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Development of a Capillary Rheometer for the Characterization of Heavy and Extra-Heavy Oils

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ABSTRACT: Viscosity and flow behavior of crude oils are key properties for the characterization, management, and development of petroleum reservoirs. To measure the rheological properties of heavy crude oils with a high accuracy, a capillary rheometer was designed and built at the Ruhr University in Bochum (RUB), Germany. The capillary rheometer was calibrated with three Newtonian reference blends of 1000, 10 000, and 50 000 mPa s. For these standards, the viscosity tables have been extended up to 1000 bar in the temperature range of 20−120 °C. The extended viscosity data were determined with a falling body viscometer at the Universidad Nacional Autónoma de México (UNAM), obtaining average deviations better than 2.0% at high pressure. The capillary rheometer is then calibrated with the same reference fluids at 40, 60, 80, and 100 °C at pressures from 1 to 260 bar. The deviation from the reference value was found to be lower than 3% at high pressure. The flow behavior of a heavy oil sample is determined at 80, 100, and 120 °C at atmospheric pressure. Furthermore, the viscosity is measured from atmospheric pressure to 180 bar. The investigated heavy oil shows a Newtonian behavior at high temperatures. This is consistent with rheological measurement carried out at UNAM. The viscosity increases with increasing pressure at all temperatures. For example, at 40 °C, the viscosity increases by about 35% by increasing the pressure from 1 to 100 bar.

1. INTRODUCTION

The global energy needs will increase until 2050.[1] This is partly due to the economic development and the demographic growth in the emerging economies and the increasing energy consumption in industrialized countries. Currently, the world's demand for energy increases by 8% annually.[2] Although renewable energy sources are becoming increasingly important, the energy supply is still based on raw fossil fuel materials.[3] It is projected that renewable energy will account for around half of the global energy consumption by 2040. At present, oil still covers about 30% of global energy needs.[4] Extra-heavy oils and oil sands represent almost 7% of this supply.[5] Also in the future, heavy crude oils will contribute as a relevant hydrocarbon source.[5,6] Because of the declining oil reserves, an effective recovery of the available resources is becoming more and more important.[7] The estimated amount of heavy oils is about 6 trillion barrels, 6 times the conventional oil reserves. For example, the size of the estimated heavy oil and oil sand resources in the United States amounts to 100 and 62 billion barrels of oil in place, respectively.[8] Although this represents a small fraction of the total world reserves, it is about one-third of the total estimated crude oil discovered in the United States. The processing and production of high viscous, heavy oil is a big challenge for the oil industry. The flow behavior of extra-heavy mineral oil under reservoir conditions, which means high pressure and temperature, is not known. During production, transport, and processing, the oil undergoes large variations in the temperature, which lead to variations in viscosity of orders of magnitude. Under typical conditions, light petroleum fluids behave Newtonian, while extra-heavy oils tend to behave non-Newtonian. Heavy oils, with an American Petroleum Institute (API) gravity of around 20°, may show viscosity values of around 500 mPa s at 20 °C and mostly Newtonian behavior. Extra-heavy oils (with API gravities below 15°) can show dramatic rheological differences. At 20 °C, between just 12° and 11° API gravities, one can find viscosities that may range from 10 000 to 300 000 mPa s or even higher[9] and a clear non-Newtonian behavior.

Although published studies on non-Newtonian oils are scare in comparison to the extensive literature available about lighter Newtonian fluids, different authors have reported the rheological behavior of heavy crude oils. Between others, the flow behavior of a heavy oil sample at atmospheric pressure and its mixtures with light crude oil to reduce the viscosity was investigated with a RheoStress RS100 rheometer experimentally by Hasana et al.[10] Ghannam et al.[11] investigated heavy crude oil, showing a non-Newtonian shear thinning behavior over the examined range of the shear rate from 0.1 to 750 s^{-1} at temperatures from 25 to 65 °C. The viscosity of heavy oil samples from Mexican reservoirs with an API gravity from 11.5° to 19.4° was measured at a temperature range from 27 to 124 °C using an electromagnetic viscometer by de la Cruz et al.[12] The viscosity and density of 50 dead crude oil samples collected from various Kuwaiti oil fields were measured by Alomair et al.[13] at temperatures ranging from 20 to 160 °C. The rheological behavior of almost 20 Mexican base oils (from light to extra heavy) and 30 blends have been studied from 20 to 100 °C by Soto et al.[9] as well as the effect of the water content[14] and temperature[15] in the same batch of oils. The friction theory (FT) was shown to be capable of delivering simple and accurate viscosity models for a wide range of petroleum reservoir fluids, from light to heavy, and viscosities up

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to 10 000 mPa s under usual reservoir conditions.[16] Currently, the FT is being extended for the modeling and prediction of over 50 oil blends made of more than 20 Mexican fluids ranging from extra light to extra heavy.[17]

Actually, there is a lack of information concerning the non-Newtonian behavior of heavy oils, particularly at high pressure and high shear rates. In our experience, heavy oils appear to develop viscosity thinning at high shear rates and exponential increment of the viscosity with pressure. Measuring these properties with conventional devices is not a simple task. Conventional rheometers take advantage of open geometries (bob and cup, parallel plates, etc.) but cannot go to high shear rates because of the fact that the fluid may destabilize or spill out (even heavy oils). There are also some pressure chambers for high-pressure rheometric measurements, but many of them have magnetic couplings, which may also decouple before viscosity thinning is observed. In addition, it may happen that the gas used to pressurize the rheometer may dissolve into the oil, resulting in non-negligible viscosity changes. Therefore, the scope of this work focuses on the development of a closed geometry apparatus that may allow for the study of the flow behavior of heavy oils at a broad range of pressures and shear rates. One option is the use of a device with the capability of controlling wide ranges of fluid flow, e.g., a capillary rheometer. The aim of this work is to be able to provide extended information on the rheology of non-conventional fluids that is crucial for the design of pipelines as well as the production facilities.

In this paper, we present initial results of a joint research project between RUB and UNAM. To study the rheological properties of heavy crude oils, a capillary rheometer was designed and manufactured at RUB in Germany in collaboration with UNAM. With this device, the viscosity and flow behavior can accurately be measured up to 300 bar and 200 °C.

To accurately measure the viscosity of heavy oil samples, calibration at high pressure and temperature is needed. The calibration of the capillary rheometer is performed in two steps: First, high-pressure calibration tables were produced using a falling body viscometer at UNAM. For this purpose, the design of a hydraulic system for high pressure and temperature was implemented. The experimental setting is validated against pure reference fluids, such as water, obtaining viscosity deviations lower than 0.5% at high pressure. Three Newtonian viscosity reference blends of 1000, 10 000, and 50 000 mPa s have subsequently been studied, and their reference tables have been extended from atmospheric pressure to 1000 bar in a temperature range of 20−120 °C. In a second step, the high-pressure calibration tables were used to calibrate the capillary rheometer at high pressures and temperatures. After that, the rheological properties of one stabilized Mexican heavy oil sample have been analyzed with this machine. First, the flow behavior of the investigated sample is analyzed. The rheology behavior of the fluid is studied at 80, 100, and 120 °C by varying the shear rate from 1 to 120 s^{-1} and compared to rheological measurements made on the same fluid at UNAM. Thereafter, the viscosity is measured from 1 to 180 bar at 40, 60, 80, 100, and 120 °C.

2. MATERIALS

For the calibration of the capillary rheometer, three Newtonian viscosity reference blends of 1000, 10 000, and 50 000 mPa s from a calibration laboratory (ZMK) in Wolfen, Germany, are used.[18] The sample of 1000 mPa s consists of dec-1-ene trimer (hydrogenated), and the other two are mixtures of polybutene (isobutylene/butene copolymer) of various viscosities. The relative uncertainty (U) of the viscosity given in the calibration certificate amounts to $U = 0.25\%$. The expanded uncertainty, which results from the standard uncertainty times a coverage factor $k = 2$, is given. The measured value is found within the attributed interval with a probability of approximately 95%.

The fluid properties and the rheological behavior of a Mexican heavy oil sample, jointly studied at UNAM and RUB, are presented in this paper. The oil has an API gravity of 12.6°, a density of 978 kg/m^3, and a zero-shear viscosity of 12.9 Pa s at 20 °C.

3. REFERENCE MEASUREMENTS

3.1. Atmospheric Reference Data. The viscosity of the reference fluids was verified at UNAM with a Stabinger Anton-Paar viscometer and an Ares G2 rheometer using a bob and cup geometry in the range of 20−100 °C. The uncertainty of the UNAM measurements is of 2% with a coverage factor of $k = 2$. In the range of overlapping, the agreement between the Stabinger viscometer and the Ares G2 rheometer is better than 1%. Typically below 10 Pa s, the Stabinger viscometer is expected to be more precise, but above 10 Pa s, the measurements require the use of the rheometer. The largest deviations between the ZMK reference data and the UNAM measurements corresponded to the highest viscosity point of the 50 000 mPa s fluid with an uncertainty of 3.7%. At a logarithmic scale, however, the agreement is better than 0.4% in all cases. Given the fact that viscosity behaves exponentially with the temperature, the agreement is considered to be within reference accuracy (Figure 1).

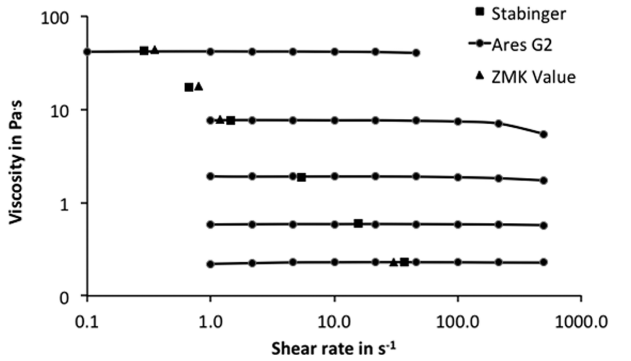

Figure 1. Comparison of viscosity values between UNAM measurements and the reported ZMK reference values for the 50 000 mPa s calibration fluid.

3.2. High-Pressure Reference Data. The extension of the reference data to high pressure was performed with a falling body technique. There are not many fluids for which their viscosity has been accurately measured to high pressure. The accuracy of the procedure that was applied was verified against reference values for water[19] up to 40 °C and 1000 bar. In this case, the average absolute deviation was of 0.17%, with a maximum deviation below 0.5% at 1000 bar and 40 °C. In the case of the ZMK high-viscosity standards, the estimated uncertainty in the increment of viscosity from atmospheric to 1000 bar was 2% with a cover factor of $k = 2$ at normal scale and under 0.5% in logarithmic scale. The experimental setting and the detailed description of the procedure are reported elsewhere.[20] Figure 2 shows the viscosity results for the 10 000 mPa s ZMK as extended up to 1000 bar. Here, we should remark that the reference tables that were created in this way are specific to the actual batch of the calibration fluid used.

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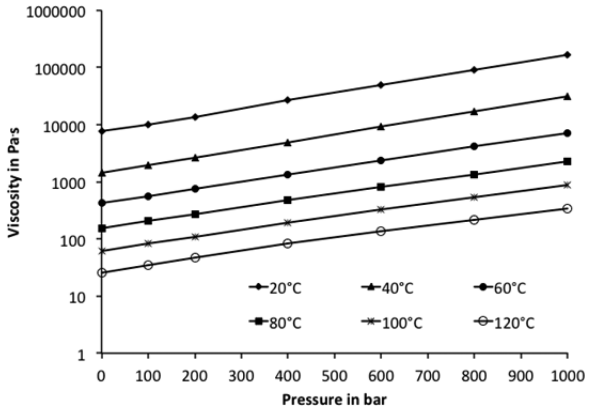

Figure 2. High-pressure viscosity for the 10 000 mPa s calibration fluid.

4. CAPILLARY RHEOMETER

4.1. Working Equations. With the capillary rheometer, conditions up to 300 bar and 200 °C can be generated. Viscosities between 0.5 and 50 000 mPa s can be measured under different shear rates and shear stress. The operating area of the apparatus using a capillary with a diameter of 2.1 mm and a length of 283 mm is shown in Figure 3.

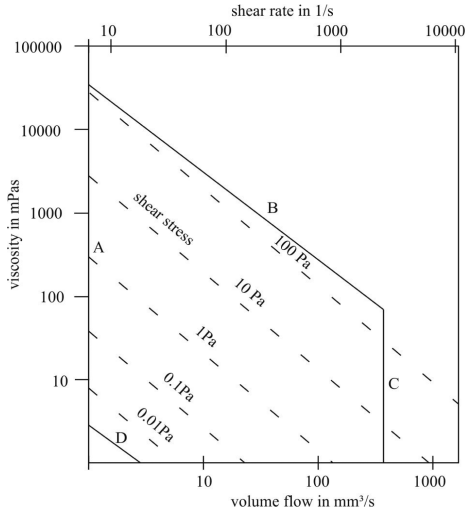

Figure 3. Operating area of the capillary rheometer using a capillary with a diameter of 2.1 and a length of 283 mm: (A) minimum flow rate, (B) maximum shear stress, (C) maximum flow rate, and (D) minimum shear stress.

The experimental arrangement is shown in Figure 4. It consists of two high-pressure vessels (A) connected by a tube system and a calibrated capillary. Before the oil sample is fed into the main vessel, dissolved air is removed from the oil under vacuum conditions. To introduce the oil into the rheometer, the system has to be evacuated and connected to the sample flask. When a valve is opened, the fluid is suctioned into the machine; if necessary, the fluid may be heated to around 60 °C to reduce its viscosity while filled. Most components of the rheometer are mounted in a high-precision drying oven, which ensures a constant temperature during the measurement. The pressure is generated mechanically by a hand pump. Both vessels are equipped with a hydraulically operated metal bellow (B) that can be varied in size by electrically driven spindle presses (S). Two spindle presses are operated in parallel to increase the maximum

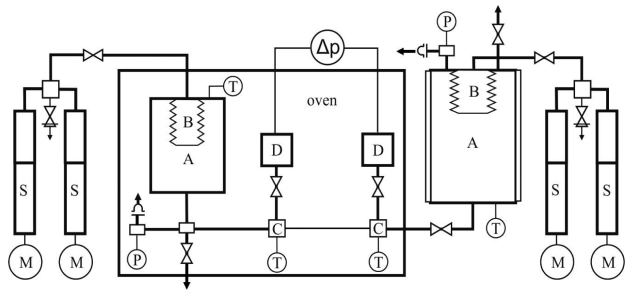

Figure 4. Schematic principle of the capillary rheometer: (A) autocalve, (B) bellow, (C) connection blocks, (D) differential pressure sensor, (M) motor, (P) pressure sensor, (S) spindle press, and (T) temperature sensor.

rate of flow. When one bellow is inflated and the other one is simultaneously shrunk, the fluid is pressed through a capillary at a constant flow rate. The capillary is mounted between two connection blocks (C), which are also connected to a highly sensitive differential pressure manometer (D). The range of the manometer is between 0 and 640 mbar, with an accuracy of 0.1%. In comparison to the diameter of the capillary, the connection blocks have a large inner volume. Therefore, the flow velocity in the connections is close to zero, and dynamic pressure effects do not have to be considered during the differential pressure measurement.[21]

The viscosity of the fluid is calculated from the volumetric rate of flow and the pressure drop with the Hagen−Poiseulles law solved for the viscosity (eq 1)

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

where Δp is the pressure drop, R is the radius, L is the length of the calibrated capillary, and Q is the volumetric rate of flow. The Reynolds number has to be far below $Re = 2300$, so that laminar flow is guaranteed. Upon entering the capillary, additional friction energy is necessary; at the outlet of the capillary, energy is dissipated. These effects are corrected with the Hagenbach−Couette correction (eq 2).[22]

$$\eta = \frac{\Delta p \pi R^4}{8QL} - \frac{m\rho Q}{8\pi L} \tag{2}$$

In eq 2, ρ is the density of the fluid and m is a constant. Analytical solutions for the value of m for circular tubes having a high length/diameter ratio ($L/D > 100$) are provided by Kestin et al.[23] and Philippoff et al.,[24] who gave a value of $m = 1.12$. This effect mainly occurs in small capillary cross-sections, which are used for the measurement of low-viscosity fluids. For the measurement of heavy and extra-heavy oils, larger capillary cross-sections are used. Therefore, the Hagenbach−Couette correction has only a small influence. These equations are only valid for Newtonian fluids. The viscosity of Newtonian fluids is independent of the shear rate and, therefore, constant over the flow cross-section. The viscosity of shear thinning fluids decreases near the wall, and therefore, for non-Newtonain fluids, the Hagen Poiseuille law only provides a so-called apparent viscosity. The actual viscosity can be found with the Weissenberg−Rabinowitch correction. Equation 3 gives the shear stress at the wall of the capillary.

$$\tau_W = \frac{\Delta p R}{2L} \tag{3}$$

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For Newtonian fluids, the viscosity can be determined by a single measurement, because it is independent of the flow rate. For non-Newtonian fluids, the viscosity changes with the shear rate. To obtain information about the flow behavior, a number of measurements is necessary.[25] The apparent shear rate D_{ap} at the wall is calculated from the known flow rate Q.

$$D_{ap} = \frac{4Q}{\pi R^3} \tag{4}$$

According to DIN 53014,[26] the shear rate at the wall D_w is calculated from the apparent shear rate D_{ap} with eq 5.

$$D_W = \frac{D_{ap}}{4}\left(3 + \frac{d \log D_{ap}}{d \log \tau_W}\right) \tag{5}$$

The differential quotient can be determined graphically or using a polynomial-based approach. In this work the differential quotient is determined graphically. For the graphical determination, the individual measurement points of the apparent flow curve $D_{ap} = f(\tau_W)$ are plotted in a double logarithmic diagram, as shown in Figure 5.

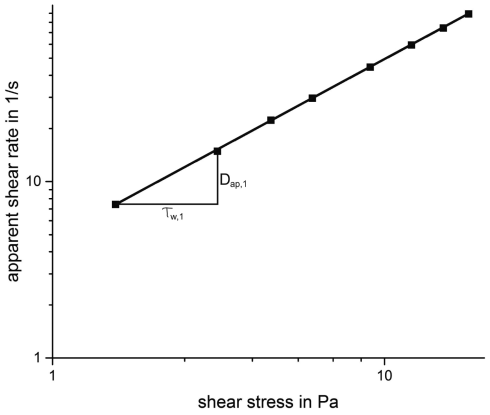

Figure 5. Graphical approach to determine the differential quotient in a double logarithmic diagram.

A linear function is fitted to the measurements. The slope of this function gives the value for the differential quotient. From the shear stress at the wall τ_W and the calculated shear rate D_w, the viscosity at the wall is determined with eq 6.

$$\eta_W = \frac{\tau_W}{D_W} \tag{6}$$

4.2. Calibration of the Capillary Rheometer. The radius of the capillary is calibrated according DIN 53014 part 2.[26] The effective radius can be found with the help of a Newtonian fluid, whose viscosity is exactly known. Therefore, distillated water is used.[19] This is calculated by the Hagen−Poiseulle law at different temperatures. The radius of the capillary was given by the manufacturer with R = 1.05 mm. The effective radius was found to be R_{eff} = 1.04 mm. Applying this new value, the measurements differ from literature values by 0.5%.

The capillary has a high length/diameter ratio with $L/D > 100$. Frictional heat is generated during the flow through the capillary, resulting in a temperature rise. The temperature was found to be negligible, because the heat is dissipated through the capillary wall.

The relative error of the viscosity measurement is calculated with the law of error propagation. Table 1 shows the accuracy of the measurement equipment. The calculated relative error of the viscosity measurement is 2.25%.

Table 1. Uncertainty of the Measurement Equipment[a]

instrument	uncertainty
pressure sensors	0.1%
temperature sensors	0.1 K
volume flow	0.07%
differential pressure	0.1%

[a]The uncertainty describes the deviation of the final value.

5. RESULT AND DISCUSSION

5.1. Calibration of the Capillary Rheometer. Before the first measurements, the capillary rheometer was calibrated with two Newtonian viscosity reference blends at different temperatures and atmospheric pressure. The data, standard deviation, and error of the measurement are shown in Table 2. The measurements were repeated 3 times, and the arithmetic average was calculated. A good reproducibility of the results was found.

Figure 6 shows the results of the calibration of the 1000 mPa s sample at 20, 30, 40, 80, 100, and 120 °C. The deviation to the calibration standards was less than 3%. The standard deviation was found to be ±0.05 mPa s. Thus, the uncertainties of the measurements are within the uncertainty specified by the calibration laboratory. Figure 7 shows the calibration of the 10 000 mPa s sample in a temperature range of 23−100 °C. Also in this series of calibrations, the deviation to the standard is within 3%.

Figure 8 shows the high-pressure results from 1 to 260 bar of the 1000 mPa s sample at four temperatures in comparison to the tables from UNAM. The deviation of the measurements is between 0.5 and 2.8% without any high-pressure calibration of the apparatus. This sample shows a strong viscosity rise, by about 60%, when increasing the pressure up to 260 bar. Figure 9 shows the results of the high-pressure measurements for the 10 000 mPa s sample in the same pressure and temperature range as with the 1000 mPa s sample. The deviation to the standard is less than 3.5% for 40 and 60 °C and 2.5% at 80 and 100 °C. Figure 10 shows the results for the 50 000 mPa s sample. The viscosity of this sample is doubled by increasing the pressure up to 260 bar. The deviation of the standard is around 5% at 40 °C and less than 3% at the other temperatures.

5.2. Rheological Properties of an Extra-Heavy Oil Sample. The rheological properties of a Mexican extra-heavy oil sample have been investigated. The viscosity was measured at different shear rates and temperatures from 80 to 120 °C. The flow behavior of the heavy crude oil is investigated using the capillary rheometer. Figure 9 shows a typical behavior for the results at different temperatures. It is observed from Figure 11 that the crude oil apparently exhibits Newtonian flow behavior at high temperatures. There is a significant viscosity reduction over the tested temperatures from 80 to 120 °C that can be attributed to the chemical structure of heavy components of the crude oil. While the viscosity appears to slightly decrease at 80 °C with increasing shear rate, the viscosity is constant at 100 and 120 °C. At present, the flow behavior at low temperatures can only be determined at very low shear rates, because of the range of the differential pressure sensor; therefore, it is difficult to analyze the flow behavior over the full shear range. In the future, the measuring range will be extended; thus, extra-heavy oils can be measured also at a high shear rate.

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Table 2. Deviations in Relation to the Standard Reference Values

standard	T in °C	η_{ZMK} in mPa s	$\eta_{capillary\ rheometer}$ in mPa s	deviation in %
1000	20	861.15	843.6 ± 0.011	−2.08
	30	448.53	452.4 ± 0.013	0.85
	40	276.85	271.5 ± 0.010	−1.93
	80	54.67	54.03 ± 0.012	−1.17
	100	30.41	31.02 ± 0.034	1.96
	120	19.07	19.69 ± 0.043	3.14
10000	23	5956	5864.1 ± 0.012	−1.54
	30	3298	3342.5 ± 0.011	1.35
	40	1530	1546.5 ± 0.01	1.08
	60	396	408.9 ± 0.052	3.15
	90	156	155.45 ± 0.031	−0.35
	100	66.42	67.88 ± 0.048	2.20

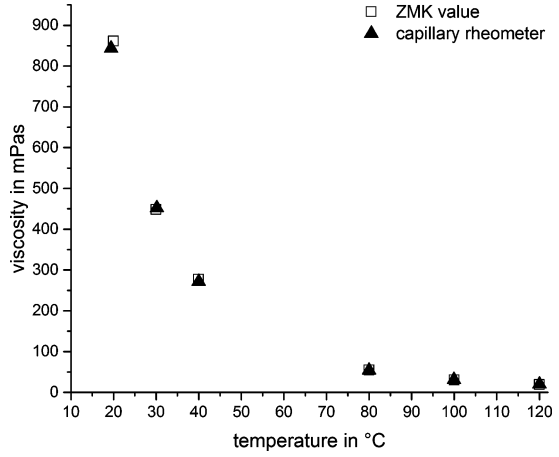

Figure 6. Calibration of the capillary rheometer with a Newtonian standard of 1000 mPa s at different temperatures and atmospheric pressure.

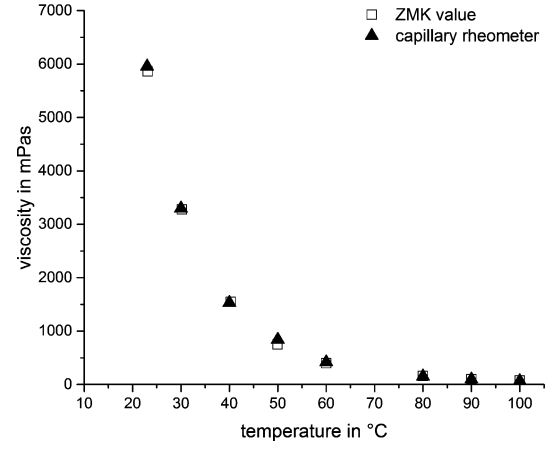

Figure 7. Calibration of the capillary rheometer with a Newtonian standard of 10 000 mPa s at different temperatures and atmospheric pressure.

Furthermore, the pressure influence on the viscosity was determined. Figure 12 illustrates the general trend of the experimental viscosity measurement data at different pressures and temperatures (40, 60, 80, 100, and 120 °C). From this figure, it can clearly be seen that the viscosity increases monotonically with the pressure. At low temperatures, the pressure influence on the viscosity is much bigger than at the higher temperatures. For example, at 40 °C, the viscosity increases 30% by increasing the pressure 100 bar, while at 100 °C, the viscosity increases just 15% by increasing the pressure up to 150 bar.

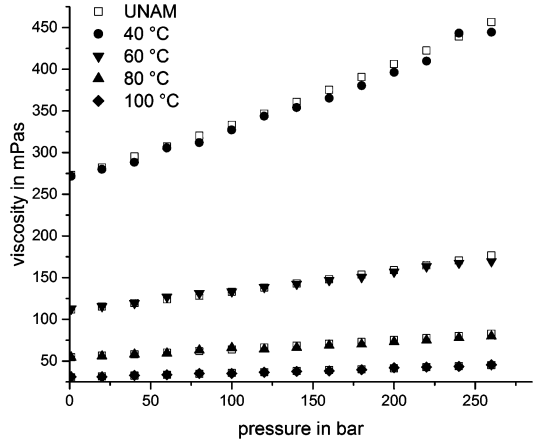

Figure 8. High-pressure results for the 1000 mPa s standard at four different temperatures.

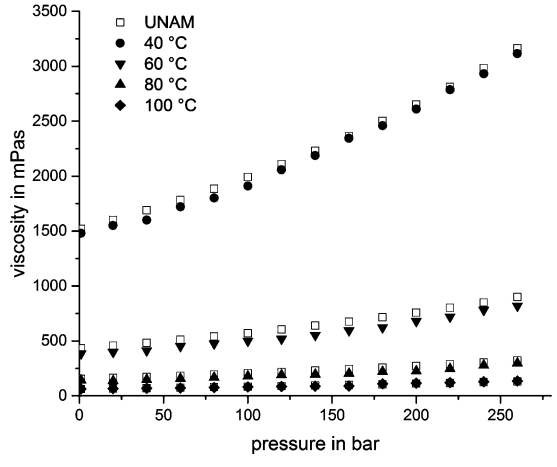

Figure 9. High-pressure results for the 10 000 mPa s standard at four different temperatures.

6. CONCLUSION

The current study is carried out to develop a capillary rheometer for high pressures and temperature applications to be able to investigate rheological properties of heavy crude oils at conditions that are required in the oil industry, from ambient to reservoir conditions. The capillary rheometer was constructed

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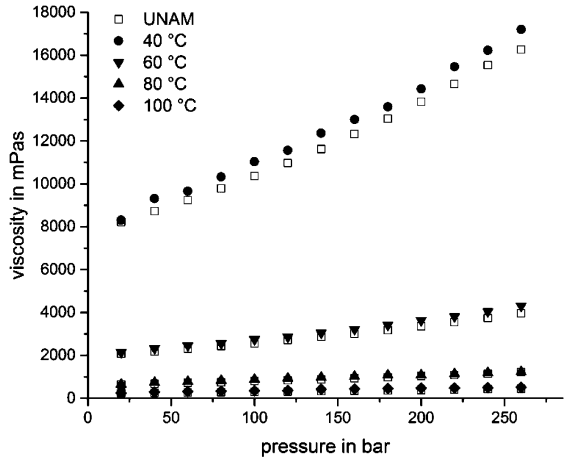

Figure 10. High-pressure results for the 50 000 mPa s standard at four different temperatures.

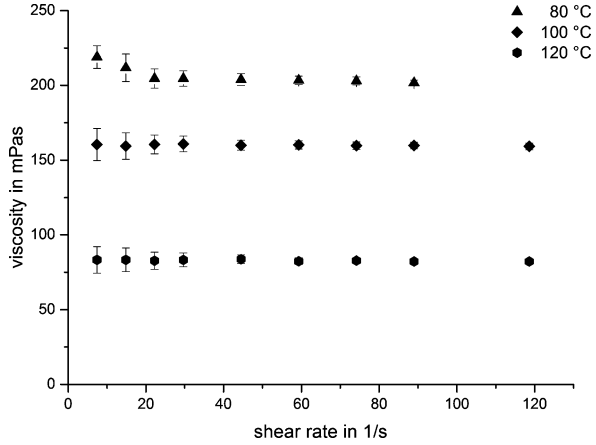

Figure 11. Flow behavior of a heavy oil sample at different temperatures and atmospheric pressure.

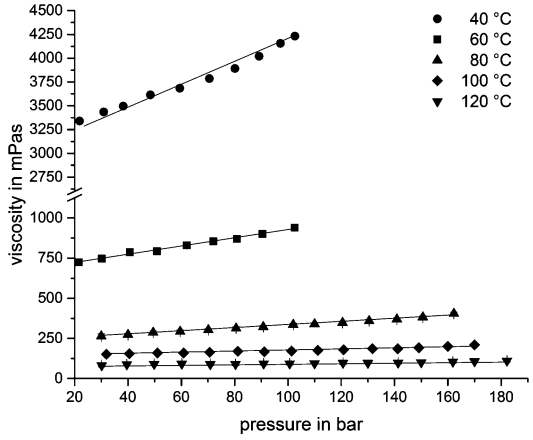

Figure 12. Dynamic viscosity of a heavy oil sample versus pressure at different temperatures.

and manufactured at RUB for measuring viscosity and flow behavior of heavy and extra-heavy oils at high pressure and temperature (up to 300 bar and 200 °C). To measure viscosities with a high accuracy, the capillary rheometer is calibrated with two Newtonian reference blends of different viscosities at different temperatures and atmospheric pressure. The deviation to the reference fluids was found to be lower than 2%.

In the pressure range studied in this work, the apparatus was able to accurately reproduce high-pressure viscosity standards without a need of any pressure correction to the atmospheric calibration. High-pressure viscosity was measured at 40, 60, 80, and 100 °C and pressure from 1 to 260 bar. The measurements agree very well with the tables from UNAM.

After verification of the calibration, the viscosity and the flow behavior of heavy oil were determined at 80, 100, and 120 °C at different shear rates. The heavy crude oil appears to show a Newtonian flow behavior under the conditions examined. Within the studied pressure range, the viscosity increased with increasing pressure at all temperatures. At 40 °C, the viscosity is increased by 30% by increasing the pressure up to 100 bar.

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Notes

The authors declare no competing financial interest.

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