# Rheology and Pipeline Start up Pressure of Raguba Crude Oil

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**Abstract:** Waxy crude oil is a valuable soft that undergoes long journeys from the well to the terminals through thousand of miles of pipeline in various parts of the globe that include deserts, arctic areas and sub-sea beds. Under such conditions, pipeline cease pumping with serious consequences both in cost of interruption of production and damages to the pipeline.

This paper describes research carried out on Libyan waxy crude oil namely (Raguba) to study (1) the rheology of this crude oil under various conditions and (2) the start-up pressure of this oil when temperatures decrease below the gel point. The rheological data to be presented are extensive in that they cover a range of conditions of temperature below and above gel and pour point and cooling histories (from very low to rapid cooling, 0.1 to 6.5°C/min representative of conditions under pipeline operations). Steady and unsteady states rheological tests were carried out under controlled shear stress and shear rate using an MCR-501 Anton Paar rheometer. Experiments were also carried out to measure the extent of visco-elasticity. As for the start-up pressure measurement, the paper describes a laboratory pipeline rig developed for the purpose of conditioning the oil (setting a measured cooling history) and measuring the corresponding start-up pressures after a range of stoppage time of 1, 2, 4 and 6 hours.

The data obtained shows a good agreement between the yield stresses measured in the rheological tests and the start-up pressure tests but only when conditioning of the oil in the pipeline rig is duplicated in the rheological tests. The data also give practical guidelines on the minimum pressures required after certain time of pipeline stoppage. The data are useful in practice as they reduce excessive waste of power during pumping the oil.

**Keywords:** Rheology, Waxy crude oil, Gel point, shear rate, shearing time, cooling rate, yield stress.

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## INTRODUCTION

Waxy crude oils present challenging transportation problems especially when sudden drop in temperature arise to the oil gelling, effectively crystallising the paraffin and heavy hydrocarbons molecules. Pipelines transporting waxy crude oils may be shut down regularly for normal operational reasons and occasionally for emergency reasons, such as power failure, line damage, or even earth-quake, etc. In this case, the warm crude oil in the pipeline may be cooled statically below its pour point, leading to a strong waxy crystal interlocking network forming in the oil. Typical pipeline of 1m in diameter and 500 km long carries 1 million barrels of oil per day, such transport is expensive in terms of pumping cost ($million/year) and in any events that may lead to pumping failure and re-start add further to these costs and inevitably to the price of oil. Such event is related to the composition of the crude oil which if it contains sufficient wax which can gel or solidify in the pipe when the temperature drops below the gel point. One may think that the temperature has to drop significantly for oils to gel but this is not always the case. Libyan crude oils for example and these are the ones considered in this study, can gel at temperature 3, 5, or even 39$^0$C which are normal temperatures in winter at night times in Libya. In such conditions, pumping failures can be frequent and re-starting pumping costs become critical to the cost of operating the pipeline. Waxy crude oils are not limited to Libya and are common to many fields (North Sea and Canada are two examples) particularly new explored fields, so the waxy crude oils re-start problem is universal and of great interest to the petroleum industry. Rheological properties are important to pumping and re-start

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pressures estimation and require thus careful evaluation. This is one aspect of this paper and furthermore, using the most advanced rheological instruments to characterize these oils and establish their time-shear dependent viscous properties, yield stresses and viscoelasticity. Another important aspect of this paper is the careful measurement of the start-up pressures after gelling which we have established in our laboratory using a purpose built small-scale pipeline rig. The final and may be most important achievement of this paper is reconciling the measured start-up pressures with the predicted start-up pressures using the rheological data in a simple analytical model. Previous studies have not been able to achieve such a fit because they have not -as we shall show- considered the important need of duplicating the deformation-flow conditions in the rheological tests and in the pipeline tests. The yielding of the gelled oil, which is a fracture process of waxy crystals interlocking network, is the critical mechanism to track down and measure carefully and unless it is achieved in the rheological tests and in the pipeline test from the same *history* point, there should be no expectation that model predictions (regardless of how comprehensive the model may be) should match the measurements.

Waxy crude oils deform and eventually flow much in the same way as materials with a structure, for example polymer melts or concentrated suspensions. In other words, the yielding is not sudden (there is more than a single yield stress) but gradual, which must begin at a critical minimum stress which is best defined as the elastic lower limit yield stress, $\tau_e$. Above this lower limit, the gelled oil will deform elastically and reversibly until it reaches the upper elastic limit yield stress $\tau_{s,}$ 'static' (start of flow from rest) when irreversible creeping begins. Beyond this point, the structure disintegrates rapidly but requiring little extra stress to give way eventually at $\tau_{d\ ,}$ to flow or dynamic flow, mostly by shear in this instance. Both $\tau_{s,}$ and $\tau_{d,}$ can be labeled 'dynamic' (determined during flow) in relation to the 'static' definition of the yield stress $\tau_{s,}$ the static and dynamic yield stresses are shown in Fig. 1. This three-yield-stress description is now accepted as being the most appropriate (Chang *et al* (1999;), Bonnecaze and Brady, (1992); Benkreaira and Amhamed, (2008) superseding the *"myth"* or no yield stress debate sparked of by Barnes and Walter, (1985).

We propose in this work to perform rheological tests along the three-yield-stress model and apply the model in a simple analytical way to predict start-up pressures in a laboratory pipeline under isothermal conditions. We shall ignore in our work compressible effects which occur in practice as a result of the crude oil shrinking upon gelling when cooled and developing gas voids in the pipe.

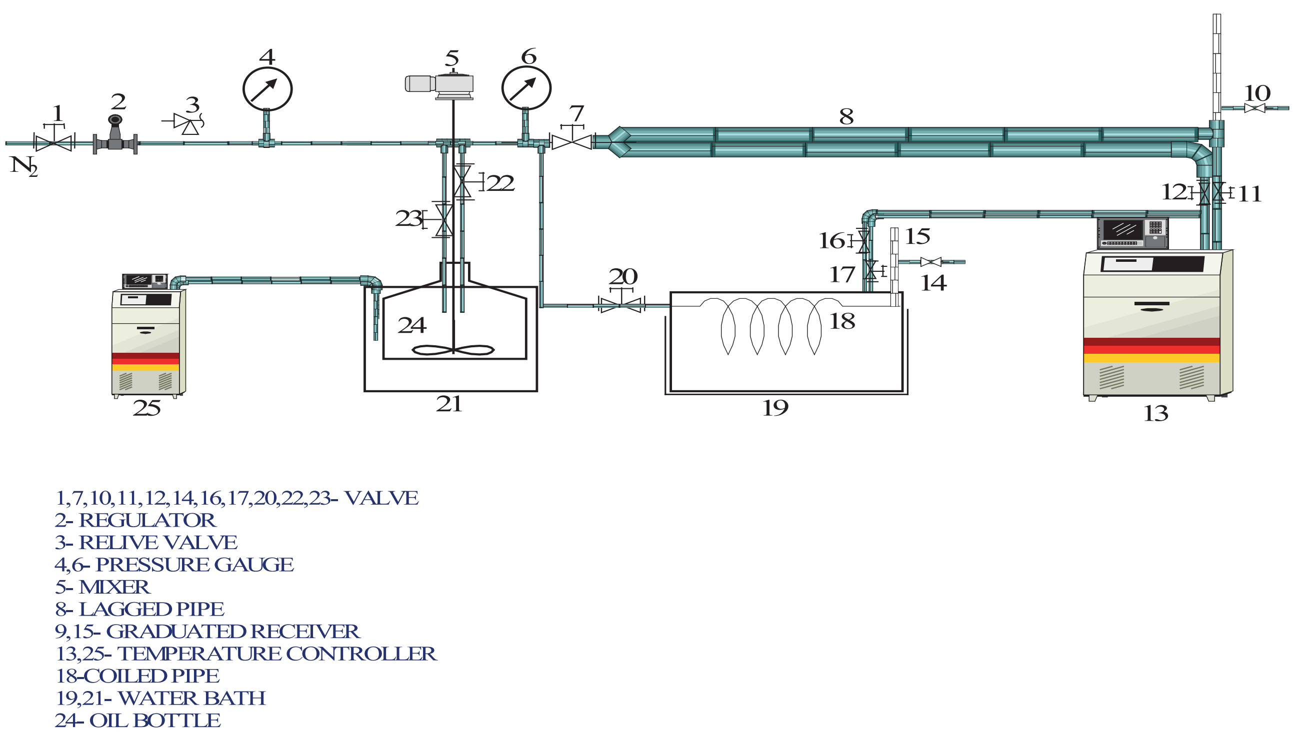


Fig. 1. Static and dynamic yield stresses of waxy crude oils (ref.17)

## EXPERIMENTAL METHOD

The sample source was the Ragouba field of Sirte oil Company and was drawn from the well-head. Its specific physical properties as quoted by the operating company are given in Table1, which include wax content and pour point. The pour point is useful indicator of gel or wax formation temperatures and will guide us in the design of our rheological and pumping experiments. The oil sample was collected near the well-head by the company personal into two 5 litre tins. These two tins were then transported to the University of Bradford in England. The oil in the five litre tins was transferred and mixed into a ten-litre container. Samples, which were used in experimental work, were taken from the 10 litres tin by preheating in hot water. The experiments started with establishing the rheological properties of the oils using advanced rheometers capable of detecting yield values then measuring start-up pressures in a suitably designed pipeline rig.

*Table 1.* Physical characteristics of Ragouba Waxy Crude.

| Test | Method | value |
|---|---|---|
| API gravity@60°F | Calculated | 41.86 |
| Wax content (wt%) | UOP46/64 | 6 |
| Pour point, °C | IP 15/D97 | 7 |
| Asphalten content (wt%) | IP 143/84 | 0.1 |
| Total Sulphur (wt%) | IP 61/84 | 0.03 |

***Rheology of waxy crude oils***: The rheological tests were performed using Anton Paar MCR301 and MCR 501 operated at controlled shear stress or shear rates. The rheometers are provided with an air bearing with digitally controlled shear stress CSS ($10^{-7}$ Pa to 3000 Pa) and control shear rate CSR ($10^{-6}$ $min^{-1}$ to 3000 $min^{-1}$) tests. The shear tests were performed using a cone and plate geometry (diameter 5cm and cone angle 1°) and the dynamic storage $G'$ and the loss $G''$ moduli were measured in a plate-plate geometry with an appropriately roughened upper plate to avoid slip during the test. A peltier device accurately controlled the temperature to within +/- 0.1 $^0$C. Prior to the measurement of the rheological properties, a strict protocol for the preparation of the sample was followed to ensure a tractable history and one that could be reproduced in the pipeline rig.

***Sample preparation***: The oil was retrieved from the two 5 litres original containers and transferred to a single stirred vessel fitted with a hot water jacket. The oil was heated to 48$^0$C and stirred for 20 minutes. Samples were poured into a half-litre stirred glass container then heated to 80$^0$C for two hours while stirring. After this treatment, the oil was left to cool quiescently at room temperature for at least 48 hours before testing. Such a procedure, also used by other researchers [7-10], removes the fluid memory and this was confirmed by a series of repeat experiments in the rheometers. In all the subsequent text we shall refer to this oil as the no-memory base stock or sample.

***Determination of gel point***: In these experiments, the oscillatory test is used to examine the gelation temperature. The amplitude sweep test has to be done to determine the linear viscoelastic range (LVE) of the sample. In the liner viscoelastic (LVE) range at low amplitudes, each of the G' and G" shows a constant plateau value. The strain was kept at 1%, frequency 1 Hz and the gap was set at 0.5mm. The initial temperate in all these tests was fixed at 35°C; final test temperature was 5°C .The tests were performed at different cooling rates of 0.5, 1, 3, 5 and 6.5°C/min, full details of test can be seen in Table2. The storage $G'$ and the loss $G''$ module are also recorded.

*Table 2.* Oscillatory test conditions for Ragouba waxy oil

| Sample | Initial Temp., ºC | Final Temp.,ºC | Cooling rate °C/min | Frequency Hz | Strain % |
|---|---|---|---|---|---|
| Ragouba Oil | 35<br>35<br>35<br>35<br>35 | 5<br>5<br>5<br>5<br>5 | 6.5<br>5.0<br>3.0<br>1<br>0.5 | 1 | 1 |

***Effect of static cooling rate on rheological properties***: For these experiments, 70-100 ml samples were loaded onto the rheometer plate, pre-set at the rheological temperature desired. The sample was left to rest for 15 minutes on the plate to form its equilibrium structure.The temperature is pre-set above the gel point.The sample is cooled statically from 30 to 5°C using different cooling rates. When the sample reaches 5°C, the rheological properties are measured at dynamic conditions over a range of shear rates from 1 to 1000 $s^{-1}$. This procedure (Table3) was repeated at 15 and 25 °C using a fresh sample for each run.

***Pipeline Rig measurements***: As for the start-up pressure test, a new laboratory pipeline rig developed for purpose of conditioning the oil and measuring the corresponding start-up pressure after a range of stoppage time of 1 to 2, 4, and 6 hours. In this measurement the oil cooled dynamically and statically in two stages in order to mimic the actual conditions in the field. The essential pressure to restart flow in a full-scale pipeline blocked with gilled waxy crude oil is calculated by a force balance equation:

$$\Delta P = 4L\tau y/D \qquad (1)$$

This formal comes from the simplified idea that the force pushing the gel $\Delta P\frac{\pi D^2}{4}$ must be equal to the shear resistance along the wall $\pi D\ \tau y$ [20].

The main assumption is that the value of yield stress $\tau y$ is constant along the length of the pipeline. Where $D$ is the internal diameter of the pipeline, $\Delta P$ is the applied pressure differential and $L$ is the length of the pipeline. Fig. 2 shows the lab model pipeline assembly used in the present research. The experimental rig which consists of a straight jacketed pipeline, 9 m long and 6.5 mm inside diameter copper tube or 17 m long coiled pipeline of 6.5 mm inside diameter copper tube having 21 circular rings immersed in a tank. In both cases nitrogen gas is used to pump the oil. The pressure drop between two ends of the pipeline can be measured by means of a pressure gauge/manometer. The first water bath will be used for cooling the sample whilst the oil agitated by Rushton turbine impeller at a controlled temperature. The dynamic cooling is carried out in a 2-litre aluminium pot. The second stage cooling is under static conditions (i.e. whilst the oil stationary in the pipe) in either the coil pipeline or straight pipeline.

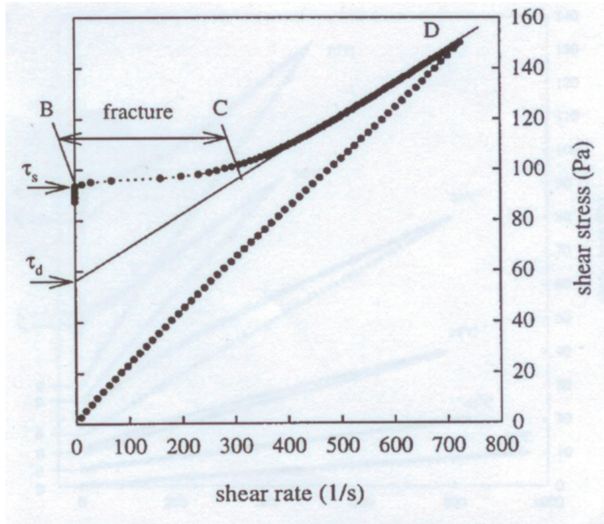


Fig. 2. The start-up pressure pipeline rig. Items 25 and 13 are temperature controller. , Nitrogen gas is used to pump, 8 is a straight pipeline (9m x 6.5mm ID) and 18 an alternative coiled pipeline (17m x 6.5mm ID).

## EXPERIMENTAL PROCEDURE

(i) About 0.65 - 1 litre of crude oil is poured into the pot, which is connected to the 6.5 mm pipeline; the oil was kept for 30 min. under stirring (80 rpm).

(ii) Then the oil was dynamically cooled with preset rate of cooling to a final temperature, and left for 30 min. at this temperature.

(iii) The oil was introduced into the pipeline by use of Nitrogen gas from the bottle flask, and cooled statically to the test temperature under a specified rate of cooling. At this temperature, the oil is held for a certain period of time of 1, 2, 4 and 6 hours to simulate static condition.

*Table 3.* Parameters of cooling test for Raguba waxy crude oil.

| Sample | Overhead temperature (ºC) | Test temperature (ºC) | Cooling rate, ºC/min. |
|---|---|---|---|
| Raguba Waxy Crude Oil | 30<br>30<br>30 | 5<br>15<br>25 | 6.5, 1, 0.5, 0.1<br>“<br>“ |

(iv) Finally, by applying Nitrogen gas pressure, shear force was increased in steps of 0.25 psi until continues movement of oil occurs, i.e. when 4-8 ml of oil flows out of the model pipeline within 10-20 minutes. The shear stress applied at this stage is equivalent the value of yield stress. Data for this test is shown in Table 4.

*Table 4.* Heating and cooling conditions for Raguba crude oil.

| Raguba waxy Crude Oil | Conditions |
|---|---|
| Dynamic cooling<br>Initial Temperature, °C<br>Final Temperature, °C<br>Rate of cooling °C/min. | 50<br>40<br>0.16,0.6,1 |
| Static cooling<br>Initial temperature, ° C<br>Final temperature, °C<br>Rate of cooling °C/min<br>Hold up time , h | 40<br>3.5, 5.5, 10<br>0.16,0.6,1<br>1, 2, 4, 6 |

## RESULT AND DISCUSSION

***Gel point***: The experimental results obtained of Ragouba crude oil are shown in Fig. 3(a), (b), (c), (d) and (e) where the storage *G'* and the loss *G"* moduli are plotted versus temperature during cooling. For this crude oil, the crossing of moduli occurs at 26 to 16.1°C according to the used cooling rate. This may be called the gel temperature ($T_g$). At a temperature below the gel point, the value of *G'* is higher than the value of *G"*. This indicates the formation of a rigid network even above the pour point. Fig. 3(e) shows the effect of cooling rate on the gel point. For this sample, it can be seen that the gel point decreases as the cooling rate increased for the temperature range studied. It has to be said that the pour point may not be a precies indication of gel point which is strongly dependent on cooling rate. This is expected as crystallisation is crucially controlled by cooling rate.

A very slow cooling rate will allow crystals to form and grow whilst a fast cooling rate will prevent this occurring. Clearly relying on the pour point to inform operation and design of pumping waxy crude oil is not recommended as we observe gel points to be larger than pour points. There is however an important difference to make. Gelation temperature only means that wax is beginning to form, it does not mean that all the wax has solidified. In other words whereas pour point is related to "flow" as measured crudely, it is still an indication of flow. While Gel point is not and flow in the pipeline may not be hampered until well below the gel point when all the wax has solidefied.

***Effect of cooling rate on rheological properties during static conditions***: In general, the cooling rate is expected to influence the rheology by the possibility of modifying the wax crystal size distribution. Moreover, it is well known that relatively low temperatures adversely affect the rheological properties of waxy crudes. This is because it modifies the oils rheological behaviour to a non-Newtonian character at a certain temperature. Under static cooling, an interlocking crystal network is built up in the waxy crude causing adverse flow properties. This is a real problem in pipeline when the oil cools down quiescently on a prolonged shutdown. Therefore, we tried to apply the same field conditions, which may help to understand the rheological flow properties at static conditions. Fig. 4 shows the effect of static cooling rate on the flow properties of Raguba waxy oil

A low cooling rate could help the growth of large crystals, which could give rise to the pumping problems Venkatesan *et al* (2002) & Chang *et al* (1999). Very rapid cooling could form a rigid interlocking crystal structure, causing a high yield pressure and resistance to flow. In case of Raguba waxy crude oil, by plotting the resultant equilibrium flow curves, the low cooling rate gives to approximately same equilibrium flow curves of viscosities at same temperature, telling that the low cooling rate has no influence on the flow properties of Ragouba crude oil. As the rapid cooling rate, e.g. 6.5ºC/min has a clear influence on the viscosity of this oil. It has to be said that there is an increase in viscosity due to the build up of a rigid interlocking crystal structure. In addition, viscosity reduction is due to structural breakdown in the shear field, which starts at about 10 $s^{-1}$.

### Yield stress measurements

***Measurements by Rheometers***: The reproducibility of yield stress measurements has been found to be poor due to many reasons, e.g. the confused definition of yield stress, limitation of the instruments, methods, the procedure of the test and the poor control of the thermal conditions of the test (Bonnecaze *et al* (1992); Venkatesan *et al* 2002). Nowadays, due to

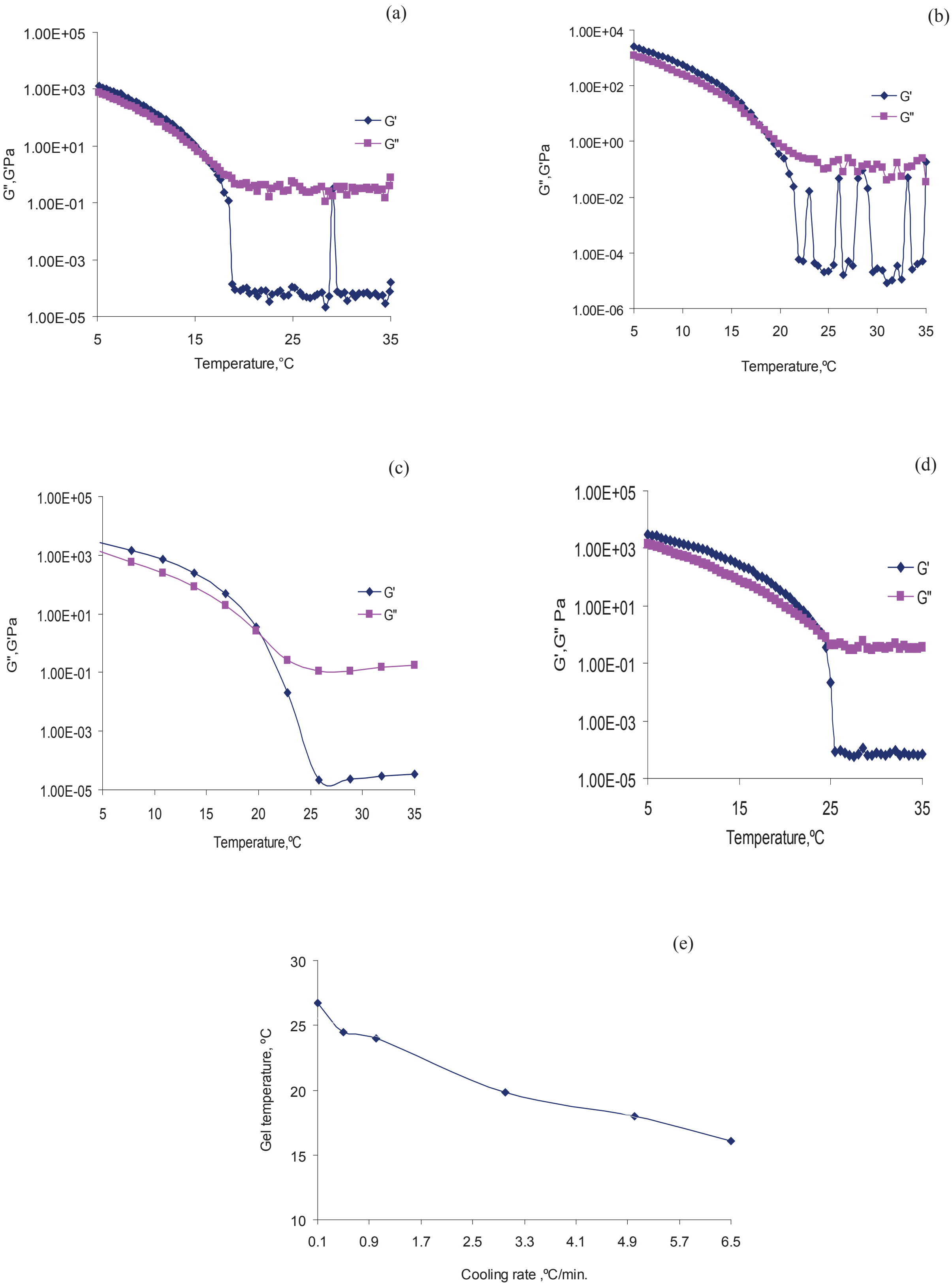


Fig. 3. Gel point temperature of Raguba oil at various cooling rate, (a) 0.1º/min.,(b) 0.5º/min, (c) 5º/min., (d) 6.5º/min. and (e) deviation of gel point against the cooling rate.

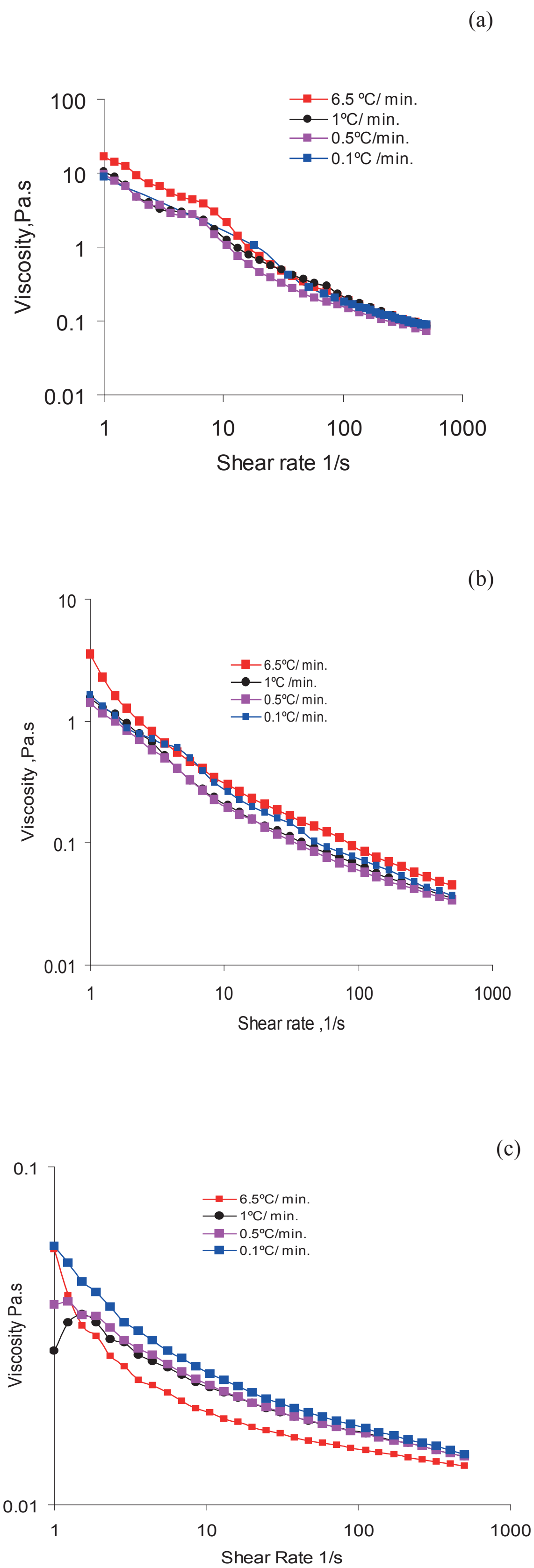


Fig. 4. Variation of Viscosity with Cooling Rate for Raguba Oil. (a)T= 30→5ºC ,(b) T=30→15ºC and (c) T=30→25ºC.
note: Viscosity measured at 5, 15 and 25ºC, respectively.

the availability of controlled stress rheometers with cone and plate geometries, the true yield stress of waxy crude oil using direct method can be easily determined. In these measurements, a fundamental problem with concept of yield stress fluids is the complexity in determining the yield stress value. There is no single method that has been commonly established as the standard for measuring the yield stress. It is to be stated that it is not unusual to get a different values of yield stress for the same material, prepared and tested in the same laboratory even by using same Rheometer, and obtained by using different methods (Moller *et al* 2006; Barnes 1999; Nguyen and Boger, 1992; Naguyen *et al* 2006). The apparent yield point, which is determined as the limit of elastic range, is not material constant and depends on time, pre-treatment and the test conditions (Mezger, 2002). In this research work, direct method is used to determine the yield stress of this sample according to a prescribed procedure. So the yield stress data covered are:

1. Time dependency yield stress.
2. Instantaneous yield stress.
3. Yield stress beginning from an equivalent state in the pipeline.

The yield stress is the value that shows a critical massive change in viscosity. Fig. 4 shows this clearly for the Ragouba oil tested where time dependency of yield stress or its instantaneous threshold is monitored. Conceptually, this method of identifying the yield is feasible and from an engineering perspective reasonable but nevertheless a value must be determined and there lies the problem. Which value in the changes of viscosity is the most representative of a true yield stress? Mathematically, it could "pin" the yield stress as the value in the viscosity curve which shows an inflection point or the first point at which the viscosity begins to drop. It does observe in the data however, that the viscosity goes through a reduced region before plummeting down completely to a very low value. We can thus distinguish between:

1. A lower yield stress or the beginning of shear flow
2. An upper yield stress or the value where shear flow is fully "on", i.e. beyond this point the structure has fully disintegrated and the "solid" part of the oil has disappeared.

Thus the lower yield stress is the most appropriate as it is that indicates the start of shear flow.

Having defined all the terms and their meaning we can note the following observations on the results. Yielding is strongly time dependent. For example, at $5^0$C and 15 minutes, Ragouba oil will yield at 20, 30, 40 and 50 Pa over a range of time. For the same temperature and 2 hrs as shutdown time sample will yield at higher value of yield stress starting from 30, 40, 50 and 100 Pa as can be seen in Fig. 4. The main conclusion is that we have already defined a series of yield stress not just one!

Yielding in the critical region, i.e. in the narrow region where the viscosity begins to drop considerably occurs in a range of time, i.e. in a range of stresses.

On an instantaneous basis, i.e. when we remove time dependency, yielding must occur over a range of stresses and there will be, as we indicated above, a lower yield stress and an upper yield stress. Because shear flow will begin at the upper value, this value is the "true" yield stress at least from an engineering perspective. A corollary to the above observations is that below the lower limit, the crude oil behaves as an elastic solid. In other words, if we remove the stress it should revert back to its original state. i.e. it is now shear- flowing.

The data obtained from rheological measurements on Ragouba waxy crude oil, which fully conditioned and measured by an Anton Paar rheometer are shown in Fig. 5, these measurements were performed at 5 ºC with 15 min. and 2 hrs as shutdown time. It can be seen from the data that after yielding point, the viscosity dropped by six to five orders of magnitude, which indicates that the viscosity of oil could be reduced to low value and oil could be pumped at certain conditions even below the pour point.

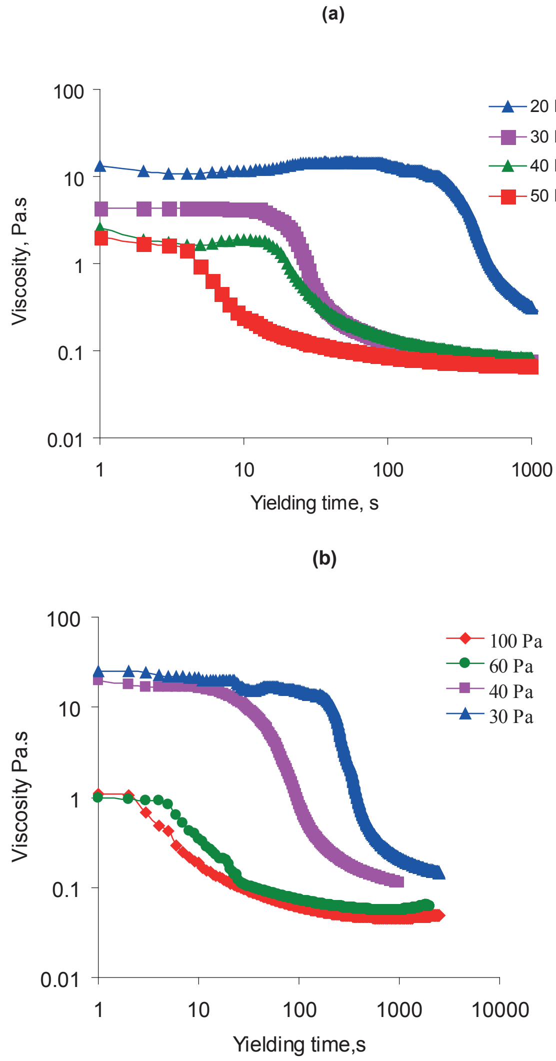


Fig. 5. Yielding time measurements for Raguba oil.
(a) T = 5ºC, 15min. shut downtime;
(b) (b) T = 5ºC, 2hrs shut downtime.

***Yield stress measurements from pipeline rig***: The aim is to use the pipeline rig in order to measure start-up pressure and use the simple force balance model Equation 1 to calculate the yield stress and compare this stress with the stress measured using the rheometer. This is effectively our experimental work underpinning of the theoretical work it assumes here the rheological measurements are in fact the theoretical information or more precisely the fundamental information.

The experimental equipment and procedure in terms of measurements are described above, but the important details here are the conditions under which the stresses (start-up pressures) are measured. We aim all along to replicate the conditions covered in the rheometer measurement, i.e. start temperature, end temperature, cooling rate, hold up–time but very importantly to start from a base line where the memory of the sample being pumped is erased before it was placed in the pipeline and proceeding with mimicking the conditions that would occur in a typical shut –down. The results of these experiments are presented in Figs. 6 to 9. Here it is interested to compare the stresses measured in the pipeline with rheological results obtained from Rheometers.

The ultimate aim is to demonstrate that rheological tests carried out carefully are able to replicate the performance in the pipeline. This is indeed the main aim of our research and many previous researches in this field. The lower yield stress and upper yield stress are discussed earlier and could only distinguish between the two because of the accuracy in stepping up in stresses at very small steps in our rheometer. Also, the cooling equipment connected to the rheometer is very effective so a range of scenarios could be applied. As far as the pipeline rig, the creative feature is the way we erase the memory and condition the sample in a purpose made holder-mixer under controlled temperature conditions. It is the key to the experimental protocol.

The results presented in Figs. 6-9 reveal the following points:

1. The yield stress (Start up Pressure) was significantly reduced by increasing the test temperature.
2. The change of yield stress will be insignificant when the change of temperature occurs at or above the pour point temperature. In this region the oil behaves slightly Newtonian due to presence of a small amount of wax crystals.
3. The data obtained shows that yield stress decreases with an increase in test temperature.

The yield stress for the first hour was 4 Pa and increases to 7.77 Pa after two hours of shut downtime. Data obtained shows that the yield stress even after 4 or 6 hours of shut downtime remains constant at about 8 Pa. This data enables us to say that there is no extra power required to restart up the Raguba pipeline after six hours of shutdown at a low cooling rate and a 2.5ºC below the pour point.

The data also show that the high cooling rate gives higher values of yield stresses. This is due to the different morphology developed by the paraffin crystals during different rates of rapid cooling. Moreover, the increased yield stresses for this oil indicate that they contain $C_{36}$ and $C_{32}$ components. This component leads to an increase of yield stress with increasing cooling rate (Benkreaira *et al* 2008).

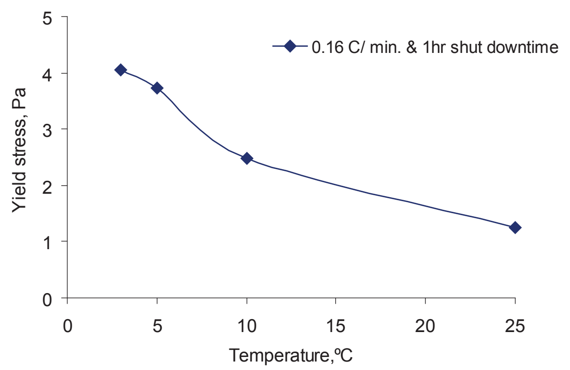


Fig. 6. Effect of test temperature on yield stress values for Raguba oil.

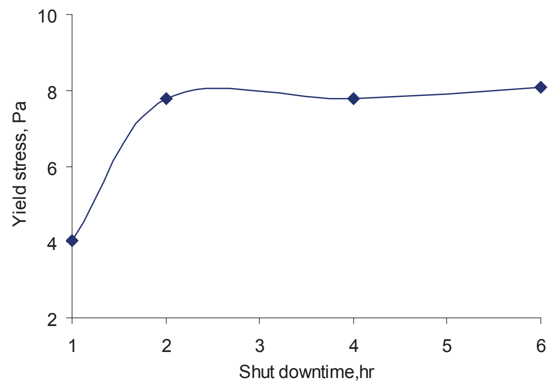


Fig. 7. Effect of shut downtime on yield stresses values of Raguba waxy oil at 0.16 ºC/min. and 3.5ºC.

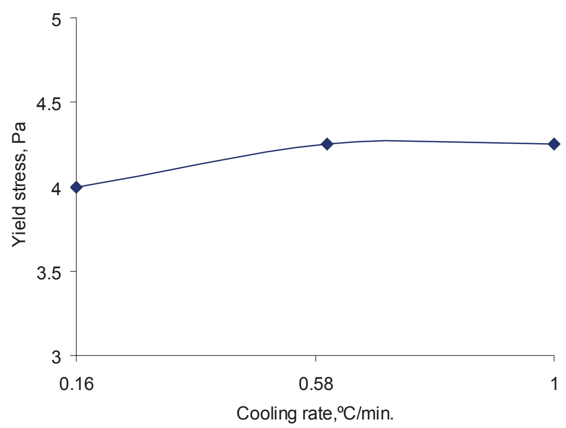


Fig. 8. Effect of cooling rate on yield stresses values of Raguba waxy oil. (3.5 ºC, 1 hr shutdown time).

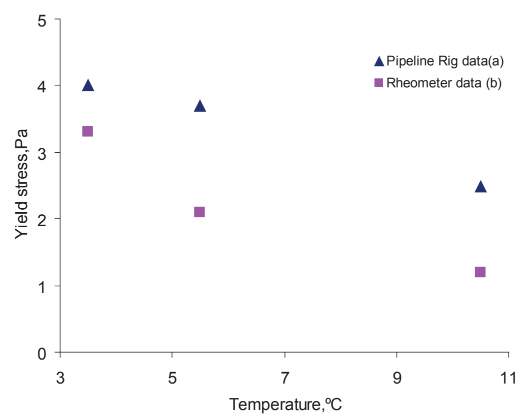


Fig. 9. yield stresses measurements for Raguba oil in Pipeline rig and MCR 501 Rheometer. 3.5 ºC and 1hr shut downtime.

## CONCLUSIONS

It is to conclude that Ragouba waxy crude oil is a shear-sensitive and slightly time-dependent non-Newtonian fluid below the gel point temperature. It behaves as a strong non-Newtonian at and below the pour point temperature. The cooling temperature adversely affects the rheological properties of waxy crudes in such a manner that it modifies their rheological behaviour to a non- Newtonian character on approaching a certain temperature. The effect of cooling rate was investigated, the data obtained shows that the low cooling rate gives almost identical equilibrium flow curves of viscosities, suggesting that low cooling rate has no influence on the flow properties of Raguba oil, whilst the rapid cooling, 6.5ºC/min has a clearly influenced the viscosity especially at low shear rate. The effect of rapid cooling rate is strongly related to low shear rate, because at high shear rate the effect is negligible.

In the second class of experiments, which is done using pipeline rig, it is found that the value of yield stress decreased with increased test temperatures, and increases with increased rate of cooling and hold up time. Moreover, a good agreement found between yield stresses measured using the rheometer with those measured in the pipeline rig via the measurements of start-up pressures at certain conditions. From data comparsion, it is clear there is a good agreement. This gives confidence in the prediction using the rheometer which is cheaper option than using a pipeline rig data for restarting pressure estimation. The data obtained also give practical guidelines on the minimum pressures required after certain time of pipeline stoppage. The data are helpful in practice as they reduce excessive waste of power during pumping processes

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