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Rheological behavior from light to heavy oils. Construction of master curves.

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ABSTRACT: In this work, the rheological behavior of heavy and light stabilized (dead) oils is presented. The viscosity measurements were carried out in two different apparatus to ensure the repetitiveness and accuracy: a Stabinger viscometer and an Ares G2 strain controlled rheometer. The samples were previously conditioned with an established methodology. For heavy oils, shear thinning was observed. The zero shear viscosity appears to correlate with American Petroleum Institute (API) gravity for the case of light oils. However, for heavy oils the viscosity definitively does not correlate with the API gravity. For example, the viscosity of fluids with API gravity around 12 may differ up to a full order of magnitude because of differences in molecular structure. SARA analysis is also reported. Viscosity master curves were used to characterize oils. They were produced by applying the time-temperature superposition (TTS) principle to the isothermal shear rate sweeps. Williams, Landel, and Ferry (WLF) equation was used to correlate the shift factor with temperature. Rheological curves for all were quite reproduced with this method all the operational the oils in range.

1. Introduction

Oil still represents 30% of global energy needs. With the combination of an increase in world energy demand and the decline of conventional oils, heavy crude oils have been presented as a relevant hydrocarbon resource for use in the future. Extra-heavy oils and oil sands represent almost 7% of this supply. Oils are usually classified by it's American Petroleum Institute (API) gravity and viscosity.¹ Economical production of heavy and high viscosity oils represents a major challenge for the oil industry. Higher viscosity results in lower throughput volumes, operation expenses increases and larger capital expense projects. In order to achieve exportation as well as refinery feed stream requirements, there is a necessity to mix heavy and light oils.² In this sense, the knowledge of the rheological properties is essential³ for the economically viable production of these non-conventional resources. Under typical circumstances, petroleum fluids might behave as Newtonian fluids, but in the case of heavy oils, these tend to behave as non-Newtonian fluids.⁴ Viscosity data for heavy oils such as Canadian heavy oil and bitumen,^{2, 5-6} Mexican heavy oil⁷⁻⁹ or others¹⁰⁻¹¹ are easily found in literature. Although not as extensive, some rheological behavior of heavy crude oils have also been reported by different authors.^{5, 7, 12} The scope of this work consists on the rheological study of oils at a broad range from light to extra heavy (API from 8 to 40). Most oil production facilities are designed for conventional Newtonian fluids, this work, however, provides extended information on the rheology of non-conventional fluids that is key for the design of pipelines, production facilities as well as for the formulation of heavy oil blends. The measurement of rheological and viscosity properties in heavy oil is usually a very challenging task. Sampling handling, sample restoration and the selection of dewatering process play an important role in obtaining high quality viscosity and rheological data for heavy oils. It is important to ensure that the sub-sample taken out for viscosity and

rheological measurements is properly conditioned and restored. Moreover, the sub-samples need to be properly preserved during the measurements.

In this paper, the rheology of wide varieties of oils from Mexico was investigated. From all results master curves were constructed in order to reproduce values in all the operational range. The procedure called time-temperature superposition¹³⁻¹⁵ is based on the idea that increasing temperature has an equivalent effect on viscosity as decreasing shear rate.¹⁵ If this effect is quantitatively the same at all shear rates, this implies that a single *horizontal shift factor* can be used to shift data on a log-log plot taken at several temperatures along the shear rate axis to coincide with those measured at a *reference temperature* T_0 . This shift factor $a_r(T)$ is given by Equation 1.

$$a_{T}(T) = \frac{h_{0}(T)}{h_{0}(T_{0})}$$
(1)

Since $h_0(T_0)$ is a constant for a given master curve, the shift factor is proportional to $h_0(T)$. Thus, a viscosity *Master Curve* can be prepared by plotting $h(T)/h_0(T)$ versus $\dot{\gamma} \eta_0(T)$.¹⁵

2. Experimental

Twenty different oils from different regions of Mexico were analyzed. All the oils were conditioned in order to eliminate solids and water. Extensive studies of the rheological effect that emulsified water and solid particles have on the rheological hysteresis of the fluids were carried out.¹⁶ Based on these preliminary studies a procedure for conditioning the samples was developed so that no demulsifiers or other chemicals were used in order to ensure that other substances do not tamper the basic rheological properties of the fluids. A wide variety of fluids with an API gravity ranging from 8 to 40, a density ranging from 0.81 to 1.01 g/cm³ at 20°C and a viscosity ranging from 0.002 to

700 Pas at 20°C were studied (see Table 1). The API gravity, density and zero shear viscosity were all measured with an Anton-Paar Stabinger Viscometer SVM3000, which measures the properties of oils and fuels according to ASTM D7042. The precision of the instrument was set to standard mode and the time required for the proof depended on the viscosity of oil (normally between one and two hours). One advantage of this instrument is that it consists of a closed geometry made of a rotational cylinder inside a tube, which also provides the shear rate at which the viscosity is measured. The same sample is placed in an Ares G2 rheometer. The geometry chosen depends on the viscosity of the fluid. If the oil is extra heavy, parallel plate geometry is necessary, if the oil is heavy to medium the bob and cup single wall geometry is used and if the oil is light the double wall bob and cup geometry is required. The Ares G2 rheometer with double wall bob and cup geometry can accurately measure viscosities as low as 0.002 Pas. For extra light fluids, however, Newtonian behavior prevails and the Stabinger measurements suffice.

The range of operational temperatures goes from 20 to 100 °C, however, Ares G2 geometries are open and sometimes it is not possible to measure the rheology of fluids that may boil below 100 °C. The reproducibility of the rheometric measurements was better than 2%, and the accuracy better than 0.5% for standards of calibration; the stabilization time for the temperature was set to five minutes before each measurement. Rates of shearing vary between 0.1 to 1000 1/s, depending on the oil viscosity. The viscosity measured with Stabinger viscometer is compared with the Ares G2 rheometer viscosity; if measurement is correct and there is no loss of light end compounds both values must coincide. If measurement is performed on a fluid susceptible of losing light ends, the rheometric measurement will be slightly above the Stabinger results. To solve this problem, master curves for all fluids are made in order to extend the applicability of the measurements.

3. Results and discussion

Physical properties for 20 oils are summarized in Table 1.

Table 1. API gravity, ρ (density) and $\eta 0$ (apparent viscosity) for twenty oils.

for twenty ons.						
Oil	API gravity grades	ρ (20°C) g/cm ³	η (20°C) Pa.s			
1	12.6	0.9811	20.03			
2	12.2	0.9834	42.00			
3	19.5	0.9360	0.41			
4	12.3	0.9832	44.47			
5	18.5	0.9426	0.65			
6	19.3	0.9376	0.44			
7	20.3	0.9312	0.28			
8	12.9	0.9787	6.43			
9	7.3	1.0182	457.33			
10	12.4	0.9823	15.19			
11	12.6	0.9808	12.87			
12	7.8	1.0150	688.97			
13	8.6	1.0094	140.77			
14	38.4	0.8321	0.0046			

15	40.3	0.8227	0.0023
16	31.4	0.8679	0.0109
17	29.7	0.8769	0.0163
18	40.4	0.8225	0.0032
19	10.0	0.9994	207.66
20	21.4	0.9245	0.17

As observed, the oils vary from extra light oils (Oil 15) to extra heavy oils (Oil 12). In Figure 1, a set of curves of viscosity as a function of the temperature from oils of different API gravities is shown. These curves were obtained from the measurements in Stabinger Viscometer. Viscosity varies in five decades, which turns this work very interesting because of the broad range of viscosities. Heavy oils tend to decrease their viscosity more than 100 times with the increment of temperature; meanwhile, light oils do not decrease too much their viscosity when increasing temperature.



Figure 1. Viscosity curves from 20 to 100°C for: •: Oil 2, **•**: Oil 11, • : Oil 5, **•**: Oil 17, **•**: Oil 18.

The rheology was obtained only for oils with a viscosity higher than 0.016 Pas, assuming that oils below this viscosity have Newtonian behavior for all temperatures. Above this value, the rheometer can accurately measure the viscosity and oils may or may not behave as Newtonian.



Figure 2. Stabinger (■) and ARES (●) measurements results of Oil 7 rheology as a function of temperature.

In Figure 2, rheological flow curves as a function of temperature for Oil 7 are shown. The API gravity of this oil is 20.3 and it behaves as Newtonian fluid, i.e., its viscosity does not change with shear rate. The results obtained with Stabinger and ARES rheometer coincide, which ensure the accuracy of the measurements. This oil starts boiling before 100 °C, so the rheological curve for this temperature cannot be measured with this open rheometer. For 80 °C it is observed that Stabinger is slightly below the ARES G2 result due to the loss of light compounds.

Non-Newtonian behavior appears for some oils at temperatures below 40 °C.¹⁶ The 20 °C isotherms for five non-Newtonian heavy oils are illustrated in Figure 3. Each oil has a Newtonian regime at low shear rates and as shear rate increases, the viscosity decreases, exhibiting shear thinning. This thinning is the so-called non-Newtonian behavior. The rheological curves observed in Figure 3 correspond to heavy oils with almost same API gravity (around 12), but some of them have up to three times difference in viscosity.



Figure 3. Rheological behavior at 20°C for: (●) Oil 2, (■) Oil 4, (♦) Oil 1, (▲) Oil 10, and (▶) Oil 11.

This contrast with the fact that viscosity is many times correlated with API gravity.¹⁷⁻²⁰ In the case of light oils this is a reasonable approximation but it is not the case for heavy oils. As the fluids become heavier (lower API gravity), the viscosity strongly deviates from any possible correlation simply based on the API gravity. In Figure 4 zero shear viscosity at 20 °C as a function of API gravity is shown for all the oils. The zoom in both axes from Figure 4 shows five oils with similar values in API gravity but with very different values in viscosity. It is then not correct to correlate viscosity directly with API gravity for crude heavy oils, it has to be taken into account the chemical structure of the fluids in terms of saturates, aromatics, resins and asphaltenes (SARA) content. SARA analysis for the twenty oils can be observed in Table 2.



Figure 4. Zero shear viscosity as a function of API gravity for all oils at 20°C. The zoom stands for oils with API around 12°.

Table	2.	SARA	content	for	twenty	oils	with	API	gravity
aroun	d 12	2°.							

Oil	Saturates	Aromatics	Resins	Asphaltenes	
0.11	(wt%)	(wt%)	(wt%)	(wt%)	
1	24.3	28.0	30.7	14.5	
2	30.4	25.1	23.0	19.1	
3	36.0	27.6	25.8	10.5	
4	26.4	30.6	27.3	18.5	
5	39.5	24.1	23.9	10.0	
6	32.8	31.6	24.8	10.0	
7	38.2	35.1	18.6	7.6	
8	28.4	34.0	23.7	13.7	
9	17.8	34.9	34.4	12.2	
10	21.4	37.2	28.5	12.2	
11	22.4	34.7	28.9	12.0	
12	24.8	31.9	28.0	17.1	
13	26.9	33.8	24.7	12.2	
14	75.5	17.7	5.0	0.12	
15	77.5	13.4	9.5	0.05	
16	61.5	27.4	7.6	2.2	
17	56.6	28.1	13.8	0.8	
18	72.8	17.6	7.2	0.62	
19	23.2	23.2	32.8	17.9	
20	50.0	29.9	13.7	5.3	

In Figure 5, viscosity of oils is presented as a function of the content of asphaltenes and saturates. The non-Newtonian behavior seems to appear in oils with high content of asphaltenes (above 10%).



Figure 5. Zero shear viscosity as a function of (\bullet) Asphaltenes content, (\bullet) Saturates content at 20°C.

4. Construction of Master Curves

In Table 3, experimental data for Oil 11 is shown in the first four columns. The reference viscosity η_0 is the zero shear viscosity measured with Stabinger Viscometer for each temperature. Next two columns are obtained by arithmetic calculations. Viscosity master curves were prepared by plotting η/η_0 versus $\dot{\gamma} \eta_0$, see Figure 6.

To fit points, an empirical approximation was used:

$$\frac{\eta(T)}{\eta_0(T)} = \frac{b_1}{1 + b_2 \dot{\gamma} \eta_0} + C_1 \tag{2}$$

where b_1 , b_2 and c_1 are fitting constants. According to Equation 2, when shear rate is very low, viscosity tends to a constant; which is the zero shear viscosity. For greater values of shear rate, the viscosity decreases (shear-thinning). A good fit between data for Oil 11 (Table 3) and Equation 2 can be observed in Figure 6.



Figure 6. Reduced viscosity versus reduced shear rate. Data (symbols) and fit with Equation 2 (line).

Constants b_1 , b_2 and c_1 for Oil 11 were found to be 0.9426, 1.68x10⁻⁴ and 0.06282 respectively. The value R² was 0.9993. The resultant curve is the so-called *Master Curve* and is useful

to reproduce the rheological flow curves of an oil. The applicable shear rate can be extended beyond the range actually accessible using a given viscometer. The range of temperatures goes from the melting point to the temperature at which the fluid starts to decompose.¹⁵

Table 3. Rheological data for Oil 11.

	Shoor	Vis-				
Т	roto	cosity	n_0		h	
°C	Tale y	n	(Pa.s)	$\gamma \eta_0$	$h_{\rm e}$	a_{T}
	(1/s)	(Pa.s)	(1 415)		0	
20.0	0.25	13.62	12.87	3.23	1.06	1.00
20.0	0.39	13.61	12.87	5.12	1.06	1.00
20.0	0.63	13.59	12.87	8.12	1.06	1.00
20.0	1.00	13.57	12.87	12.87	1.05	1.00
20.0	1.59	13.55	12.87	20.40	1.05	1.00
20.0	2.51	13.50	12.87	32.33	1.05	1.00
20.0	3.98	13.43	12.87	51.24	1.04	1.00
20.0	6.31	13.32	12.87	81.21	1.04	1.00
20.0	10.00	13.07	12.87	128.72	1.02	1.00
20.0	15.85	12.64	12.87	204.00	0.98	1.00
20.0	25.12	12.23	12.87	323.32	0.95	1.00
20.0	39.81	11.79	12.87	512.43	0.92	1.00
20.0	63.10	11.40	12.87	812.14	0.89	1.00
20.0	100.01	10.85	12.87	1287.15	0.84	1.00
40.0	0.63	2.04	1.97	1.24	1.04	0.15
40.0	1.00	2.02	1.97	1.97	1.02	0.15
40.0	1.59	2.01	1.97	3.12	1.02	0.15
40.0	2.51	2.00	1.97	4.95	1.01	0.15
40.0	3.98	1.99	1.97	7.84	1.01	0.15
40.0	6.31	1.98	1.97	12.43	1.01	0.15
40.0	10.00	1.97	1.97	19.70	1.00	0.15
40.0	15.85	1.97	1.97	31.23	1.00	0.15
40.0	25.12	1.96	1.97	49.49	0.99	0.15
40.0	39.81	1.95	1.97	78.44	0.99	0.15
40.0	63.10	1.94	1.97	124.31	0.98	0.15
40.0	100.01	1.91	1.97	197.02	0.97	0.15
60.0	1.00	0.48	0.48	0.48	1.00	0.04
60.0	1.58	0.48	0.48	0.76	1.00	0.04
60.0	2.51	0.48	0.48	1.21	0.99	0.04
60.0	3.98	0.48	0.48	1.91	0.99	0.04
60.0	6.31	0.47	0.48	3.03	0.99	0.04
60.0	10.00	0.47	0.48	4.80	0.99	0.04
60.0	15.85	0.47	0.48	7.61	0.99	0.04
60.0	25.12	0.47	0.48	12.06	0.99	0.04
60.0	39.81	0.47	0.48	19.11	0.98	0.04
60.0	63.10	0.47	0.48	30.29	0.98	0.04
60.0	100.01	0.47	0.48	48.01	0.98	0.04
80.0	1.00	0.17	0.16	0.16	1.03	0.01
80.0	1.59	0.16	0.16	0.25	1.03	0.01
80.0	2.51	0.16	0.16	0.40	1.02	0.01
80.0	3.98	0.16	0.16	0.64	1.02	0.01
80.0	6.31	0.16	0.16	1.01	1.03	0.01
80.0	10.00	0.16	0.16	1.60	1.03	0.01
80.0	15.85	0.16	0.16	2.54	1.03	0.01
80.0	25.12	0.16	0.16	4.02	1.03	0.01
80.0	39.81	0.16	0.16	6.37	1.03	0.01
80.0	63.10	0.16	0.16	10.10	1.02	0.01
80.0	100.01	0.16	0.16	16.00	1.02	0.01

The horizontal shift factor a_T is calculated with Equation 1; in this case, the reference temperature was set to 20 °C. In Table 3 the shift factor calculated for Oil 11 is presented. In order to correlate the shift factor with temperature, the Williams, Landel, and Ferry (WLF) equation^{13, 21} was used:

$$\log a_T = -\frac{d_1(T-T_0)}{[d_2 + (T-T_0]]}$$
(3)

where d_1 and d_2 are fitting constants. Figure 7 shows the shift factor against temperature for Oil 11.



Figure 7. Shift factor as a function of temperature for Oil 11. Data (symbols) and fit with Equation 3 (line).

For Oil 11, d_1 and d_2 were found to be 12.9289 and 117.919 respectively. The value R^2 was 1.00. With this fit, the reference viscosity for all temperatures can be calculated as:

$$\eta_0(T) = a_T \eta_0(T_0) \tag{4}$$

Viscosity and rheological flow curves for Oil 11 measured with Stabinger viscometer and ARES G2 rheometer, respectively, are depicted in Figure 8. These values are shown up to $T = 80^{\circ}$ C, after this temperature this oil starts boiling at atmospheric pressure and viscosity is no longer measurable with any apparatus. Non-newtonian behavior can be observed for $T = 20^{\circ}$ C.

In Figure 8, rheological flow curves reproduced with master curve are also presented. Zero shear viscosity for temperatures 30, 50, 70, 90 and 100°C were calculated with Equation 4. Viscosity was calculated with Equation 2 for shear rates even greater than operational range. As observed, master curve is capable to reproduce the shear thinning behavior for low temperatures even if no data is available (case of T = 30° C), but also can reproduce the Newtonian behavior for greater temperatures. Moreover, a second Newtonian plateau can also be obtained with the aim of the master curve.

Characterization described for Oil 11 was repeated for all the oils. Master curves and shift factors were obtained for each one of them. Rheological curves for all the oils were well reproduced with this method for all the temperatures and shear rate ranges. A relationship with viscosity and shift factor was found.



Figure 8. Zero shear viscosity obtained from Stabinger (\blacksquare), viscosity as a function of shear rate obtained with rheometer Ares G2 (\bullet) and rheological flow curves obtained from master curve (line) for Oil 11.

As shown in Figure 9, the shift factor from light to extra heavy oils decrease as viscosity increases. That means that an extra heavy oil will decrease its viscosity even three decades less with the effect of temperature than a medium or heavy oil.

Figure 9. Shift factor in logarithm scale as a function of temperature for heavy and extra heavy oils.

5. Conclusions

Viscosity and rheological behavior were studied in detail for 20 different oils with API gravity ranging from 8 to 40 degree. The quality and validity of zero-shear viscosity measurements was checked between different apparatus, finding an overall standard deviation of less than 10%. For five heavy oils close to API gravity of 12, the viscosity was very different from each other due to the difference in their molecular structure. SARA analysis for all the oils was reported. Non-Newtonian behavior was found in oils with more than 10% asphaltenes content. Viscosity master curves were constructed for all the oils with the time-temperature superposition procedure (explained in detail for Oil 11). Rheological flow curves for all the oils were accurately obtained in the full range of operation. Shift factor curves for all the oils were plotted as a function of

temperature. A relationship with viscosity was found: the higher the oil's viscosity, the higher the decrease in its viscosity.

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