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1 Executive Summary

This project has helped to increase the awareness of exactly how difficult it is to control and determine the fluid mechanical properties of everyday fluids such as ketchup as well as highly customised and engineered fluids such as drilling mud. The project has prompted several industrial organisations to redirect and focus their efforts to design measurement methods geared towards determining the viscosity, density and solids content of such fluids.

The Problem

Traceable metrology of the fluid mechanical properties of complex fluids, i.e. fluids whose properties depend on the state, duration and history of their present and past exposure to stress, remains an elusive goal.

Neither certified reference fluids nor traceable instrumentation currently exist. Formulating reference fluids with acceptable shelf lives and well defined properties is also hampered by the contrasting requirements of cost, the simplicity of recipes, the quantities required and in the case of solid inclusions the specification thereof. Existing commercial instrumentation (rotational rheometers) incorporate exhaustive motor controls which have so far resisted attempts to ensure traceability of the basic measured quantities of torque, rotational speed and temperature.

The Solution

In response to this challenge the project aimed to develop a set of four reference fluids, to model the fluid mechanical behaviour of complex fluids in rotational rheometers and to establish a traceability chain extending to field instrumentation for use on drilling rigs. The objectives were to:

- Formulate and certify a set of four reference fluids with the desired complex behaviour which may serve as transfer standards.
- Calibrate, model and analyse the fluid mechanics of complex fluids with and without solid content in rotational rheometers by both experimental, numerical and theoretical means.
- Evaluate a range of commercially available field instrumentation under conditions representative of real world drilling rigs and establish their absolute accuracy and repeatability.
- To set forth the findings in recommendations for new standards for the use of field instrumentation such as with the American Petroleum Institute (API), International Organization for Standardisation (ISO), Norsk Sokkels Konkuranseposisjon (NORSOK i.e. standards developed by the Norwegian Technology Centre) and other standardisation bodies.

Extensive use was made of the real world drilling facilities and the expertise available at the International Research Institute Stavanger (IRIS) as part of a researcher excellence grant (REG).

The Impact

Results obtained represent mostly an awareness of the fact that fluids which change when you touch them or alternatively change when left alone to themselves are an entirely different class of materials for metrology. Silly putty may be “kid’s play” but it is no small task to define reproducible, traceable metrology for it.

- The ring comparison of the candidate reference fluids helped foster the insight that complex fluids are much more difficult to produce reproducibly than ordinary fluids. The formulation, the recipe, the raw materials as well as handling and storage procedures require attention beyond the customary.
- Modern rotational rheometry systems incorporate extensive features to handle and manipulate complex fluids. It appeared that such features hamper the efforts to calibrate the instruments in a traceable way. Therefore, static definitions of torque and rotational speed should be replaced with dynamic ones.
- Extending the traceability beyond the metrology lab into the real world of drilling rigs or industrial plants requires not only well specified reference fluids but also cheap and easy to produce fluids.

In conclusion, the metrology of complex, so called non-Newtonian fluids requires first and foremost a step back and a reflection on common metrological approaches.

2 Project context, rationale and objectives

Overview

Substances which behave in a complex manner when exposed to mechanical shear are abundant in everyday life. They include engineered and naturally occurring substances such as paint, concrete, drilling fluids, toothpaste, peanut butter, honey, blood, mud and natural latex rubber. Such complex substances are collectively known as 'non-Newtonian', which is a reference to their contrasting behaviour to 'Newtonian' fluids such as air and water which have simple behaviour (that was well understood in the days of Sir Isaac Newton).

Typical non-Newtonian fluids contain a large amount of solids and have a viscosity that changes with the shear rate. For example, a good fresh paint holds the solids in place and does not flow due to gravity, but it should flow to some extent, while painting a wall.

Accurate, traceable, metrology for the complex behaviour of non-Newtonian substances remains an elusive goal, and this lack of metrology turns the engineering of such substances for particular uses into more of an art than a science.

This project aimed to address this issues by developing a traceable calibration method for the measurement of the viscosity of non-Newtonian fluids, including a set of calibration fluids in the form of Certified Reference Materials (CRMs). However, due to difficulties in reproducibly formulating the calibration fluids the project was unable to produce a set of CRMs. This also impacted the project's testing of calibration methods for the inline measurement of viscosity (i.e. direct measurements taken using sensors or instruments that are situated within a flow system). Four commercially available inline instruments for the measurement of viscosity were tested under conditions simulating the operation of a drilling rig, and using the project's calibration fluids. However, due to significant temperature swings, the viscosity of the fluid deviated by an order of magnitude, and the results of the comparison were severely impaired.

Need for the project

The property that describes the behaviour of substances under mechanical shear is viscosity. Currently, a variety of techniques are commercially available for the measurement of viscosity of fluids. However; only one technique, capillary flow, has SI traceability and it is only for Newtonian fluids. All other types of instrumentation are calibrated using Newtonian standard reference fluids that are available in a restricted range of viscosities and within a defined temperature range.

For non-Newtonian fluids, no dedicated instrumentation is available for the measurement of viscosity. Instead current industry practice calls for the use of instruments for Newtonian fluids and overlooks the lack of traceability.

The overall goal of this project was therefore to address this need and close the gap between the well-established and traceable techniques available for Newtonian fluids and the lack of traceable instrumentation and standard reference materials for non-Newtonian fluids. In particular there is a need to develop a calibration and measurement capability for non-Newtonian (reference) fluids and guidelines for calibrating instrumentation used for measuring the viscosity of non-Newtonian fluids. There is also a shortage of industrial inline, automated instrumentation for non-Newtonian fluids that exhibit the full range of complex behaviour for example drilling fluids used in oil or gas wells.

Currently, the greatest need for traceable, accurate non-Newtonian inline viscosity measurements is in the in oil and gas industries. The impact of accurate, traceable, metrology for non-Newtonian viscosity measurement techniques would support: improvements in operational efficiency; the construction of more complex wells to develop oil or gas reservoirs that are harder to reach; as well as the automation of drilling rigs.

Scientific and technical objectives

The aim of this project was to develop a traceable calibration method for the measurement of the viscosity of non-Newtonian fluids. A set of calibration fluids in the form of Certified Reference Materials (CRMs) exhibiting the desired complex behaviour when exposed to shear stress were to be developed. Existing instrumentation was also to be used to study the complex behaviour of non-Newtonian fluids and this was to be augmented with numerical models. Collectively, this should have led to guidelines and CRMs for the calibration and use of existing or new inline field instrumentation.

The project had the following objectives:

1. To develop a set of calibration standards in the form of CRMs for complex fluids in a range of viscosities, densities and temperatures that are of interest for stakeholders.
2. To study the physical behaviour of complex fluids using existing viscometer techniques and rheometers. To do this, current models of the relation between shear rate and viscosity will be compared to experimental results.
3. To focus on sensors and calibration methods for inline measurement of viscosity, density and solids content. This will include:
   - Identification of existing and alternative non-Newtonian viscosity, density, and solids content measurement methods for in line operational conditions.
   - Selection of suitable sensors for viscosity, density, particle size distribution and solids content for full scale testing and define
   - Testing of the selected sensors at full scale under operational conditions and including relevant CRMs

3 Research results

3.1 Development of a set of calibration standards in the form of CRMs for complex fluids in a range of viscosities, densities and temperatures

Objective

The aim of this work was to develop a set of calibration standards in the form of CRMs for complex fluids in a range of viscosities, densities and temperatures that are of interest for stakeholders.

Results

VSL and PTB developed four candidate non-Newtonian liquid (NNL) CRMs, one exhibiting Newtonian behaviour (NNL1) as a reference base case, one geared towards the desired behaviour for drilling operations (NNL2), one to test rotational rheometers with visco-elastic characteristics (NNL3) and one with near Newtonian fluid properties (NNL4) to evaluate the resolution of rheometers. Recipes for the formulation of the four candidate CRMs as well as an inter-comparison protocol were produced, shared with and used by all project partners. However, large deviations occurred in the result due to difficulties in reproducibly formulating the calibration fluids (using the recipe). The protocol could not be closely adhered to by any party and there were differences between the instruments used. For example for NNL2 at one specific rate of shear the resulting uncertainty in the viscosity was estimated to be 20 % which is 4 times higher than the target uncertainty of 5 % and 10 times higher than that required by the Norwegian standard for drilling operations (NORSOK).

Conclusions

The project was unable to reproducibly produce a set of validated calibration standards in the form of CRMs for complex, non-Newtonian fluids. External to this project, attempts by the National Institute of Science and Technology (NIST) in the USA to formulate a Standard Reference Material (SRM) for concrete have been plagued by similar issues with reproducibility over the course of at least 7 years. Inadequate definition of the formulation and preparation method is believed to be the most significant cause of this.

3.2 Study of the physical behaviour of complex fluids using existing viscometer techniques and rheometers.

Objective

To study the physical behaviour of complex fluids using existing viscometer techniques and rheometers. To do this, current models of the relation between shear rate and viscosity will be compared to experimental results.

Results

At project partners VSL, PTB, METAS, IPQ, INRIM and CNAM new, advanced, rotational rheometers were purchased and added to their previously available (capillary flow) instrumentation for measuring the viscosity of Newtonian fluids. A rheometer measures the way in which a liquid flows in response to applied stresses, and these new rotational rheometers measure torque and (rotational) speed and can operate in such a way that either of these is maintained at a programmable constant value.

For non-Newtonian fluids, where viscosity may change with the applied mechanical shear, knowledge of the duration of the shear, its rate and direction of change (i.e. either increasing or decreasing) is vital. Modern electronically commuted motors under software and firmware control can be used for this. However, traceable calibration to the SI base units of torque, speed and temperature measured using such advanced techniques remains a challenge.

IMBiH developed a Computational Fluid Dynamics (CFD) model for a rotational rheometer using two different arrangements of the rotor and stator and for non-Newtonian fluids with or without added particulate matter. The CFD model helped to evaluate the limits of operation of rotational rheometers.. In particular, the onset of instability in fluid motion inside the measurement geometry was studied.

As part of a researcher exchange program between IPQ and PTB (receiving host) the influence of viscosity on density measurement devices using vibrating parts was successfully quantified. In addition, a method for using laser doppler velocimetry to measure the flow of translucent non-Newtonian fluids was developed.

Project partner, INRIM investigated the use of pycnometry for density measurements of non-Newtonian fluids. CNAM and METAS also investigated the effect of particulate material on the viscosity of a (non-) Newtonian carrier fluid.

Conclusions

Despite the learnings and insight gained into the workings of modern, advanced rotational rheometers attempts to make even one of such an instrument traceable for the basic measured quantities of torque, rotational speed and temperature failed. None of the participating NMIs were able to add a new Certified Measurement Capability to their existing capabilities.

3.3 Focus on sensors and calibration methods for inline measurement of viscosity, density and solids content.

Objective

To focus on sensors and calibration methods for inline measurement of viscosity, density and solids content. This will include:

- Identification of existing and alternative non-Newtonian viscosity, density, and solids content measurement methods for in line operational conditions.
- Selection of suitable sensors for viscosity, density, particle size distribution and solids content for full scale testing and define
- Testing of the selected sensors at full scale under operational conditions and including relevant CRMs

Results

A researcher grant at IRIS evaluated four commercially available inline instruments for the measurement of viscosity under conditions simulating the operation of a drilling rig. For this evaluation, IRIS produced non-Newtonian liquids using the recipes for the candidate CRMs from objective 1/section 3.1. However, during the preparation of the fluids the recipe for the production of the second candidate CRM (NNL2) could not be followed because of limited heating capacity of the mixing equipment at IRIS. During the evaluations heat dissipation resulting from pressure loss in the recirculating loop resulted in temperature swings of more than 2 ºC which caused the viscosity of the fluid to change by an order of magnitude. As a result, the comparison of the four inline instruments was severely impaired. In addition, it was not possible to compare the instrument's viscosity measurements with those determined by a rotational rheometer in a laboratory as the candidate CRMs (non-Newtonian fluids) used in each exhibited very different characteristics.

Conclusions

Validation of the field instrumentation in use on the drilling rig was hampered by the impracticality to run the rig with large quantities of the candidate CRM NNL2. A more cost effective replacement was substituted but required heating to temperatures above the capability of the rig's fluids handling systems.

IRIS was unable to evaluate an instrument for solids content and/or particle size distribution as the manufacturer of the candidate instrument withdrew their support to the project. IRIS did, however evaluate a standard straight tube density meter (Coriolis flow), but the absolute accuracy of the Coriolis flow meter could not be determined due to the issue IRIS had with preparing the NNL2 candidate CRM (as outlined above).

4 Actual and potential impact

The project has produced 2 publications for the International Journal of Metrology and Quality Engineering and the journal of Applied Rheology. The project website has been used to disseminate information to stakeholders and end-users. The project has also been presented at 7 conferences such as the International Congress of Metrology, the 4th European Flow Measurements Workshop 2016 and the Annual European Rheology Conference (AERC) 2017. In addition, the project was presented to the BIPM consultative committee for mass (CCM) and to the Society of Petroleum Engineers, the Committee for Drilling System Automation Technical Section chapter C on Sensors, Instrumentation and Measurement systems.

The complexity of quantitatively understanding the behaviour of non-Newtonian fluids was communicated by the project to industrial stakeholders. Awareness of this prompted a number of parties from industry and academia to join the project as collaborators, such as IFPEN (The French Institute of Petroleum), the University of Texas and Aspect Imaging (a world leader in the design and manufacture of imaging systems for medical and industrial applications). It is anticipated that such organisations will continue with the efforts to develop reference fluids that exhibit non-Newtonian characteristics and to develop measurement techniques for rheometry.

Currently and external to this this project, new instrumentation for the inline measurement of viscosity of real world fluids, i.e. opaque fluids laden with particulate material exhibiting non-Newtonian temperature dependent behaviour, are being developed and introduced. A pulsed ultrasound velocity profile meter developed for food industry applications has recently become a candidate for drilling rig applications and magnetic resonance imaging velocimetry is being adapted to fit practical field requirements. In the future, whirling tube type instruments may well also be used for the evaluation of non-Newtonian fluids but currently such instruments suffer from unacceptable temperature sensitivity. In the meantime there are still no dedicated and traceable instruments for the measurement of viscosity for non-Newtonian fluids.

It is now recognised that the technical solutions currently used with commercial rotational rheometers for the evaluation of non-Newtonian fluids, are significantly impacting the effort required to traceably calibrate such instruments. Therefore, a recipe is urgently needed to reproducibly produce non-Newtonian reference materials for the calibration of such instruments. This will require strict controls and adherence to protocols and should build upon the lessons learnt within this project.

5 Website address and contact details

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6 List of publications

[1]. P. Ballereau, D. Truong, A. Matias Absolute falling ball viscometer adapted to low viscosities of liquids, International Journal of Metrology and Quality Engineering, **Approved, awaiting publication**

[2]. E. Borovac, M. Torlak, R. Pagel, P. Ballereau Combined experimental and computational analysis for flow stability and non-linear effects in coaxial cylinder rheometers, Applied Rheology **Declined**