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Development and calibration of a high pressure high shear rate capillary rheometer

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ABSTRACT

For the characterization of the viscosity and the flow behavior of heavy and extra heavy oil, a high pressure, high shear rate capillary rheometer has been developed. Due to the commercial rise in the exploitation of heavy and extra heavy oils, non-Newtonian flow behavior becomes relevant. Information on the flow properties of these heavy oils, particularly at high pressure and high shear rates is essential for recovery and processing methods. The capillary rheometer consists of two high-pressure vessels connected by a tube system including a calibrated capillary. Each vessel is equipped with a hydraulically operated metal bellow that can be varied in size to make oil flow from one vessel, through a capillary, into the other one at a controlled rate of flow. From the measured pressure drop the viscosity of Newtonian fluids is calculated with Hagen-Poiseuille's law. The capillary rheometer is designed for ambient operating conditions up to 1000 bar and 200 °C. Fluids with a viscosity between 1 and 100,000 mPas can be measured under high shear rates. The maximum possible shear rate depends on the viscosity of the fluid. For fluids with viscosities under 4000 mPas, shear rates up to 50,000 1/s are possible. Viscosities up to 100,000 mPas still allow shear rates of 4000 1/s. Before being set into operation, the apparatus is calibrated in a range up to 500 bar with viscosity standards that have been independently measured to high-pressure conditions. In this range, the apparatus works with an overall accuracy of about 3%.

1. Introduction

During the last quarter of the 20th century, the global need for crude oil showed a stable annual growth rate of about 1% in average (Speight, 2013). As a result of the economic development and the demographic growth in the emerging economies and the increasing consumption of energy in industrialized countries, this demand has visibly increased during the first years of the 21st century (OPEC World Oil Outlook, 2013a). Currently the world's demand for energy increases by 8% each year (Agency, 2014). Different international studies foresee that in the next 20 years at least 80% of the world energy supply will come from raw fossil fuel materials (Nakićenović et al., 1988; OPEC World Oil Outlook, 2013b; Trenberth et al., 2009). Accordingly, oil will be the most important source of energy for the next decade. Heavy oil represents at least half of the recoverable oil resources of the world (Chopra et al., 2010). As the available reserves are limited, effective recovery processes gain in importance (Meyer et al., 2007). It is estimated that there are about 6 trillion barrels of heavy oil reserves, six times the conventional amount (Bitumen and heavy crudes, 2006). Rheological properties such as the dynamic viscosity as a function of temperature, pressure and shear conditions are vital for Enhanced Oil Recovery (EOR) as well as for transportation and processing of heavy oils. During production and refining, the oil is subject to large variations in temperature resulting in cuts that go form light fuels to asphalt residues which lead to variations in viscosity of orders of magnitude. Nevertheless, the flow behavior of heavy and extra heavy mineral oils is barely investigated. Usually light petroleum fluids tend to behave Newtonian while heavy oils show non-Newtonian behavior (Soto et al., 2015). Under reservoir conditions, which can be in the range of several hundred bars and temperatures around 100 °C, it is found that the non-Newtonian behavior can be enhanced, particularly if the oil is subject to gas separation during production.

Measuring these properties with conventional devices is not a simple task. Rotational rheometers work with open geometries such as bob and cup or parallel plates. High shear rates cannot be generated because the fluid may destabilize or spill out. There are some high-pressure devices for rheometric measurements, but many of them have magnetic couplings, which may decuple before viscosity thinning is observed. If gas is

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used to pressurize the rheometers, it may dissolve into the oil and cause non-negligible changes in viscosity. With a falling body viscometer good results can be achieved, but the time required is considerable, especially for highly viscous fluids, and rheological properties cannot be determined directly.

Although at an incipient stage, some high-pressure capillary rheometers have been described by different authors. A capillary viscometer is developed by Galvin et al. (1981) to determine the tribological properties of lubricants in order to analyze the lubricating behavior between rollers, bearings and other machine parts at pressures up to 2000 bar, temperatures between 20 and 100 °C and shear rates reaching from 0.1 up to $2{\cdot}10^5$ 1/s. The viscosity range of this apparatus reaches from 0.1 to 1000 mPas. A high-temperature, high-pressure rheometer has been designed by Khandarea et al (Khandarea, 2000) to measure the viscosity of pitch material up to 100 Pa at elevated temperatures. This apparatus is pressurized with gas, which is not soluble in pitch. The substance can be investigated at pressures up to 76 bar with a maximum temperature of 500 °C. Standard viscosity fluids are used at varying temperatures for calibration. A high pressure capillary rheometer has been built by Couch et al (Couch and Binding, 2000) to measure polymer fluids with viscosities from 0.1 to 10,000 mPa at pressures up to 800 bar and at shear rates between 50 and 2500 1/s. A capillary viscometer especially designed to be operated over a broad range of temperatures and pressures up to 100 bar of saturated liquids that may be corrosive and incompatible with elastomers with kinematic viscosities smaller than $2{\cdot}10^{-7}$ m^2/s is described by Ripple (1992); the accuracy of this viscometer is approximately 3%. Schurz and Prassl (1993) developed an automated high-pressure capillary rheometer in which a hydraulic device is used to drive the sample fluid through capillaries. With this apparatus polymer solutions in a viscosity range from 0.001 to 10,000 mPas can be measured.

In the literature several high pressure capillary rheometers are described. Some of them are pressurized with gas that may dissolve into the oil resulting in non-negligible viscosity changes. Others have a small viscosity range and are not suitable for measuring heavy oils. To measure heavy oils at reservoir conditions a rheometer with a pressure range up to 1000 bar and a high range in viscosity and shear rate is needed. Therefore, we developed a rheometer that provides high pressure and high shear rates similar to those that may be found during processing of heavy oils.

In a current research project between the Ruhr University Bochum (RUB) and the Universidad Nacional Autónoma de México (UNAM) a high pressure, high shear rate capillary rheometer has been designed and developed. The principles of construction and operation are described in this paper. Crude heavy oil is pressed through a calibrated capillary at high pressure and a constant rate of flow while the pressure drop is measured. The viscosity of Newtonian fluids is calculated from the pressure drop with Hagen-Poiseuille's law. In case of non-Newtonian fluids, corrections have to be applied. The capillary rheometer is rated up to 1000 bar and 200 °C. Fluids with a viscosity between 1 and 100,000 mPas can be measured under different high shear rates.

To make measurements as accurate as possible, the rheometer needs to be calibrated at equivalent conditions for pressure and temperature. Reference viscosity values were first measured at UNAM at ambient pressure with an Anton-Paar Stabinger viscometer or an Ares G2 rheometer. Subsequently the reference tables were extended for high-pressure conditions by means of a falling body viscometer. Two Newtonian viscosity reference blends were used and their viscosities were measured from atmospheric pressure to 1000 bar and in a temperature range from 20 to 120 °C. Afterwards, the same Newtonian reference fluids were measured in the capillary rheometer at the Ruhr-University within a pressure range from 1 to 500 bars and at temperatures from 30 to 100 °C. After that, hydraulic oil has been measured at high pressure and high shear rates to proof the functionality of the apparatus.

2. Material and methods

2.1. Materials

For the calibration of the capillary rheometer blends of polybutene (isobutylen-/buten-copolymer) of various viscosities from a calibration laboratory in Wolfen, Germany are used. The relative uncertainty U of the viscosity amounts to U = 0.5% according to the calibration certificate. U is the expanded uncertainty, with a coverage factor k = 2. (ZMK Unternehmen, 2014).

To set the apparatus into operation temperature stable hydraulic oil type HLP 46 (Shell) is used. The oil has a viscosity of 72 mPas and a density of 780 kg/m^3 at ambient conditions.

Viscosity and flow behavior of a Mexican oil sample with a density of 1 002 kg/m^3, an API-Grad of 9.6 and a zero-viscosity of 283 Pa at 20 °C and 1 bar is measured in the capillary rheometer. Density and viscosity are measured by the authors.

2.2. Methods

The basic equation of the capillary flow is the Hagen-Poiseuille equation for the dynamic viscosity η of Newtonian fluids (Deutsches, 1994a),

$$\eta = \frac{\Delta p \cdot \pi \cdot R^4}{8 \cdot Q \cdot L} \tag{1}$$

where Δp is the pressure difference, Q the volumetric rate of flow, R the radius and L the length of the capillary. While the viscosity of Newtonian fluids is independent of the shear rate and therefore constant over the cross section of the capillary, the viscosity of shear thinning fluids decreases near the wall. Therefore, for non-Newtonian fluids Hagen-Poiseuille's law only provides the so-called apparent viscosity. This is because the viscosity is a function of the shear rate, which in turn is a function of the radial position in the capillary and therefore not constant. The shear rate has to be corrected with the Weissenberg-Rabinowitch equation, which is used for non-Newtonian fluids and provides the viscosity at the wall. Eq. (2) gives the shear stress τ_w at the wall:

$$\tau_W = \frac{\Delta p \cdot R}{2 \cdot L} \tag{2}$$

The apparent shear rate at the wall is calculated from the known flow rate Q:

$$\dot{\gamma}_{ap} = \frac{4 \cdot Q}{\pi \cdot R^3} \tag{3}$$

According to DIN 53014 part 2 (Deutsches, 1994b) the shear rate at the wall $\dot{\gamma}_w$ is calculated with Eq. (4) from the apparent shear rate $\dot{\gamma}_{ap}$,

$$\dot{\gamma}_w = \frac{\dot{\gamma}_{ap}}{4} \cdot \left(3 + \frac{d \log \dot{\gamma}_{ap}}{d \log \tau_W}\right) \tag{4}$$

The differential quotient in this equation is determined graphically, the procedure is described elsewhere (Hüttemann et al., 2015). The dynamic viscosity at the wall for non-Newtonian fluids can be calculated from Newton's law of viscosity:

$$\eta_W = \frac{\tau_W}{\dot{\gamma}_W} \tag{5}$$

3. Capillary rheometer

3.1. General description

With the capillary rheometer, pressures up to 1000 bar and temperatures up to 200 °C can be generated. The system is designed for highly

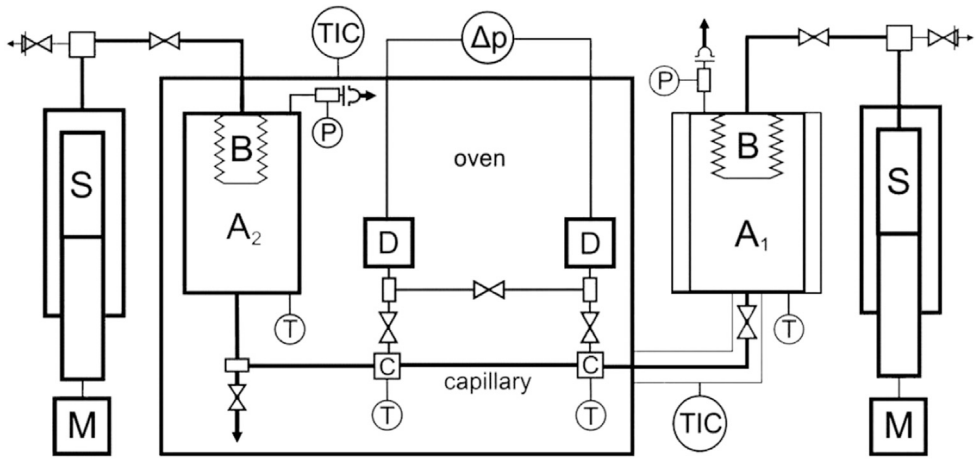

Fig. 1. Schematic drawing of the high-pressure high shear rate capillary rheometer: (A_1 and A_2) autoclave, (B) bellow, (C) connection blocks, (D) differential pressure sensor, (M) motor, (P) pressure sensor, (S) spindle press, (T) temperature sensor.

Fig. 2. Connection blocks with large inner volume to reduce dynamic pressure effects.

viscous liquids with viscosities up to 100,000 mPas. The experimental setup is shown in Fig. 1.

It consists of two high-pressure vessels (A_1 and A_2) connected by a hydraulic system including the calibrated capillary. The autoclave A_1 is mounted on a tiltable rack, so that the opening can be turned upwards and the sample can be filled in easily. This autoclave has a volume of 1 L and is heated with a double jacket. The other autoclave is integrated into the oven and has a volume of 1.2 L. Both vessels are protected against over-pressure with a rupture disc. Most components of the rheometer are mounted in a high-precision oven to ensure a constant temperature during the measurement. Both vessels are equipped with a hydraulically operated metal bellow (B) that can be varied in size by two electrically driven spindle presses (S). The capillary itself is mounted between two connection blocks (C), which are also connected to a highly sensitive differential pressure manometer (D). The apparatus has a volume of 1.1 L and is fully automated and controlled by a PC unit and a Labview-program.

3.2. Connection blocks

The connection blocks, which are made from a high-temperature resistant stainless steel (1.4980), have a spherical inner volume. In order to measure the differential pressure as accurate as possible, a measurement in a quasi-static fluid is required. Therefore the 15 ml inner volume of the connection blocks is considerably larger in comparison to the diameter of the capillary. The flow velocity in the connection blocks is close to zero and thereby dynamic pressure effects are avoided during measurement. In addition to the differential pressure the temperature is measured in both connection blocks. Fig. 2 shows the connection blocks with a tension screw.

3.3. Pressure and temperature control

In both vessels the absolute pressure is measured with a pressure sensor (Dynisco) with a design pressure up to 1000 bars. The sensor is calibrated with a pressure calibrator by Digiquartz (model 765). After that, the accuracy of the sensor is 0.5% of the full scale. The temperature control is done at four different points with PT 100 temperature sensors. The temperature is measured in both vessels as well as at the inlet and outlet of the capillary. Thanks to the temperature measurement at the outlet of the capillary, a possible increase in temperature due to friction could also be determined. The sensors have an accuracy of 0.1 K after calibration. All signals are evaluated with an IO-card by National Instruments and are logged in a Labview-Program.

3.4. Spindle presses and bellows

The flow is generated with two electrically driven spindle presses by Dustec (Germany). Each spindle press has a volume of 308 ml. The rotational speed can be controlled with a frequency converter. Thereby the flow rate can be varied between 1 ml/min and 340 ml/min. For low viscosity oil with 10 mPas and a density of 970 kg/m^3, the maximum possible flow rate in the smallest capillary leads to a Reynolds Number of 665. Thus, the assumption of a laminar flow as a premise for Eq. (1) is fulfilled. For significantly lower viscosities, the flow rate may have to be reduced. The piston displacement is controlled by a step motor and divided into 15700 steps. That allows selecting the flow rate with high precision. Each spindle press is connected to a bellow by a tube system and is filled with temperature stable hydraulic oil. The hydraulic oil is preheated in a coiled tube inside the oven, so only pre-heated hydraulic oil goes into the bellow and no unwanted temperature effects are generated. Both spindle presses are equipped with an overflow valve against high pressure.

3.5. Differential pressure transmitter

A differential pressure transmitter (Fuji Electric, France) is used to measure the pressure drop over the capillary. The instrument is designed for a pressure drop from 0.01 to 5 bar with an uncertainty of 0.1%. The instrument utilizes a membrane with a microprocessor sensor to measure the pressure drop. The pressure transmitter is able to measure a large

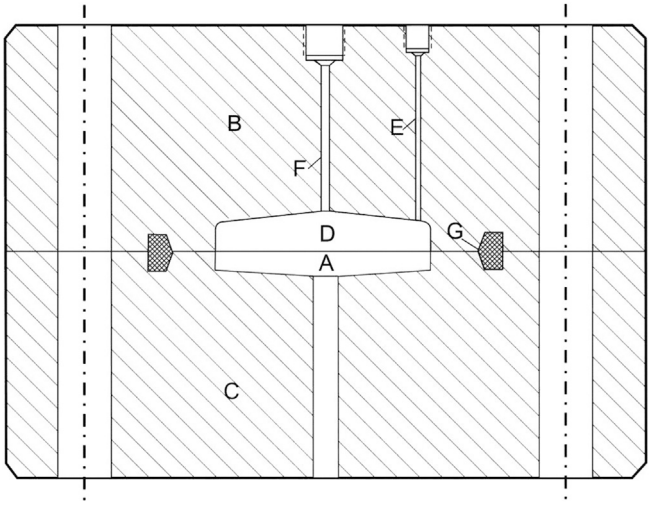

Fig. 3. Flange for connecting the differential pressure instrument.

Table 1
Shear rate as a function of the diameter of the capillary.

capillary diameter in mm	viscosity in mPas	shear rate in (1/s)
2.8	600–200,000	10–2000
2.4	50–100,000	10–4000
2.1	0.3–60,000	20–6000
1.59	0.1–20,000	40–13,000
1.05	0.01–4000	150–50,000

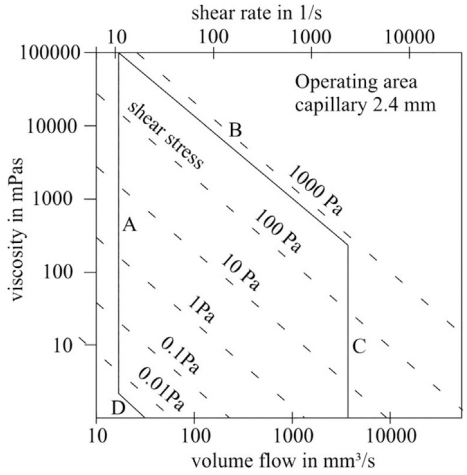

Fig. 4. Operating area of the capillary rheometer using a capillary with a diameter of 2.4 mm and a length of 283 mm: (A) minimum flow rate, (B) maximum shear stress, (C) maximum flow rate and (D) minimum shear stress.

range of differential pressures with an acceptable uncertainty under very high absolute pressures. The large range of differential pressure is necessary to realize different shear rates. On both sides, the differential pressure transmitter is connected to a flange with a metal membrane (A) to 'pick up' the pressure. Each of them is connected to a flange (B), which was designed by the authors. Fig. 3 shows a drawing of such a flange connected to one side of the differential pressure sensor (C). The system has an internal volume (D) of 40 ml. The fluid flows into the flange via connection (E), so that entrapped air bubbles can be vented over connection (F) when the rheometer is filled with oil. The flange is made from a stainless steel (1.4407) and sealed with an elastic seal ring (G) of PTFE.

3.6. Capillaries

For the measurements described here, long capillaries (200–300 mm) are used, so that inlet effects play only a minor role and, if necessary, can be corrected. The capillaries are made from a 1.4404 stainless steel tube. With the radius of the capillary the range of operation of the apparatus can be described and the shear rate can be calculated. Table 1 shows the range of operation for five different capillaries. All capillaries are straight pipes with a length of 283 mm.

It can be seen that with higher shear rates the viscosity range becomes increasingly limited. Fig. 4 shows an estimation of the range of operation for the capillary rheometer using a capillary with 2.4 mm in diameter.

3.7. Measuring procedure

The sample to be investigated is filled into the main autoclave where the bellow is contracted to the minimum volume. To fill the oil into the other components of the rheometer, the system has to be evacuated first. Then, by opening a valve and simultaneously enlarging the bellow, the fluid is fed into the machine. If necessary the fluid may be heated up to 60 °C to reduce its viscosity while filled in. The pressure build-up is done mechanically by a hand pump. At the beginning of the measurement, the bellow in the main autoclave is expanded to its maximum volume and the bellow in the other autoclave is in its minimum volume position. By expanding the bellow and simultaneously shrinking the other one, the fluid is pressed through a capillary at a constant flow rate. The bellows have a volume of ±150 ml around its zero-position, so 300 ml of the fluid are moved during a measurement. The resulting pressure drop is recorded and the viscosity is calculated with the procedure described above. The measurement is controlled by a Labview-Program, which logs all data during the experiment. The temperature has the biggest influence on the viscosity. According to DIN 53014 (Deutsches, 1994a) the temperature fluctuation has to be below 0.1 K for temperatures < 50 °C and below 0.25 K at temperatures > 50 °C to avoid too large uncertainties. During a measurement the program saves about 5000 data points providing a precise temperature record. For the evaluation, the arithmetic average of the pressure, pressure drop and temperature is calculated from a stationary period during the measurement.

4. Result and discussion

4.1. Corrections

The basic equations of the capillary flow require idealized conditions and a steady laminar flow. The flow has to be fully developed. The viscous losses and therefore the pressure drop are negligibly small so that the absolute pressure and thereby the viscosity can be considered as constant along the tube. For experimental setups these conditions cannot always be met. Entrance-effects can influence the pressure drop. Temperature and pressure can affect the length of the capillary and lead to mistakes during the evaluation. Frictional heat in the capillary can result in significant changes in temperature with a strong influence in the fluid's viscosity. Therefore, it is investigated which of these effects, if any, influence the viscosity measurements and have to be corrected.

4.1.1. Bagley-Correction

When a fluid is pressed through a capillary, inlet effects occur. The inlet pressure loss, which occurs at the capillary entrance and at the first part of the capillary until a laminar flow is developed, is a part of the total pressure drop. The inlet pressure loss depends on the rheological properties of the fluid, the flow profile and the diameter of the capillary. The same applies for the outlet of the capillary. The sudden enlargement of the cross section results in a loss of kinetic energy, which provides an additional contribution to the pressure drop. Both the additional pressure losses are identifiable and correctable using the Bagley-Correction (Branes et al., 1989). It includes the Hagenbach-Couette correction,

Table 2
L/D ratios of three different capillaries.

Diameter in mm	L/D ratio
2.4	118
2.1	135
1.59	177

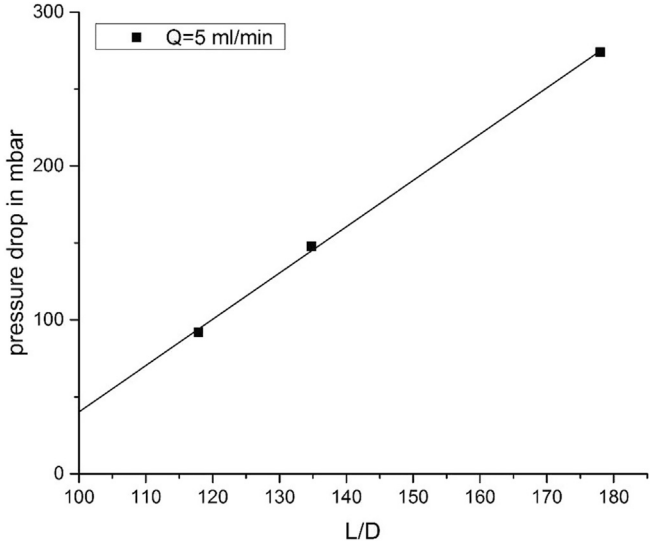

Fig. 5. Bagley-Correction for a flow rate of Q = 5 ml/min.

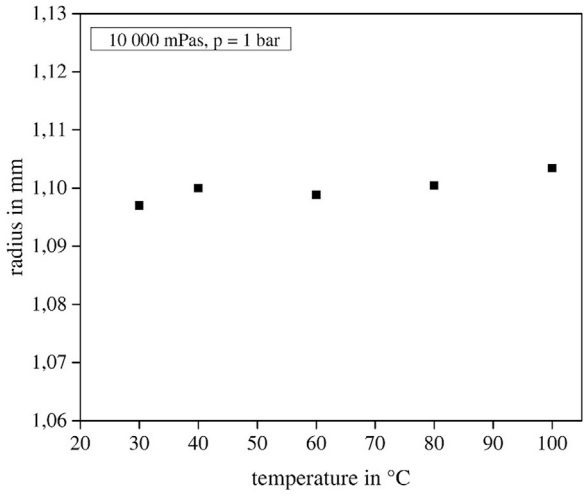

Fig. 6. Inner diameter of the capillary as a function of temperature.

which does not need to be calculated separately (Deutsches, 1994b). The method is based on the use of various capillaries with a different length to diameter ratio. Table 2 shows the different L/D ratios of the three capillaries.

For the Bagley-Correction, an apparent flow curve (Δp vs. Q) for each capillary is measured. The apparent shear rate is calculated from the volume flow with Eq. (3). First, the apparent flow curve for each capillary is plotted against the differential pressure in a logarithmic diagram. The data is then converted into a plot of Δp vs. (L/D) for different flow rates Q (Fig. 5). The y-intercept (offset) of each curve gives the Bagley-Correction for the respective flow rate.

The Bagley-Correction is often used for short capillaries and diameters smaller than 0.5 mm. The evaluation of the results shows, that the additional pressure drop is negligibly small in comparison to the "main" Δp and has no influence on the results.

4.1.2. Thermal linear expansion

A change in length of the capillary can occur during measurements at high pressures and/or temperatures. This change either needs to be considered or avoided. The linear thermal expansion is described by the coefficient of linear expansion at a given temperature difference. It can be calculated with Eq. (6).

$$\Delta L = L_0 \cdot \alpha \cdot \Delta T \tag{6}$$

where ΔL is the change in length, L_0 the reference length of the capillary at a reference temperature T_0, α is the thermal expansion coefficient and ΔT the temperature difference between the actual temperature and T_0. From DIN 53014 (Deutsches, 1994a) α is given with $16.5 \cdot 10^{-6}$ 1/K for stainless steel. The length of the capillary is 283 mm. The length of the capillary increases by about 0.5 mm when the temperature increases by 100 K. This influences the calculated viscosity by around 0.17%.

4.1.3. Frictional heat and temperature increase

When the fluid is pressed through the capillary, mechanical energy is converted into frictional heat. The heat P can be calculated using Eq. (7). For the most measurements frictional heat is generated in the order of 0.1 W and not considered further.

$$P = \Delta p \cdot Q \tag{7}$$

4.2. Calibration of the capillary rheometer

The capillary rheometer is calibrated with two standards at different temperatures and pressures. The standards used were two Newtonian ZMK (ZMK Unternehmen, 2014) viscosity reference fluids of 10,000 mPas and 50,000 mPas nominal viscosities. However, the original tables of these fluids apply only to ambient pressure and had to be extended to high-pressure, as already indicated in a previous publication (Hüttemann et al., 2015). For that purpose, the high-pressure calibration tables were obtained in two steps:

Step 1: Extension of the atmospheric tables to the full range of temperature combining a high-precision Anton-Paar Stabinger SVM 3000 viscometer and an Ares G2 rheometer using a bob and cup geometry. Both devises were independently calibrated according to manufactures indications obtaining an overall agreement better than 1%.

Step 2: High-pressure correction using a falling body viscometer. It should be remarked that this procedure does not involve an absolute calibration of the falling body instrument but only a high-pressure correction at a given temperature. The correction considered only buoyancy effects due to changes in the densities of the fluid and the falling body. As previously remarked (Hüttemann et al., 2015), this approach allowed us to reproduced reference values for water up to 1000 bar and 40 °C with an overall accuracy of 0.17%. These error estimations were for extreme-light measurements and it was not possible to measure higher temperatures due to the low water viscosity resulting in the body falling too fast. In the case of higher viscosity fluids, however, the falling time substantially increases obtaining a more precise time reading and consequently higher accuracy. Therefore, the estimated correction at high-pressure is expected to have an uncertainty under 0.5% with a cover factor of 2. Overall, we estimate the accuracy of the reference calibration table to have been of 2% at high pressure with a cover factor of 2. The tables of high-pressure viscosity standers were extended to a range of 20–120 °C and up to 500 bar. This exceeded the range of interest for this project that was 20–100 °C and up to 500 bar.

Then, reference standards of the same batch were used in Germany for the calibration of the capillary rheometer. The calibration has been done at low shear rates, temperatures between 30 and 100 °C, and a pressure range from 10 to 500 bar. As the properties of reference fluids

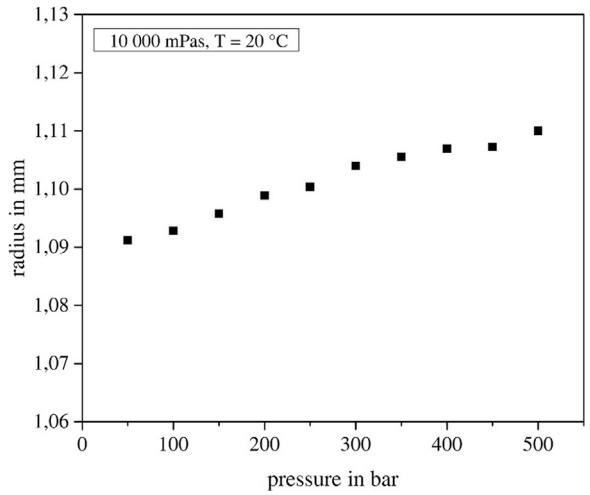

Fig. 7. Inner diameter of the capillary as a function of pressure.

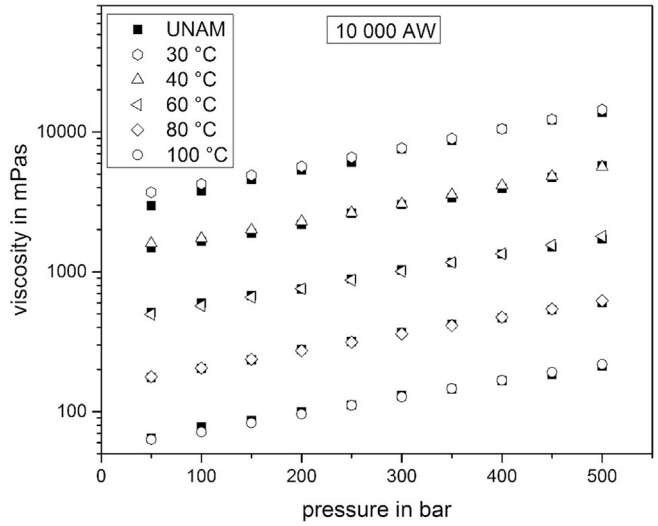

Fig. 8. Comparison of the viscosity curves of RUB and UNAM after the high-pressure calibration for the 10,000 mPas standard at different temperatures. The shear rate used was 50 1/s.

vary with the batch, the calibration tables are only valid for the actual batch of the calibration fluid. From the calibration measurements, the radius of the used capillaries is determined with Hagen-Poiseuille's law.

$$R = \sqrt[4]{\frac{8 \cdot Q \cdot L \cdot \eta}{\Delta p \cdot \pi}} \tag{8}$$

This calibration is conducted in two steps. First, the radius is determined at different temperatures while the pressure is kept constant at 1 bar (Fig. 6). Then the radius is determined at different pressures while the temperature is kept constant at 20 °C (Fig. 7). It can be seen that the diameter of the capillary increases with both quantities but much more with the pressure than with the temperature. The influence of the temperature is estimated as negligibly small.

In Fig. 8 the results after calibration of the capillary diameter from 1 to 500 bar of the 10,000 mPas sample at 30, 40, 60, 80 and 100 °C are fitted against the viscosity tables from UNAM. The deviation of the measurements is between 0.1 and 3%. This sample shows a strong viscosity rise with the pressure, mainly at the lower temperatures 30 and 40 °C.

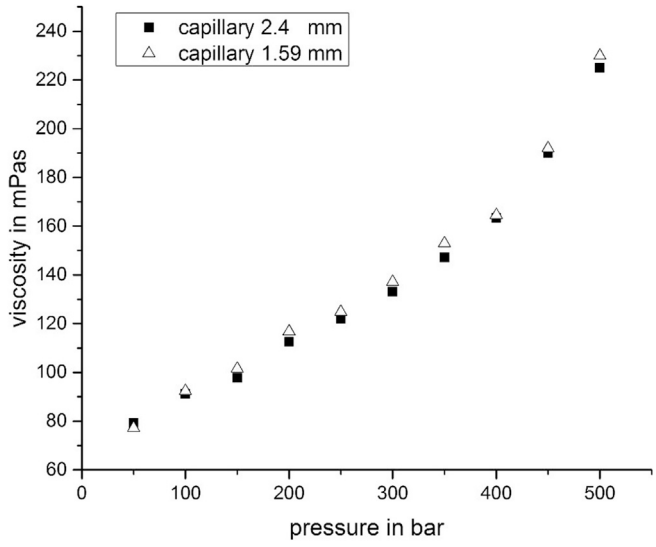

Fig. 9. High-pressure results for the hydraulic oil HLP 46 and two capillaries.

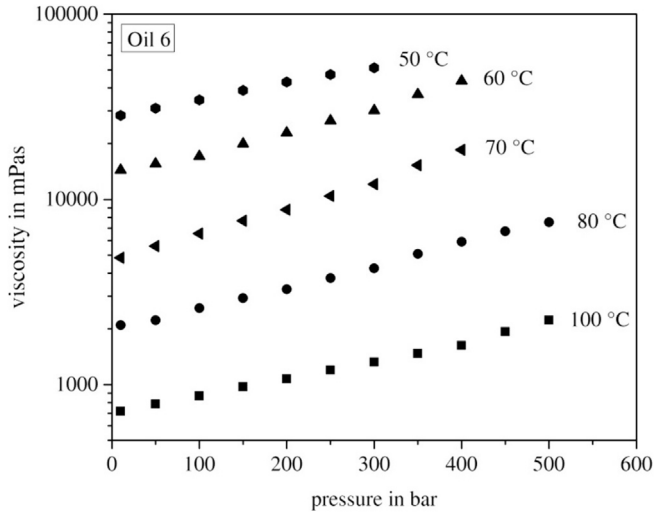

Fig. 10. Viscosity-pressure diagram for a heavy oil sample.

4.3. Putting the capillary rheometer into operation

The functionality of the high-pressure capillary rheometer is tested with temperature stabile hydraulic oil. First, some measurements are made at high pressure with different capillaries. Fig. 9 shows the viscosity increase for hydraulic oil with pressure increasing up to 500 bar for two capillaries.

It can be seen from Fig. 9, that the viscosity increases noticeably with the pressure. The result is independent of the installed capillary. The results differ around 2.5%, which is within of the presumed accuracy.

4.4. Viscosity and flow behavior of an extra-heavy oil

Fig. 10 shows the influence of the pressure on the viscosity in a temperature range between 50 and 100 °C.

It can be seen from Fig. 10, that the pressure has a strong influence to the viscosity. For example, the viscosity increases from $\eta = 2045$ mPas to $\eta = 4\,825$ mPa at 80 °C. Fig. 11 shows the flow behavior of the same sample at 200 and 300 bar and 70 °C.

The first Newtonian plateau is found at low shear rates (10 - 20 s^{-1}) and a second Newtonian plateau can be seen at shear rates from 150 to

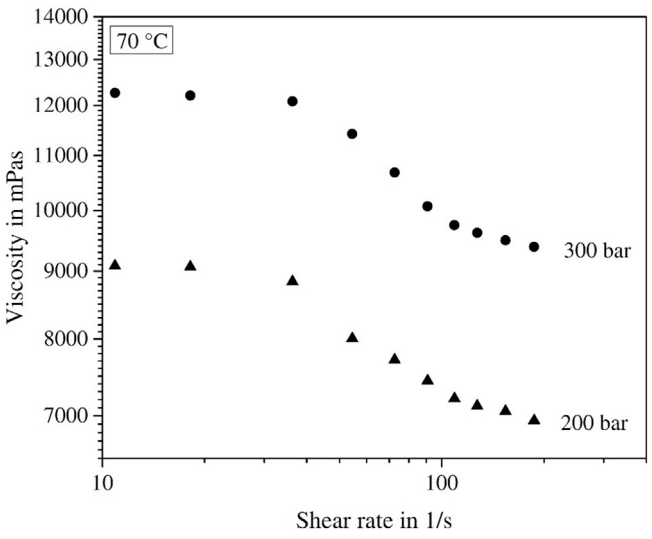

Fig. 11. Viscosity-shear rate diagram for a heavy oil sample.

200 s^{-1}. Furthermore, the distance between the two Newtonian plateaus enlarges with increasing pressure. The viscosity and flow behavior of other heavy oil samples with non-Newtonian flow are described elsewhere (Hüttemann, 2016).

5. Summary and conclusion

This paper describes the design and development of a capillary rheometer for high pressures and high shear rates. The apparatus is built in order to investigate rheological properties of heavy crude oils at reservoir conditions and under conditions similar to those in Enhanced Oil Recovery (EOR) processes. To determine the flow behavior of high viscous fluids under high shear rates, the capillary rheometer is equipped with two spindle presses, which can generate a highly volumetric rate of flow. To investigate fluids with very different viscosities, a differential pressure transmitter with a large pressure range is chosen. Connection blocks with a large inner volume up- and downstream the capillary are used to connect the differential pressure manometer. By this way, dynamic pressure effects are kept negligibly small and do not contribute to the differential pressure measurement.

To measure viscosities with high accuracy the capillary rheometer is calibrated with two Newtonian reference blends of different viscosities at 30, 40, 60, 80 and 100 °C and pressures up to 500 bar. The diameter of the capillary was found to be a function of temperature and pressure. The high-pressure calibration appears to be necessary for further measurements, because the radius changes with increasing pressure and temperature. In Hagen-Poiseulle's law, the radius contributes with the 4th power, so the changing of the diameter must be considered for accurate measurements. The results of the high-pressure calibration show that the deviation from the reference fluids was lower than 3%.

After verifying the calibration, the functionality of the apparatus was tested with temperature stable hydraulic oil. Capillaries with different diameters are installed to measure viscosity at high pressure and high shear rates. The viscosity was determined at 40 °C and pressures from 10 to 500 bar, the flow behavior is measured at shear rates from 10 to 2000 s^{-1} at 80 bar and 40 °C. The hydraulic oil shows a Newtonian flow behavior under the conditions examined. The development of the capillary rheometer enables viscosity measurements under high pressure at high shear rates with very low measurement deviations.

With the apparatus flow curves of heavy oil samples can be measured under reservoir conditions. The flow curve of the investigated oil clearly shows a non-Newtonian behavior at high pressure. The viscosity decreases by over 25% when increasing the shear rate to 200 s^{-1}. To produce heavy oil economically and efficiently, the knowledge of the flow behavior under reservoirs conditions is essential.

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