Final Publishable JRP Summary Report for ENG59 NNL Sensor development and calibration method for inline detection of viscosity and solids content of non-Newtonian liquids

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.

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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 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 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

Results & Conclusions

Development of a set of calibration standards in the form of CRMs for complex fluids in the range of viscosities, densities and temperatures that are of interest for stakeholders.

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).

Thus 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.

Study of 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.

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 forces, 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.

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

o Identification of existing and alternative non-Newtonian viscosity, density, and solids content measurement methods for in line operational conditions.

o Selection of suitable sensors for viscosity, density, particle size distribution and solids content for full scale testing and define

o Testing of the selected sensors at full scale under operational conditions and including relevant CRMs

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. 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 ^{o}C 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.

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).

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.

JRP start date and duration:	May 1, 2014 - 36 Months
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