



## Flow Rate Calculations Based on Rheometric Measurements of Model Waxy Oils at Different Surface Roughness

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### Abstract

Waxy gels can be formed during offshore production upon the cooling of waxy crude oils at sufficiently low temperatures. This is particularly relevant to the petroleum industry, as severe issues can arise in the field due to precipitation and aggregation of paraffin wax crystals during the production, storage, and transportation of crude oils. In this study, the effects of apparent wall slip on rheometric measurements were quantified and flow rate calculations were performed for model waxy gels consisting of a macrocrystalline wax added to mineral oil (3.0 and 7.5 wt%). The gels were formed *in situ* in a stress-controlled rheometer, and rheological properties were obtained by oscillatory and steady-state experiments. Different geometry configurations, including smooth and grooved concentric cylinders, were employed. Flow curves were adjusted by Power Law and Herschel-Bulkley models. The calculated parameters were used in modified Poiseuille equations to estimate the flow rate for specific pumping conditions and pipeline length. The rheological data provided with smooth surfaces were greatly affected by the apparent wall slip (e.g., yield stress ~ 80% smaller) and it is reflected by unrealistic flow rate calculations. By quantifying the slippage effects, this work can provide useful information for the design of pipelines and oil transportation systems.

### Keywords

yield stress; apparent wall slip; flow rate

### Introduction

Waxy gels can be formed upon the cooling of crude oils containing paraffinic waxy compounds due to crystals precipitation at temperatures below the Wax Appearance Temperature (WAT)<sup>[1,2]</sup>. In this context, severe issues can arise in the field during crude oil offshore production since petroleum can undergo a gelation process, and the pipelines can become clogged with waxy gels<sup>[3-5]</sup>. These gels present elasto-viscoplastic thixotropy behavior that needs to be accurately measured for the proper design of transport processes involving these materials<sup>[4,5]</sup>. In this context, the yield stress ( $\sigma_y$ ) of a waxy gel is a critical property, as it effectively determines the pump capacity required to initiate or restart the pipeline flow<sup>[5]</sup>.

One big challenge in the correct characterization of waxy gels rheological behavior is the apparent wall slip during rheometric tests, which can lead to underestimated values of yield stress, and viscosity and affect data reproducibility<sup>[6]</sup>. A decrease of 60 to 85% in the yield stress of different systems is reported in the literature<sup>[7,8]</sup>. Therefore, the subsequent steps for the pipeline

design can be affected by this experimental artifact to a great extent.

In this study, the slippage effects in rheometric measurements were quantified for model waxy gels consisting of a macrocrystalline wax (3.0 and 7.5 wt%) added to a low-viscosity spindle mineral oil. Dynamic oscillatory tests and steady-state tests were employed to assess the sample's rheological behavior. The slippage effects were also accounted for in pressure drop and flow rate calculations through the balance between friction and pressure forces (Eq. 1) and modified Poiseuille equations (Eqs. 3-4).

### Methodology

Model oils were freshly prepared under controlled conditions before each rheological test to avoid thermal and shear histories variation. Linear macrocrystalline wax (29 carbons on average) with a melting temperature of 56-58°C was purchased from Sigma-Aldrich. Wax weight fractions of 3.0 wt% and 7.5 wt% were employed. After preparation, samples were immediately placed on the DHR-3 controlled-stress rheometer (TA

Instruments). Dynamic oscillatory tests were employed to assess the yield stress and steady-state tests were performed to assess the flow curves of gelled systems. Both protocols were conducted using concentric cylinder geometries with different surface roughness. The complete experimental setup and the wax and mineral oil characterization can be found elsewhere<sup>[9]</sup>.

The pressure drop ( $\Delta P$ ) needed for gelled structure breakage is based on Eq.(1), where the clogged pipe length and radius are  $L$  and  $R$ , respectively:

$$\Delta P = 2\sigma_y \frac{L}{R} \quad (\text{Eq. 1})$$

The wall shear rate ( $\dot{\gamma}_w$ ) imposed in the oil due to pipeline flow (considering a Newtonian fluid) can be obtained from Eq.(2) for a pipe of diameter  $D$ :

$$\dot{\gamma}_w = \frac{32Q}{\pi D^3} \quad (\text{Eq. 2})$$

The oil flow rate ( $Q$ ) after the gel breakage can be estimated from Eq.(3) and Eq.(4), considering Power Law and Herschel-Bulkley (HB) models, respectively.

$$Q = \pi \left( \frac{n}{3n+1} \right) R^{(3+\frac{1}{n})} \left( \frac{-\Delta P}{2kL} \right)^{\frac{1}{n}} \quad (\text{Eq. 3})$$

In Eq.(3),  $k$  is the consistency index ( $\text{Pa}\cdot\text{s}^n$ ) and  $n$  is the flow behavior index (dimensionless).

$$Q = n\pi R^3 \left( \frac{\sigma_{HB}}{m\varphi} \right)^{\frac{1}{n}} (1-\varphi)^{\left(\frac{n+1}{n}\right)} S \quad (\text{Eq. 4})$$

$$S = \left[ \frac{(1-\varphi)^2}{3n+1} + \frac{2\varphi(1-\varphi)}{2n+1} + \frac{\varphi^2}{n+1} \right]$$

In Eq.(4)  $m$  is similar to parameter  $k$  and  $\varphi$  is the ratio of the yield stress calculated from the Herschel-Bulkley model ( $\sigma_{HB}$ ) and the stress at the pipe wall, given by Eq.(1).

## Results and Discussion

Rheological oscillatory measurements provided yield stress data with geometries of different surface roughness (Fig. 1). The results are summarized in Tab.1 for model oils with 3.0 wt% wax (named as Fluid A) and model oils with 7.5 wt% wax (named as Fluid B). As can be observed, there is an astonishing decrease of ~ 80% in yield stress measurement for the complete smooth geometry (SC+SC) compared to the complete grooved geometry (GC+GC) regardless of the system composition, as already observed in our previous study<sup>[8]</sup>.

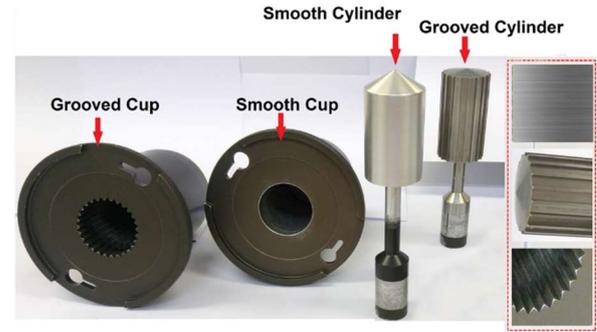


Figure 1. Concentric cylinders and surface details. Slippage effects were observed when smooth surfaces were employed in rheometric tests. Adapted from Marinho et. al<sup>[8]</sup>

Table 1. Yield stress from dynamic oscillatory tests

Fluid	Geometry setup	$\sigma_y$ (Pa)
A	Smooth Cylinder + Smooth Cup	40 ± 8
	Grooved Cylinder + Smooth Cup	64 ± 20
	Grooved Cylinder + Grooved Cup	259 ± 24
B	Smooth Cylinder + Smooth Cup	488 ± 100
	Grooved Cylinder + Smooth Cup	696 ± 130
	Grooved Cylinder + Grooved Cup	2,700 ± 252

In Fig. 2 the pressure drop required to restart a pipeline ( $\Delta P_{REQ}$ ) with  $D = 8''$  in and clogged length varying from 100 to 3,000 m is exhibited for model oils with 3.0 wt% and 7.5 wt% wax. Assuming a maximum available pressure ( $\Delta P_{AVA}$ ) of 300 bar for the model oil 3.0 wt% wax, the flow at a clogged pipeline with up to 2,250 m could be restarted, considering the cohesive breakage of the gelled waxy structure (i.e., for measurements with GC+GC geometry,  $\sigma_y = 259$  Pa). For systems with 7.5 wt%, considering the cohesive gel breakage, the maximum length is only 570 m. This highlights the role of the wax content on the flow restart. If the estimates were based on yield stress measurements employing only smooth surfaces (SC+SC), the  $\Delta P_{REQ}$  would be lower than  $\Delta P_{AVA}$  for the entire range of pipeline length. For waxy crude oils the yield stress depends mainly on paraffin content, thus underestimated yield stress represents a major drawback to estimate the restart pressure drop.

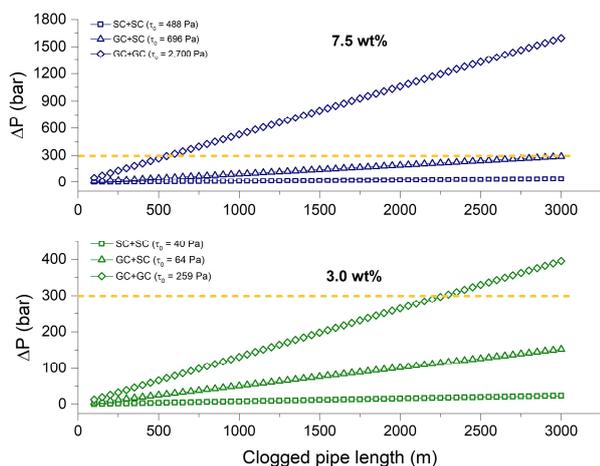


Figure 2. Pressure drop estimation for restarting the flow of clogged pipeline with  $D = 8''$ , model oil composed of 3.0 wt% and 7.5 wt% wax, and different cylinders surfaces

Fig. 3 exhibits the  $\Delta P_{REQ}$  for restarting 100 m of clogged line as a function of pipe diameter. It is important to stress that pipes with diameters ranging from 4" to 8" are commonly encountered at offshore production operations. Considering the model oil with 3.0 wt% wax, for pipe with a relatively large diameter, e.g., 10 inches,  $\Delta P_{REQ}$  varies from 0.66 to 4.1 bar/100m for SC+SC and GC+GC, respectively. For the 7.5 wt% solution in a 2" pipe, this difference is 174 bar/100m. As one can observe, the  $\Delta P_{REQ}$  progressively diverge as pipe diameter decreases and the wax content increases, representing a bottleneck for the correct pipeline design if surface roughness is not considered.

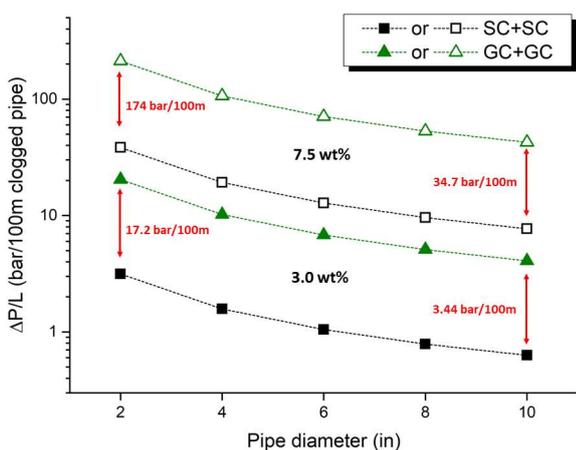


Figure 3. Pressure drop for restart flow of clogged pipelines with  $D = 2, 4, 6, 8,$  and  $10''$ , model oil composition of 3.0 wt% (full symbols) and 7.5 wt% (open symbols), and different concentric cylinder surfaces

Model oils flow behavior was assessed through steady-state rheological experiments<sup>[8]</sup>. Fig. 4 exhibits shear stress and viscosity curves for the model oil 7.5 wt% wax. The slippage effect is clear when smooth surfaces (full symbols) are compared

to grooved surfaces (open symbols). The presence of kinks (red circles) and the lower values of rheological properties have a great impact on flow rate calculations as discussed next.

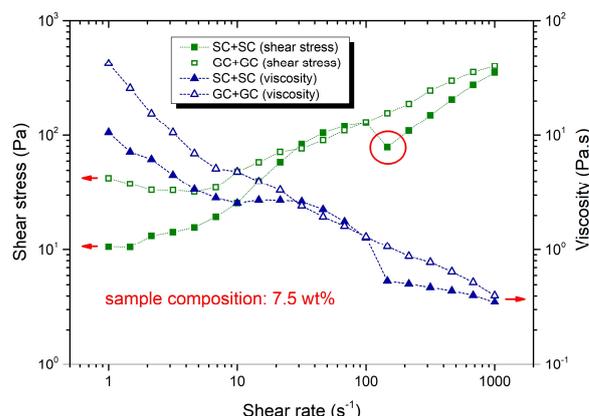


Figure 4. Flow curves and viscosity curves for model waxy oil 7.5 wt% assessed with different cylinders surfaces

The parameters for Power Law and Herschel-Bulkley models were obtained based on the flow curves (Fig. 4) in the range of 1.0 to 1,000  $s^{-1}$  and they are grouped in Tab. 2. The distance of the data to the fitted regression line is evaluated by the  $R^2$  value. It is worth mentioning that the parameter  $\sigma_{HB}$  corresponds to the dynamic yield stress, as introduced by Boger et al.<sup>[2]</sup> to describe the yielding after gel breakage. The previously mentioned oscillatory measurements represent the stress necessary to break waxy gelled structure, that is why those values are up to 52 times higher.

Table 2. Power Law and HB model parameters'

Power Law		
Model oil	SC+SC	GC+GC
3.0 wt% wax	( $R^2 = 0.93$ )	( $R^2 = 0.88$ )
k [Pa.s <sup>n</sup> ]	7.45	21.9
n [-]	0.206	0.231
Model oil	SC+SC	GC+GC
7.5 wt% wax	( $R^2 = 0.95$ )	( $R^2 = 0.96$ )
k [Pa.s <sup>n</sup> ]	29.5	68.1
n [-]	0.197	0.205
Herschel-Bulkley		
Model oil	SC+SC	GC+GC
3.0 wt% wax	( $R^2 = 0.94$ )	( $R^2 = 0.94$ )
m [Pa.s <sup>n</sup> ]	3.68	2.65
n [-]	0.436	0.604
$\sigma_{HB}$ [Pa]	3.55	44.4
Model oil	SC+SC	GC+GC
7.5 wt% wax	( $R^2 = 0.95$ )	( $R^2 = 0.97$ )
m [Pa.s <sup>n</sup> ]	4.86	4.01
n [-]	0.607	0.661
$\sigma_{HB}$ [Pa]	15.3	52.4

The calculated flow rate after gel breakage, according to the Power Law model (Eq. 3), is presented in Fig. 5 as a function of pipeline length (1,000 to 4,500 m), considering 8" pipe diameter

and  $\Delta P_{AVA} = 300$  bar. As one can observe, for 3.0 wt% model oil there is an average decrease in flow rate of  $10^3$  m<sup>3</sup>/day comparing the parameters from SC+SC and GC+GC experiments. For 7.5 wt% this difference is about  $10^2$  m<sup>3</sup>/day. Since the same experimental protocol was applied to all tests, the slippage effect on rheometric measurements is the main responsible for these differences. Despite the huge decrease, all curves presented unrealistic flow rate values. This situation occurs because, after gel breakage, the pseudoplastic behavior of the waxy oil becomes very sharp (Fig. 4) and the Power Law model does not limit the viscosity to a minimum value. Thus, mathematically the shear rate can go to infinity, and so the flow rate. Given the behavior of waxy oils after breakage, is likely that the flow curves provide very low  $n$  parameters in most situations (e.g., 0.30 to 0.10), due to the pronounced pseudoplastic behavior. For comparison purposes, a polymer melt with parameters  $k = 150$  Pa.s<sup>0.85</sup> and  $n = 0.85$ <sup>[10]</sup> was added to Fig. 5 and feasible flow rate values were calculated. The extremely high viscosity during flow and the behavior like the purely viscous fluid ( $n \sim 1$ ) contributed to this result.

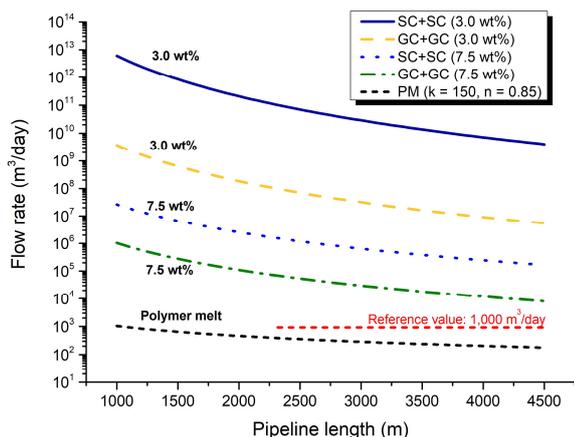


Figure 5. Flow rate calculations based on the Power Law model for the 3.0 wt% and 7.5 wt% waxy oils and pipeline length ranging from 1,000 to 4,500 m,  $D = 8''$ ,  $\Delta P_{AVA} = 300$  bar

Another approach to estimating the flow rate by Power Law or Herschel Bulkley models is to perform a flow curve experiment in reverse mode (i.e., starting from a high shear rate and descending to lower values). At the beginning of the experiment, the high shear imposed on the samples can suppress the slippage effects, because the fluid is completely unstructured as in Fig. 6. In this case, the cylinder surfaces are almost equivalent at the flow measurement in the range of 100 to 1 s<sup>-1</sup>. Although, due to the thixotropy behavior of the samples, there is a clear deviation for shear rates lower than 1 s<sup>-1</sup>. The stress is higher for the grooved surfaces, then higher viscosities are captured by this geometry.

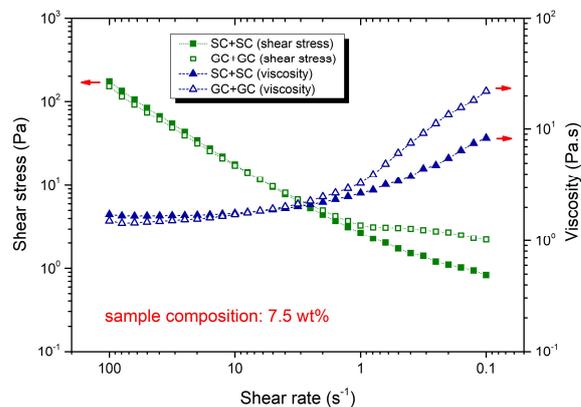


Figure 6. Flow curves and viscosity curves in reverse mode for model waxy oil 7.5 wt% assessed with different cylinders surfaces

Figure 7 exhibits the flow rate in m<sup>3</sup>/day as a function of pipeline length, considering 8" pipe diameter and  $\Delta P_{AVA} = 300$  bar. The steady-state experiments were performed in the range of 100 to 0.1 s<sup>-1</sup>. Compared to Fig 5., the flow rates were substantially lower, although once again yielding unrealistic values. Consequently, the Power Law model is not recommended for flow rate estimates involving waxy oils. Despite this fact, Fig. 7 provides useful information: (i) surface roughness is still an important issue, as calculations based on grooved geometries provided lower flow rate values; (ii) starting the rheological test from 100 s<sup>-1</sup> produces a highly sheared structure in a very short time, thus the pseudoplastic behavior is attenuated and  $n$  values are closer the unity ( $> 0.92$  for all cases), contributing to lower flow rates, as  $Q \sim 1/n$ ; (iii) as apparent wall slip is more pronounced in low shear rates<sup>[2,6]</sup> (e.g.  $< 1.0$  s<sup>-1</sup>), the difference in flow rates for SC+SC and GC+GC is also lower (comparing the same oil wax content). This reflects the fact that the fluid structure was already highly sheared when the experiment reached 1 s<sup>-1</sup>.

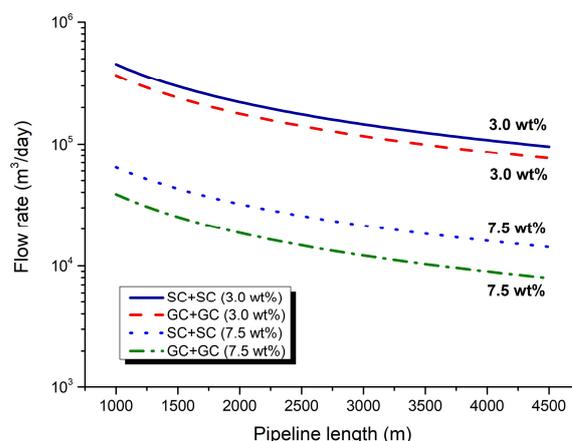


Figure 7. Flow rate calculations based on the Power Law model (reverse flow curves) for waxy oil 3.0 and 7.5 wt% and pipeline varying from 1,000 to 4,500 m,  $D = 8''$ ,  $\Delta P_{AVA} = 300$  bar

The Herschel-Bulkley model was also employed to estimate the flow rate after gel breakage of 7.5 wt%

model oil for pipelines ranging from 1,000 to 4,500 m, considering 8" pipe diameter and  $\Delta P_{AVA} = 300$  bar (Fig. 8). The estimates from direct flow curves (1.0 to 1,000  $s^{-1}$ ) were compared with reverse flow curves (100 to 0.1  $s^{-1}$ ) for different surface roughness. The behavior was the one depicted in Fig. 7, and the same conclusions stand in this case, i.e., due to apparent wall slip the flow rate is exceedingly overestimated, and the difference becomes less pronounced when reverse flow curves parameters are employed.

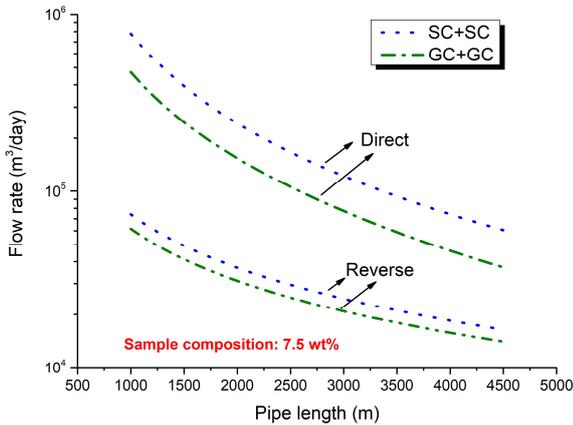


Figure 8. Flow rate calculations based on the Herschel-Bulkley model for direct and reverse flow curves, waxy model oil 7.5 wt% and pipeline length ranging from 1,000 to 4,500 m,  $D = 8''$ ,  $\Delta P_{AVA} = 300$  bar

Unlike the Power Law model, Herschel-Bulkley has the parameter  $\sigma_{HB}$ , which can be interpreted as dynamic yield stress<sup>[2]</sup>. To obtain this parameter precisely the flow curve must be in the steady-state, especially for lower shear rates as 0.001  $s^{-1}$ . Regardless of the geometry surface employed, it is difficult to achieve a steady-state at a such lower rate for complex fluids, due to elasto-viscoplastic thixotropy behavior and rheometer limitations (torque and angular position sensors have limited sensibility). Therefore, it is likely that true  $\sigma_{HB}$  is higher than the observed in the flow curves assessed in this study (limited to 1.0  $s^{-1}$ ). In this regard, Fig. 9 exhibits flow rate estimates for progressively higher  $\sigma_{HB}$  for model oil 7.5 wt% wax, with  $m = 4.01 Pa \cdot s^{0.661}$  and  $n = 0.661$  (Tab.1.), considering  $\Delta P_{AVA} = 300$  bar. The pipeline length becomes relevant for  $\sigma_{HB} = 488$  Pa and  $\sigma_{HB} = 696$  Pa, precisely the static yield stress measured from oscillatory tests for SC+SC and GC+SC geometries, respectively. Thus, one more time the slippage effects is being present in HB modeling. For  $\sigma_{HB} = 2,700$  Pa no flow rate was obtained because, in such a situation,  $\sigma_{HB}$  overcomes the shear stress at the pipe wall. According to the calculation, the maximum pipeline length for observable flow rate ( $> 0.1 m^3/day$ ) is 3,100 m for  $\sigma_{HB} = 488$  Pa and 2,150 m for  $\sigma_{HB} = 696$  Pa.

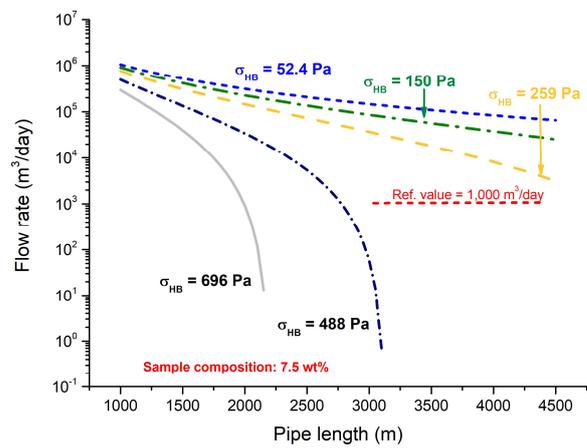


Figure 9. Flow rate based on the Herschel-Bulkley model with several  $\sigma_{HB}$  values ( $m = 4.01 Pa \cdot s^{0.661}$ ,  $n = 0.661$ ) for waxy oil 7.5 wt% and pipeline varying from 1,000 to 4,500 m,  $D = 8''$ ,  $\Delta P_{AVA} = 300$  bar

Finally, Fig. 10 exhibits the shear rate calculated from Eq. 2 for 7.5 wt% composition for Power Law and Herschel-Bulkley models. The parameters were taken from Tab. 2, except for  $\sigma_{HB}$  values (depicted in Fig. 9). The pipeline flow is associated with shear rates ranging from 10 to 1,000  $s^{-1}$ [5]. As one can observe, both models suffer from the same unbounded viscosity issue, leading to exceedingly high flow rates. Although, the parameter  $\sigma_{HB}$  helps the Herschel-Bulkley model to provide more realistic estimates, i.e., with shear rate (and therefore flow rate) tending to zero for a sufficient long pipeline, given realistic yield stresses.

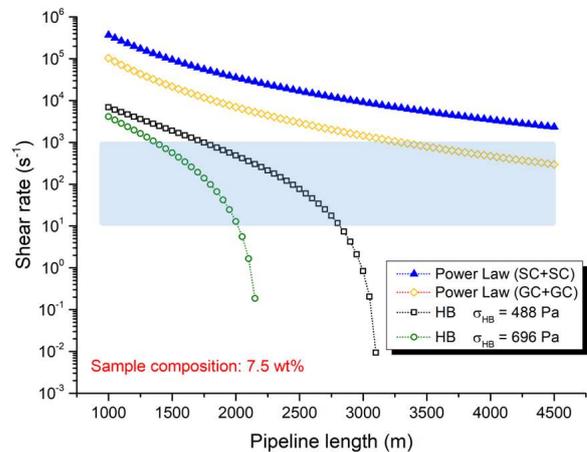


Figure 10. Shear rate at pipe wall calculated assuming Newtonian flow for Power law and Herschel-Bulkley models (sample 7.5 wt%), pipeline varying from 1,000 to 4,500 m,  $D = 8''$ ,  $\Delta P_{AVA} = 300$  bar

## Conclusions

This study demonstrated how apparent wall slip affects rheometric measurements and, therefore, the restart pressure drop ( $\Delta P_{REQ}$ ) and flow rate calculations. Model oils with 3.0 wt% and 7.5 wt% wax were employed.

Assuming a maximum available pressure ( $\Delta P_{AVA}$ ) of 300 bar, the flow at a clogged pipeline with up to 2,250 m could be restarted for 3.0 wt% model oil and only a 570 m pipeline in the case of 7.5 wt% oil. If yield stress measurements were made with smooth geometries, a pipeline length of 3,900 m could be restarted for 7.5 wt% model oil. For the 3.0 wt% system the length is 47,7 km, which is unfeasible. Also, the  $\Delta P_{REQ}$  calculations for different pipe diameters progressively diverge when smooth and grooved surfaces are used in rheometric tests. The situation worsens as the diameter decreases and wax content increases, representing a bottleneck for the correct pipeline design if surface roughness is not considered in the project.

The flow rate estimated with Power Law parameters was unable to yield reasonable results (i.e., exceedingly high flow rate values), despite the different approaches used to obtain the associated flow curves (direct or reverse mode). Although, starting the rheological experiment at a high shear rate and descending to lower values seems to be more suitable. Also, the deleterious slippage effect was seen in the simulations since an average decrease of  $10^3$  m<sup>3</sup>/day was calculated by using the parameters from grooved geometries compared to the smooth geometries.

Herschel-Bulkley model had a better performance in terms of feasible flow rates due to the parameter  $\sigma_{HB}$ , which can be interpreted as dynamic yield stress. Depending on the pipeline length, the shear rate (and, therefore, the flow rate) goes to zero, an expected physical behavior. For model oil 7.5 wt% wax, the maximum pipeline length for observable flow rate ( $> 0.1$  m<sup>3</sup>/day) is 3,100 m for  $\sigma_{HB} = 488$  Pa and 2,150 m for  $\sigma_{HB} = 696$  Pa. Also, shear rates commonly associated with pipe flow could be calculated. Further studies on the steady state conditions of the flow curves and numerical solutions for more complex models (e.g., Casson model) will be of great value for flow rate calculations of waxy oils in pipelines of varying length and diameter.

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