid fraction obtained from numerical simulations of the same geometry, and the vertical axis is scaled arbitrarily. However, the results indicate that if we can obtain simultaneous measurements of the torque [5] and solid fraction, it should be possible to test this model with unprecedented accuracy.

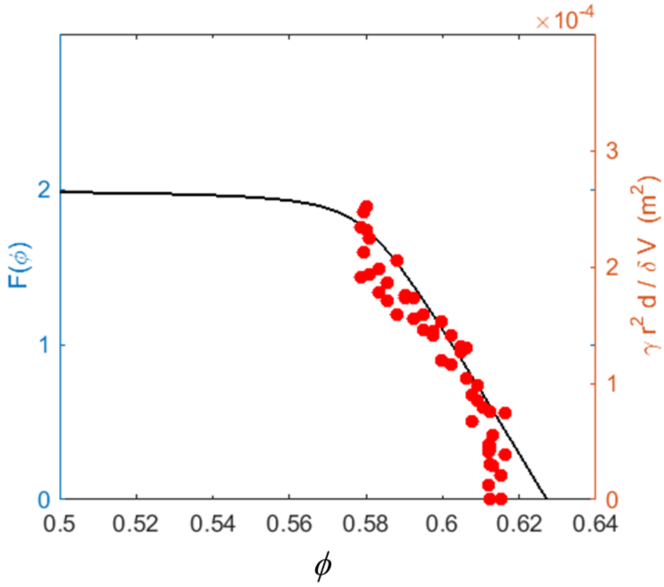

Figure 1: Comparison of granular fluidity calculated from the experimental data and F(ϕ) given by Zhang and Kamrin [2].

References

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[2] Q. Zhang and K. Kamrin, Phys. Rev. Lett. **118**, 1 (2017).

[3] O. Pouliquen and Y. Forterre, Philos. Trans. R. Soc. London A Math. Phys. Eng. Sci. **367**, 5091 (2009).

[4] D. J. Holland, et al., Ind. Eng. Chem. Res. **48**, 172 (2009).

[5] T. I. Brox, New Methods for Studying Materials Under Shear with Nuclear Magnetic Resonance by, Victoria University of Wellington, 2016.

Constructing local flow curves of complex yield stress fluids based on Rheo-MRI velocity profiles

Tatiana Nikolaeva[1], Frank Vergeldt[1], Paul Venema[2], Henk Van As[1], John van Duynhoven[1,3]

[1] *Laboratory of Biophysics, Wageningen University, Wageningen, The Netherlands*
[2] *Physics and Physical Chemistry of Foods, Wageningen University, Wageningen, The Netherlands*
[3] *Unilever Discover Vlaardingen, Vlaardingen, The Netherlands*

Rheo–MRI is a non-intrusive technique that allows a direct and quantitative view on local flow behaviour of complex fluids [1, 2]. Localized rheo-MRI velocimetry is especially helpful for studying flow behaviour of dispersions that display yield stress behaviour, since their flow behaviour cannot be understood from conventional rheological measurements. Of particular interest are dispersions that display shear banding, i.e. co-existing flowing and stationary regions. In our work we have addressed two bottlenecks for fully exploiting the potential of rheo-MRI for studying such systems: heterogeneity of stress in the flowing dispersion and chemical heterogeneity which can lead to chemical shift artefacts.

The Couette geometry, which consists of concentric cylinders with a gap in between them, is most commonly used in rheo-MRI. Although its stress distribution over the gap is well known it has hardly been exploited. The combination of local shear rates (deduced from rheo-MRI velocity profiles), and local shear stress values (derived from the applied torque) allow to construct a local flow curve [3]. Since such a local flow curve can be obtained from a snapshot rheo-MRI velocimetry experiment, it offers unique opportunities to assess transient flow behaviour of dispersions. We constructed a set of Couette geometries with variation of gap sizes (1, 2.5 and 4 mm) that can be mounted in a 300 MHz micro-MRI system and in a conventional rheometer. Several hitherto neglected practical and theoretical aspects of obtaining local flow curves will be discussed.

For chemically heterogeneous dispersions such as oil-in-water emulsions the presence of distinct chemical shift can give artefacts to compromised velocimetry profiles. We have explored the use of chemical shift selective pulses for obtaining clean velocimetry profiles.

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Investigation of the influence of fluid dynamics on polymerization kinetics via Rheo-NMR spectroscopy

E. Laryea[1], M. Schäfer[1*], N. Schork[1*], N. Schuhardt[1*], V. Lehmann[2], T. Oerther[2], D. Groß[2], G. Guthausen[1], M. Kind[1]*
[1]Karlsruhe Institute of Technology, TVT, MVM, EBI Karlsruhe, Germany,
[2]Bruker Biospin GmbH, Rheinstetten, Germany,
[*]contributed equally

Polymers have a fundamental impact on the everyday life. Therefore, the study of new production processes and their improvement and optimization are in the focus of research. To model polymerization processes and predict the characteristics of the produced polymer, a fundamental knowledge of the polymerization kinetics and their influencing parameters is crucial.

For this purpose, the influence of the fluid dynamics on polymerization kinetics of different free radical batch polymerizations were observed, using Rheo-NMR technology. The polymerization of methyl methacrylate (MMA) and styrene with p-xylene as a solvent and the initiator 2,2'-azobis(2-methylpropionitrile) (AIBN) were chosen. A Taylor-Couette reactor (TCR) was used, due to the well-known and well-adjustable fluid dynamics. This type of reactor consists of two concentric cylinders, where the inner cylinder is rotating and the outer one is at rest. The reactor was designed to fit in a standard MICWB40 probe of a wide bore NMR spectrometer. Particular care is taken for temperature control of the reacting solution.

A variation of the frequency of the rotation of the inner cylinder leads to different flow regimes. *In situ* experiments with three different shear rates ($\dot{\gamma} = 24\ s^{-1}$, $\dot{\gamma} =\ 128\ s^{-1}$, $\dot{\gamma} = 534\ s^{-1}$) at $T = 80°C$ have been executed with a time resolution of at least one spectrum per minute to monitor the reaction rate and other reaction characteristics. During the reaction, also the viscosity of the reacting solution increases during the polymerization influencing the fluid dynamics.

In situ Rheo-NMR (^{1}H)-spectroscopy was performed to observe the relation between fluid dynamics and spectroscopic information over the course of polymerization. In particular the rate of monomer depletion and the rate of polymer production could be detected under the various imposed shear conditions. In summary, Rheo-NMR spectroscopy has proven as a non-invasive and non-destructive method to observe the polymerization under shear and to gain in-depth insight to the factors influencing the polymerization kinetics.

Transient rheology from a structural perspective

Simon Rogers

One of the core tenets of rheology is that the structure of complex fluids on the smallest scale gives rise to their macro-scale flow properties. While the rheology of soft materials has been studied for nearly a century now, it is only in the last two decades that we have developed tools that allow us to interrogate the required length scales. With the development of experimental hybrid rheo-scattering and rheo-NMR techniques, we have gained a new structural perspective that has informed the analysis methods we use to understand transient rheological tests. In this presentation, I will focus on the 'rheo' side of the hybrid rheo-X techniques, showing examples from rheo-scattering studies on soft materials with a particular focus on how we gain understanding from the experiments we perform.

q-space PFG-NMR methods to characterise non-Newtonian fluids

T.W. Blythe, A.J. Sederman, E.H. Stitt, A.P.E. York, L.F. Gladden

Complex fluid rheology plays an important role in many industrial processes; from baking to catalyst preparation. The rheological characterisation of process fluids is typically performed using bench-top rheometers operating in an offline configuration. An alternative technique is therefore sought for real-time online characterisation, as is often required for process control and optimisation. Super-conducting NMR magnets are clearly not practical for industrial online measurements, however developments in lower field strength, relatively inexpensive permanent magnet technology, now make NMR an attractive technique; it is non-invasive, flow sensitive and without limitation on optical opacity.

This work describes the development of acquisition and processing protocols to enable the rheological characterisation of non-Newtonian fluids under pipe flow conditions at low signal-to-noise (SNR) and in short acquisition times. Rheological characterisation can be done by acquiring a full MRI flow image but the lower SNR obtained in low field magnets prohibits the use of MRI flow imaging in reasonable acquisition times. To this end, an alternative approach using PFG NMR is described which exploits the sensitivity of the flow propagator, and therefore corresponding PFG NMR signal, to changes in the rheology of the fluid under study. In particular, a Bayesian analysis of the acquired signal is used to provide an estimate of the rheological parameters of Herschel-Bulkley fluids through comparison with PFG NMR signal data obtained using numerical simulations. This approach offers significant undersampling, when compared to the acquisition of a full flow propagator, and demonstrates an exceptional robustness to low SNR where even with an SNR of 100, only 16 points need to be sampled to give results accurate to within 2% of the ground truth. For validation, an experimental study on a model Herschel-Bulkley fluid (Carbopol 940-in-water solution) at high field is described. Results indicate that accurate estimates of the rheological parameters can be obtained in up to 88% less time than those obtained using conventional MRI flow imaging. The need for online, or inline, rheological characterisation is highlighted with comparison to bench-top rheological measurements.

Using MRI to Understand the Interplay Between Molecular Interactions and Flow in Complex Molecular Systems

Melanie M. Britton

School of Chemistry, University of Birmingham, UK

Colloidal suspensions are ubiquitous in nature and many man-made materials, such as household and personal care products, food science, advanced materials and electrochemical technologies. The stability and properties of these suspensions are determined by the interactions between particles and their propensity to aggregate or disperse within the continuous phase. In this talk, an investigation, by NMR and rheoNMR, of the stability and rheology of silica particle suspensions, in different ionic liquids, will be presented. These systems are of increasing interest in solar cells, batteries and other electrochemical applications, for which a better understanding of the molecular behaviour which underpins their rheological properties is essential and can be uniquely probed by rheoNMR.

Dalhousie’s Rheo-NMR contributions to soft matter knowledge in fats and oils

Gianfranco Mazzanti, Ph.D.

Crystallization of triacylglycerols impacts the texture quality of chocolate, the success of baking shortening formulations, the clogging of biodiesel fuel lines, and many health issues such as cardiovascular diseases. The polymorphic forms and the mechanical, thermal and diffusional characteristics of the materials are modified dramatically by the application of shear fiels.

Over the past 10 years we have adapted Low-field benchtop NMR instruments to perform rheo-NMR measurements, chiefly by combining an Anton-Paar research rheometer head with 20 MHz Bruker units. Design experience has been transferred to labs in other parts of the world.

Additionally, we have continued our work on Rheo-XRD both in house and at the synchrotrons (NSLS I, APS, Tsukuba). We have also mentored the design, construction and operation of several Rheo-XRD devices by other teams.

We continue the development of the Rheo-XRD+NMR instrument. A first test at APS (Argonne, IL) synchrotron has given us lots of useful information.

The experience has made us strong at:

- Designing better heat transfer systems for confined spaces
- Determining solid content
- Determining intrinsic temperatures

This talk will present a few salient examples of the results obtained for the fats and oils fields, as well as the technical developments that needed to be solved to reach such results. The most important aspect remains the formation of qualified individuals.

(Reacting) Hydrate Slurry Rheology

Michael Johns

Faculty of Engineering and Mathematical Sciences, The University of Western Australia, Australia;

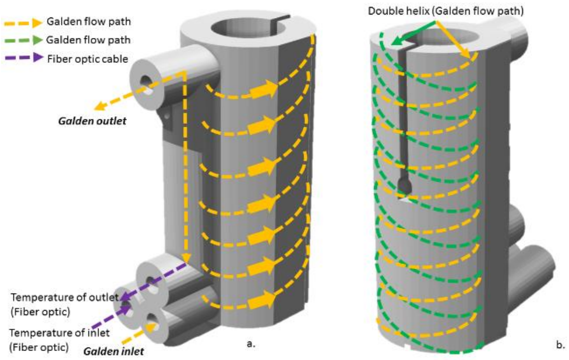
Methane hydrates in pipelines are increasingly being tolerated as oil and gas fields explore deeper reserves. Critical to risk assessment of such tolerance is the viscosity of hydrate suspensions. Here we will present preliminary results detailed the rheology of such hydrate systems, formed from water-in-oil emulsions subjected to high pressure methane, using a vane rheometer. The use of rheo-NMR will be briefly detailed in terms of an exploration of the secondary flows (necessary for methane dissolution) present in the vane rheometer. Finally the development of a 12 litre cyclopentane hydrate suspension flow loop, along with some preliminary results, will be detailed.

Design and use of a 3D printed thermal coil for an NMR probe

Tianguang Jia, Yijin Su, Gianfranco Mazzanti
Food Science, Dalhousie University

Abstract:

The combination of rheometer and NMR system required modifications of commercial instruments. One needs to control the temperature of the operating fluid in a convenient, fast and non-disruptive way. A customized heat exchanger was designed within the constraints of no-metals, as little hydrogen as possible, fast flow speed, and reduced space in the NMR probe (32 mm gap). The concept was developed in Solidworks (software) as shown in Fig 1, to be later manufactured in a 3D printer.



This exchanger is a broad-wall tube with helical inner channels. The inner wall is very thin to provide good heat transfer to the sample. This also places minimal number of hydrogen atoms of the resin near the coil. The external wall is thicker, providing insulation to the outer environment of the probe.

Galden 135 (Solvay-Solexis) fluid, a perfluorinated 'hydrocarbon' without hydrogen, was selected as the operating fluid. It enters the coil from the bottom inlet (yellow dash line) and leaves from the top outlet (as shown in Fig 1 (a), only one helix is drawn for simplicity). The Galden flow path is designed elaborately as a double helix (green and yellow dash line in Fig 1 (b)). The temperatures of inlet and outlet are monitored by fiber optic sensors at alternate orifices (purple dashed line, Fig 1 (a)). The fiber optic sensors are non-metallic, and thus do not interfere with the radio frequency or electronics of the probe.

3D printing was chosen because it was extremely hard to craft this exchanger using common machining methods, due to its complex inner structures of relatively small size. After trying several manufacturers and several designs, the heat exchanger was made by Formlabs (Somerville, Massachusetts, USA) of an anonymous "resin" material that is impermeable under experimental pressure. With this customized heat exchanger, the temperature around the sample can be controlled and changed rapidly, without affecting the temperature of the magnet. This is necessary for Rheo-NMR experiments with the Bruker mq20, because the field of the magnet is calibrated by keeping it at 40.00 °C.

USE OF THE SPIN-SPIN RELAXATION TIME TO ESTIMATE THE TEMPERATURE OF A STANDARD NEWTONIAN OIL UNDER SHEAR FLOW

Yijin Su, Tianguang Jia, Gianfranco Mazzanti
Chemical Engineering, Dalhousie University

ABSTRACT

This work is the first step of a new approach to estimate the temperature of complex fluids under shear, in the narrow gap of our Rheo-NMR cell. We usually study time changing suspensions such as crystallizing paraffin wax or triglyceride blends. The sample temperature, raised above that of the cooling fluid due to viscous heat, is not known. The spin-spin relaxation time T_2, measured in NMR experiments on liquids, is affected by the sample temperature. Experiments to quantify this effect were first conducted under static conditions, in a mini-Couette system using a standard Newtonian oil. T_2 was measured using a CPMG sequence (by Bruker) for the 20 MHz modified mq20 benchtop instrument. The temperatures explored ranged from 0 to 40 °C. Two characteristic T_2 values were found to describe the echo decay. The values of T_{2_1} ranged from 1.7 to 4.4 ms, T_{2_2} from 6.4 to 23.0 ms. The ratio of amplitude of T_{2_2} over total amplitude, A2/(A2+A1), ranged roughly from 0.3 to 0.6.

Rheo-NMR experiments were then conducted at rotational speeds of 0.1, 1, 10, 30, and 50 revolutions per second. The temperature of the medium was set between 20 and 40 °C. The correlations between sample temperature and T_2 obtained from the static experiments were applied to predict the sample temperature, using the T_2 measured under shear flow. The temperature increased at high shear rates, as was expected; whereas, no significant increase was observed at low shear rates.

Exploring Large Amplitude Oscillatory Shear (LAOS) Rheology by Rheo-NMR

Jayesha S. Jayaratne[1], Rehab N. Al-Kaby[2], Sarah L. Codd[2], Timothy I. Brox[3], Petrik Galvosas[3] and Joseph D. Seymour[1]

1. Department of Chemical and Biological Engineering, Montana State University, Bozeman, MT, USA

2. Department of Mechanical and Industrial Engineering, Montana State University, Bozeman, MT, USA

3. School of Chemical and Physical Sciences, Victoria University of Wellington, Wellington,

New Zealand

Abstract

We explore the use of MRI velocimetry based Rheo-NMR to complement large amplitude oscillatory shear (LAOS) rheometry. Shear rate spatial distributions as a function of the period of oscillation and oscillatory velocity at fixed positions across the gap in a cylindrical Couette cell for fluids ranging from Newtonian to yield stress are presented. Given the novelty of the data, the ways in which it can characterize the rheology are considered.

Planar Couette flow for magnetic resonance microscopy

S. J. Stevenson[a], T.I. Brox[a], P Galvosas[a,b]

[a] Victoria University of Wellington, SCPS, New Zealand;
[b] MacDiarmid Institute for Advanced Materials and Nanotechnology, New Zealand;

NMR is an established technique which has been used for almost three decades to study these responses by combining methodology from rheometry and nuclear magnetic resonance [1-3]. It enhances standard rheological studies of bulk properties, such as viscosity and elasticity, by exploring the molecular origins and / or local responses within a material.

This contribution reports on the development and application of a novel geometry for magnetic resonance microscopy (MRM) – a planar-cylindrical hybrid (PCH) shear cell – to study fluid mechanics and the complex response of materials under shear. The geometry includes sections of planar Couette flow with the aim to provide a simple homogeneous shear profile. Various geometries to establish planar Couette flow have been used previously to study the fluid mechanics of simple fluids [4-6] but have never been developed for NMR. Generally, they are composed of two parallel sections of planar flow connected by two semicircular sections of circular flow to give a closed flow path in the shape of a racetrack. Shear is applied by rotating a band around the inner section like a conveyor belt. This is another step in recent efforts to enrich the Rheo-NMR toolbox by removing the inhomogeneous flow field conditions as generated by the concentric cylinder / Taylor-Couette (TC) geometries [7] (for which the narrow gap approximation is harder to satisfy as compared to in conventional rheometry).

Previous work on one shear banding wormlike micelle (WLM) solution demonstrated that the curvature of TC cells used during Rheo-NMR experiments influenced the observed rheological response [8]. This work further investigated the influence of curvature by studying the local dynamics of the WLM solution using the PCH in combination with a variant of the RARE pulse sequence [9]. Commonalities and differences of the WLM solution in the PHC when compared to simple fluid systems (e.g. silicone oil) and TC geometries will also be discussed.

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Rheo-NMR and Rheo-SALS using Large Amplitude Oscillatory Shear for the Study of Complex Fluids

S. Kuczera[a,b], L. Gentile[a,c], U. Olsson[a], T.I. Brox[b], C. Schmidt[d], P Galvosas[b,e;]

[a] Division of Physical Chemistry, Lund University, Sweden;
[b] Victoria University of Wellington, SCPS, New Zealand;
[c] Department of Chemistry and Chemical Technologies, University of Calabria, Italy
[d] Department of Chemistry, University of Paderborn, Germany;
[e] MacDiarmid Institute for Advanced Materials and Nanotechnology, New Zealand;

Rheo-NMR has existed as a tool for studying fluids under mechanical deformation for nearly 25 years [1,2]. It provides spatially and temporally resolved maps of NMR spectra, intrinsic NMR parameters (e.g. relaxation times) or motion (e.g. diffusion or flow). Likewise, Rheo-SALS (Small Angle Light Scattering) is an advanced rheological method which can be used to monitor microscopic changes in the sample due to deformation. Characteristic scattering patterns arise from scattered light intensity as a function of the wave vector, yielding information on microscopic shapes and structures [3]. Both, Rheo-NMR and Rheo-SALS have been established as complementary techniques to conventional rheological measurements.

Shear-induced transformations between oriented planar lamellae and multilamellar vesicles (MLVs) due to steady shear have been studied in the past by Rheo-NMR [4,5]. However, recent advances in Rheo-NMR hardware allow for the application of Large Amplitude Oscillatory Shear (LAOS) deformations in high field NMR magnets for the first time [6]. Here we report on the combined use of Rheo-NMR and Rheo-SALS to study the formation of multilamellar vesicles (MLVs) in a lyotropic surfactant system ($C_{10}E_3$/water) using LAOS. For the range of investigated strain amplitudes (10-50) and frequencies (1 rad/s, 2 rad/s) MLV formation is observed in all NMR and most SALS cases. For LAOS it was found that the MLV size mainly depends on the frequency as opposed to previous steady shear experiments where the shear rate was the controlling parameter [7]. Additionally, the onset of MLV formation using LAOS was primarily dependent on the applied shear amplitude. Furthermore, the process of onion formation appears to be retarded during LAOS as compared to the steady shear case.

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