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Yield Stress Measurements of Waxy Crude Oil

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Abstract: Predicting the restart pressure for gelled waxy crude has always been a challenge due to the thixotropic nature of the crude apart from the thermal shrinkage and presence of gas voids within the gelled crude. Yield stress is the stress corresponding to the transition from elastic to plastic deformation, the value of shear stress above which the material flows. Existing method defines two yield stresses, one static and one dynamic. While static yield stress is more difficult to measure since most occur at very low stresses and that the measuring methods is still subjected to many debates whereas the dynamic yield stress is relatively easier to be obtained and requires linear extrapolation of viscosity data at high shear rates. There are many methods can be used to measure the yield stress which are the creep recovery test, stress ramp test, oscillatory test, vane technique and many more. In this study, the comparison will be done using all the mentioned methods and how a new method has been developed to accurately measure the yield stress for the waxy crude oil. The new proposed measurement for static yield stress was done with oscillatory testing and dynamic yield stress with steady state peak hold testing. All measurement was done under the rheometer with minimized wall slip effect for both statically and dynamically cooled sample. For the yield stress measurement, it also exhibited some interesting features of the waxy crude oil. The outcome of the study could then be used to fully understand the nature of the gelled crude.

Key words: Waxy crude oil, static yield stress, dynamic yield stress, rheometer, wall slip effect

INTRODUCTION

One of the primary interests in dealing with the transportation of the waxy crude is with the issue of restarting the flow. During temporary shutdown of production operation at platform which is usually performed for maintenance or emergency reasons, the crude is static and under such condition, the temperature within the pipeline is subjected to the external factors and drops significantly as in the case for subsea installations and in the arctic regions. The temperature decrease beyond the WAT will result in formation of paraffin crystals. The formation of the paraffin crystals or wax is known to be mainly dependent on the temperature of the crude (Roberts and Barnes, 2001). Above the WAT, crude oil behaves mainly like a Newtonian liquid and below which non-Newtonian behaviour is increasingly significant, where elastic behaviour is also observed, as the amount of wax crystals increases (Vinay *et al*., 2005). As the temperature drops further it will be transformed into gel-like structure of which the yield stress and thixotropic behaviour are further exaggerated.

In order to restart the flow of the crude oil, the gel has to be broken down. The breakage of the gel can be done by applying large pressure, usually via a liquid at the inlet of the pipe, until the gel breakdown occurs. In order to determine the breakdown pressure required to restart the flow in a safe manner, it is important to estimate the gel strength. It is measured in terms of the yield stress of the gel. Breakdown of the wax-oil gel occurs if the shear stress exerted on the gel due to the applied pressure exceeds the yield strength of the gel (Venkatesan *et al*., 2005).

A yield stress is the stress corresponding to the transition from elastic to plastic deformation (Barnes, 1995). Chang *et al*. (1998) have conducted a stress ramp experiment for a waxy crude oil known as DH19 at a low temperature. The test was performed using a controlled stress rheometer and utilizing a cone and plate geometry. Figure 1 shows the curve recorded for a waxy crude oil, DH19. It illustrates the differences between static and dynamic yield stresses.

From Fig. 1, it is shown that fracture of the gel structure starts at point B, at which there is a significant increase in the strain showing the breakdown of the oil. Yielding nature of a waxy crude oil has three distinct characteristics; an elastic response, a creep response and a fracture. The shear stress at the

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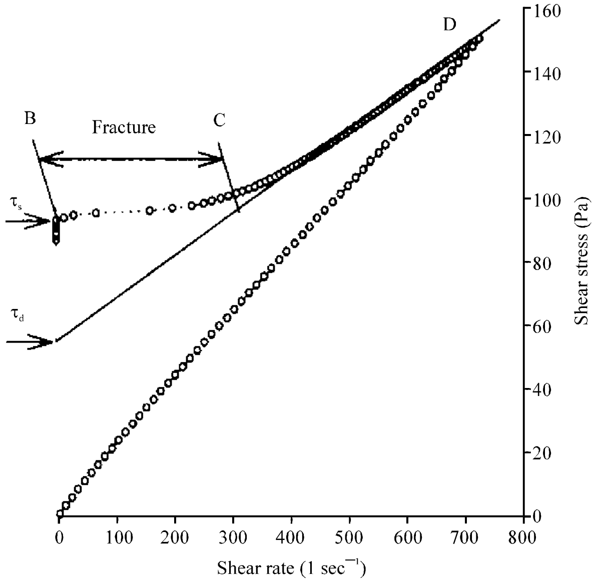

Fig. 1: Differences between static and dynamic yield stress for waxy crude oil (Controlled stress test. Stress sweep was performed from 0 to 150 Pa) (Chang *et al*., 1998)

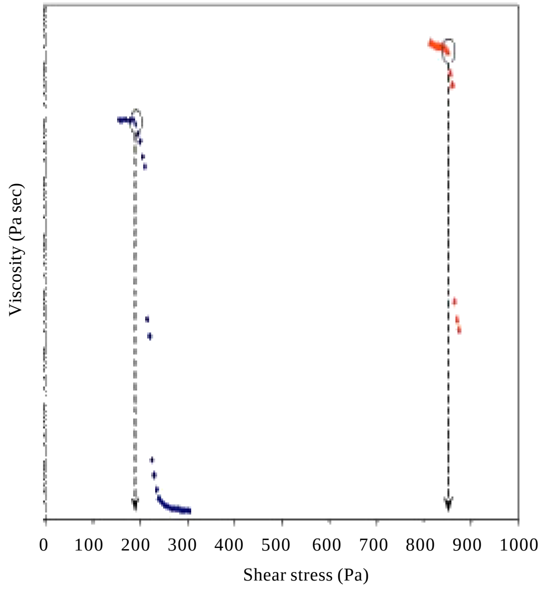

Fig. 2: Transient plot of typical rheological experiments (Venkatesan *et al*., 2005)

point of fracture is the most important value and normally taken as the yield stress (Chang *et al*., 1998). Venkatesan *et al*. (2005) conducted tests to determine the yield stress using a controlled stress rheometer with cone and plate geometry. The waxy crude oil used was Coray-15, a lubricating mineral oil, which uses a product of Exxon. The WAT of the 5% wax in oil mixture was 28.3°C. Figure 2 shows the results of the rheometric responses when determining the yield stress.

In Fig. 2, when the shear stress reaches close to the yield point, a creep response is observed and at about 190 Pa for one sample and 850 Pa for another, the point of fracture is reached as there is sudden decrease in the viscosity. Thus, the yield stress is considered as 190 and 850 Pa, respectively in the two cases (Venkatesan *et al*., 2005). One potential problem while testing for the yield stress using a rheometer is the possibility of slip at the surface. The wall slip can be observed in terms of lower viscosity and yield stress measured using a rheometer if smooth geometrics were to be utilized in the measurements (Roberts and Barnes, 2001).

This study focuses two yield stresses as one static and one dynamic. The static yield stress (τ_s) is the shear stress needed for the unbounded strain or deformation of the material and the dynamic yield stress (τ_d) is extrapolated shear stress at zero shear rate obtained from the flow curve. The dynamic yield stress is essentially as an imagined parameter which can be used in defining the oil properties at the final sheared state and therefore it is not a material property relating to the yielding process (Lee, 2008; Chang *et al*., 1998). The stresses which are related to the yielding process is the elastic limit yield stress, which is the stress at the starting of viscoelastic creep and static yield stress, which is the stress at the starting of fracture. The elastic limit and static yield stresses are dependent on the strength of the interlocking system of wax crystals in the oils before the structure is destroyed. The dynamic yield stress is dependent to the concentration and size of the wax particles in the oils after the structure is completely broken. To effectively determine the pump capacity required initiating flow and ensuring pipelines restart, engineers are keen to use the static yield stress, the stress value when the breakage occurs (Chang *et al*., 1998).

Chang *et al*. (1999) has mentioned in one of his research work that the complete yielding process can be clearly shown by conducting an oscillatory test with a gradual increase of controlled stress at a fixed low frequency. Figure 3 shows a typical curve recorded for a waxy crude oil in which the shear strain is rather measured in the test than shear rate.

As can be seen in Fig. 3, before point A which is the initial linear regime represents an elastic behaviour where the strain increases linearly with shear stress. Creep occurs after 6 point A with the stress-strain relationship slowly deviating from linear. Fracture starts at point B, showing the breakage of the oil microstructure where

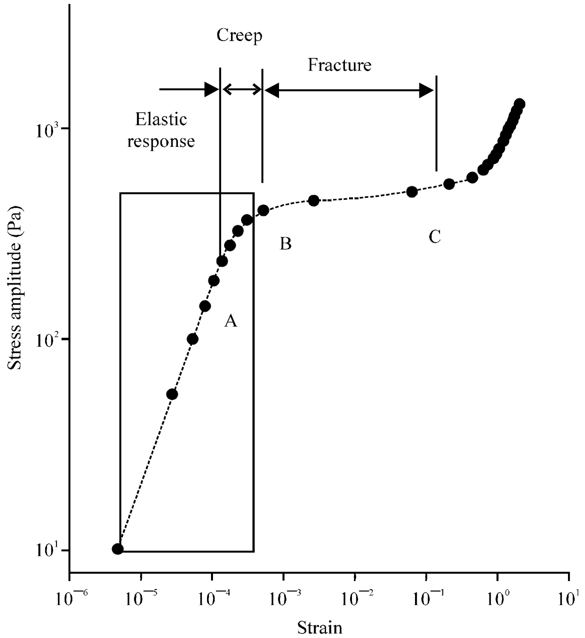

Fig. 3: Yielding process of waxy crude oil (DH19) (Chang *et al.*, 1999)

there is a significant increase of the strain. The entire process can be differentiated with two yield stresses which are the elastic limit yield stress, the stress transition between elastic and creep and the static yield stress, the stress at the start of the fracture.For dynamic yield stress is defines as an extrapolated shear stress at zero shear rate obtained from the flow curve (Chang *et al.*, 1999).

In the literature by Russell and Chapman and, Chang and Ronningsen, they have studied the effect of cooling rate on the strength of the waxy crude oil gels formed under static cooling. However the results are not in agreement. Russell and Chapman observed that larger cooling rate produced stronger gels where else Ronningsen and Chang observed that smaller cooling rates produced stronger gels. The possible reason for the decrease in yield stress with an increasing cooling rate can be explained by considering the time taken for the growth of the wax crystal. Lower cooling rates give more time for crystal growth which will result in larger formation of crystals. For higher cooling rate, the rate of precipitation is faster and less time for the crystal growth which will result in smaller formation of crystals.

Waxy crude oil under dynamic cooling, the behaviour is opposite to the behaviour of the gels under static cooling. The yield stress increased with an increasing cooling rate. The shear stress tends to break the crystal network even as if forms. When the cooling rate is low, the waxy crude oil is subjected to the shear stress for a longer time before the sample gels up and therefore the gel build up is weaker. At higher cooling rate, the gelation happens faster and the waxy crude oil is subjected to shear stress for a shorter time which results in stronger gels. The effect of gelation shear on the formation of wax crystals was studied using various shear rates using a Linkam flow cell coupled with amicroscope. When the gelation shear stress increases, the breakdown increases (Venkatesan *et al.*, 2005).

Nevertheless, there is no standard test for defining the yielding quantity of waxy crude oils that has been be implemented by the petroleum industry as the poor repeatability in any given instrument and poor reproducibility between different experimental setup. The possible reason for this is that the yield values with other rheological properties of the waxy crude oils strongly depend not only on what the sample is going through as such the temperature and shear rate but also on what the sample had went through such as thermal and shear history.

Other important reason for this is that there are many confusing definitions of the yield stress because of the lack of understanding of the complicated yielding process (Venkatesan *et al.*, 2005). Wardhaugh and Boger (1991) in one of their literature has mentioned that the possible sources of the poor repeatability can be the usual instrumental effects such as wall slip, instrument inertia, damping characteristics of the rotation body and sensitivity to the external disturbances.

There are several geometries can be used to measure the yield stress which are the cone and plate, parallel plate and vane geometry. With using these geometries, the yield stress can be obtained with creep-recovery test, oscillatory test, stress ramp test and many more.

Cone and plate geometry can be considered as one of the most well-known geometry used to determine the viscosity of viscoelastic fluid. To achieve constant shear rate in the gap, a very small sample is placed in the space between a plate and a cone of the same radius with a very small angle. At the outer edge, the sample should have s free spherical shaped surface. With using this geometry very small amount of sample is needed and the systems has very good heat transfer as well as temperature control. Even though this geometry may seemed to be perfect for rheological studies, but it has some disadvantages. The system is restricted to low shear rates and for highly elastic systems, it will not stay intact in the gap at high rotational speed. There are difficulties in eliminating evaporation and high inaccuracy results which are possible for multiphase systems in which the particles are the same order of magnitude as the gap size (Carreau *et al.*, 1997).

Parallel plate consists of two parallel discs of certain radius with a fixed gap. One of plates will rotate and the other one remains stationary. The torque and normal force can be measured at one of the plates. The advantage of this geometry is for high temperature measurements especially for multiphase system studies. The gap size is changeable to accommodate a variety of particle size (Carreau *et al*., 1997). Both the geometries, cone and plate as well as parallel plate needs small sample volume, easy to clean, have low inertia and able to achieve high shear rate. However, using cone and plate geometry, an additional advantage is that the shear rate is uniform throughout the sample where else parallel plate can accommodate large particles.

Another geometry which can be used for yield stress measuring is vane geometry which consists of a small number of blades designed at equal angles around a small cylindrical shaft. This geometry is able to eliminate of wall slip and keep any disturbances caused by the vane into the sample to a minimum. This geometry is used by gently lowering the vane spindle into the sample in a container till the vane is fully immersed. The vane is then rotated very slowly at a constant rotational speed and the torque needed to maintain the vane's constant motion is measured as a function of time (Dzuy and Boger, 1983).

Objective: The objectives of the research work proposed are:

- To explore the different yield stress measurements for the gelled crude formed and choose the best method to measure the yield stress under the static and dynamic cooling scenarios by varying cooling rates and shear rates

A new experimental method for rheological measurements in determining the yield strength of waxy crude oil will be proposed by taking into account the wall slip effect.

MATERIALS AND METHODS

The measurements were conducted using AR G2 controlled stress rheometer by TA Instrument to measure the yield stress of waxy crude oil. The wax content is 18 wt% and Wax Appearance Temperature (WAT) is 38°C. The rheological properties of waxy crude are influenced by many factors. To ensure the repeatability of the data, the experiment must be conducted in controlled condition. A solvent trap was used to avoid the possible evaporation of the waxy crude oil during the measurement.

Various measurements of yield stress were performed such as the creep test and stress ramp test using the parallel plate geometry and also vane technique was used for both creep test and stress ramp test. Following are the procedures:

Creep test for dynamic cooling: The geometry 4 cm roughened plate was used to overcome the wall slip effect and the gap was set to 800 μm. Upon placement of sample onto the peltier plate of the rheometer, the sample was heated at 45°C and sheared at 10 $\sec^{-1}$ for 3 min. It was then left to rest for 2 min. A temperature ramp step with a constant shear rate of 10 $\sec^{-1}$ was then applied with temperature reduced from 45 to 20°C. This was done at a cooling rate of 1°C $\min^{-1}$. Creep-recovery test was performed at shear stress 20 Pa for 2 h with temperature 20°C. The shear stress on the sample was reduced to zero for another 2 h. The experiment is repeated with shear stress 30, 40 and 80 Pa to investigate the effect of shear stress on the creep behaviour.

Stress ramp for dynamic cooling: This experiment was conducted for dynamic cooling so the 4 cm roughened plate was used to overcome the wall slip effect. The gap was set at 800 μm. The sample was heated at 45°C and sheared at 10 $\sec^{-1}$ for 3 min upon placement of sample onto the peltier plate of the rheometer. It was then left to rest for 2 min. A temperature ramp step with a constant shear rate of 10 $\sec^{-1}$ was then applied with temperature reduced from 45 to 20°C and cooling rate of 1°C $\min^{-1}$. The sample was then maintained isothermally at 20°C and a shear stress ramp was applied from 50 to 250 Pa for 20 min to achieve the rate of 5 Pa $\min^{-1}$ of the gelled sample. The yield stress is defined as the shear stress value at which there is a rapid decrease in the viscosity of the sample.

Vane technique for creep and stress ramp test : The same procedures were taken for the creep test and stress ramp test using the vane technique for dynamically cooled sample. Instead, the standard vaned rotor with stator inner radius of 15 mm, rotor outer radius of 14 mm and cylinder immersed height of 42 mm was used.

The best method to define the true yield stresses of waxy crude oil is direct measurements using a controlled stress rheometer. For dynamically cooled sample, the effect of cooling rate and shear rate were studied for both static yield stress and dynamic yield stress. For statically cooled sample, the effect of cooling rate was studied for both static yield stress and dynamic yield stress. Below are the experimental procedures for the various yield stress measurement.

Static yield stress measurement: This measurement is a new derived method for this study. The geometry 4 cm roughened plate was used for dynamic cooling to minimize the wall slip effect. The gap was set to 800 micron meter. Upon placement of sample onto the peltier plate of the rheometer, the sample was heated at 45°C and sheared at 10 $\sec^{-1}$ for 3 min. It was then left to rest for 2 min. The sample is sheared constantly at 10 $\sec^{-1}$ for 2 min with the temperature at 45°C in order to destroy the sample properly before further measurements. The sample is then left to rest for 2 min. An oscillatory temperature ramp step was done with constant strain of 0.5% and angular frequency of 1 rad $\sec^{-1}$ at cooling rate 1°C $\min^{-1}$ with temperature reduced from 45 to 20°C. This was performed in order to understand the evolution of the gelling process of the waxy crude oil. The sample is then again heated at 45°C and left to rest for 2 min. A temperature ramp step with a constant shear rate of 10 $\sec^{-1}$ was then applied with temperature reduced from 45 to 20°C. This was done at a cooling rate of 1°C $\min^{-1}$. The sample is then left to rest for 2 min. Strain sweep test was performed from 0.01 to 100% at temperature 20°C with angular frequency of 1 rad $\sec^{-1}$ for the gel-yielding. Subsequently, the sample was heated at 45°C and left to rest for 2 min. The sample is sheared constantly at 10 $\sec^{-1}$ for 2 min with the temperature at 45°C in to check whether the viscosity of the sample is the same as in the initial state. For dynamic cooling, to study the effect of cooling rate on the yielding behaviour of the gel, the experiment was repeated with varying various cooling rate which are 1, 3, 5, 7 and 10°C $\min^{-1}$ and was tested with shear rate at 10 $\sec^{-1}$. To study the effect of shear rate, the experiment was repeated with various shear rates which are 5, 10, 20, 50, 75 and 100 $\sec^{-1}$ and was tested with cooling rate at 1°C $\min^{-1}$. For static cooling, the same procedures were taken except for the temperature ramp step where the shear rate is zero and 2 cm roughened plate was used. The experiment was repeated with varying various cooling rate which are 1, 3, 5, 7 and 10°C $\min^{-1}$ to study effect of cooling rate on the yielding behaviour of the gel.

Dynamic yield stress measurement: This measurement is also a newly derived method for this study. For dynamic cooling, the 4 cm roughened plate was used to overcome the wall slip problem. The gap was set at 800 micron meter. The sample was heated at 45°C and sheared at 10 $\sec^{-1}$ for 3 min upon placement of sample onto the peltier plate of the rheometer. It was then left to rest for 2 min. A temperature ramp step with a constant shear rate of 10 $\sec^{-1}$ was then applied with temperature reduced from 45 to 20°C and cooling rate of 1°C $\min^{-1}$. The sample is sheared constantly at 1 $\sec^{-1}$ for 15 min with the temperature at 20°C. This is the steady state peak hold test to determine the minimum stress exerted at that particular shear rate. The sample is then sheared constantly at 10 $\sec^{-1}$ with the temperature 45°C for 3 min to remove the shear history of the sample before repeating the temperature ramp step and holding the sample with an increasing shear rate of 1, 5, 10, 20, 40, 60, 80 and 100 $\sec^{-1}$. The experiment was repeated with various cooling rate which are 1, 3, 5, 7 and 10°C $\min^{-1}$ to study the effect of cooling rate on the yielding behaviour and it was conducted with shear rate 10 $\sec^{-1}$. To study the effect of shear rate, the experiment was repeated with various shear rates which are 5, 10, 20 and 50 $\sec^{-1}$ and was tested with cooling rate 1°C $\min^{-1}$. For static cooling, the same procedures were taken except for the temperature ramp step where the shear rate is zero and 2 cm roughened plate was used. The experiment was repeated with varying various cooling rate which are 1, 3, 5, 7 and 10°C $\min^{-1}$ to study effect of cooling rate on the yielding behaviour of the gel.

RESULTS AND DISCUSSION

Results are represented as strain versus time during creep test as can be seen in Fig. 4.

As can be seen in Fig. 4, the results are represented as the average of three runs for each shear stress. A constant shear stress was applied for a time period of 2 h. For all four shear stress, the strain becomes constant after some time. Both the shear stress of 20 and 80 Pa, the strain becomes constant after 350 sec and for shear stress 30 and 40 Pa, the strain becomes constant after 750 sec. From these results it is difficult to consider the yield point at any of this shear stress as none of the slope of the curve shows a rapid creep strain increase.

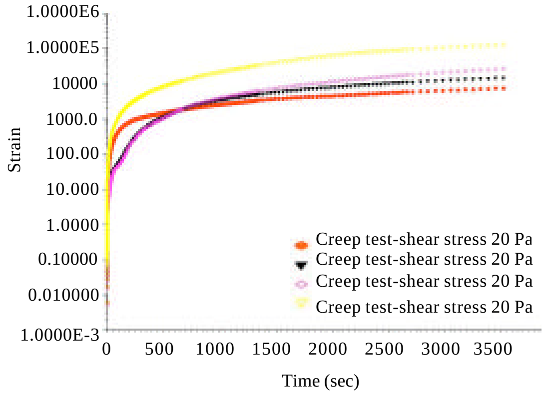

Fig. 4: Creep test for dynamic cooling-shear stress 20, 30, 40 and 80 Pa

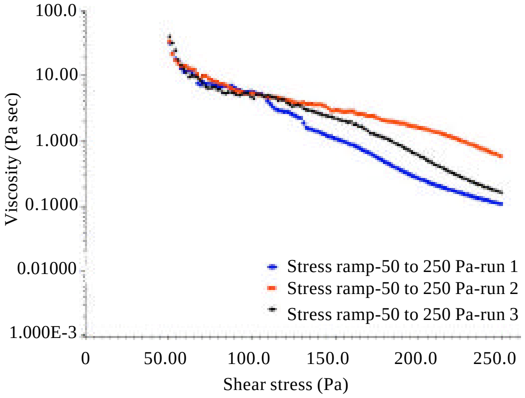

Fig. 5: Stress ramp for dynamic cooling

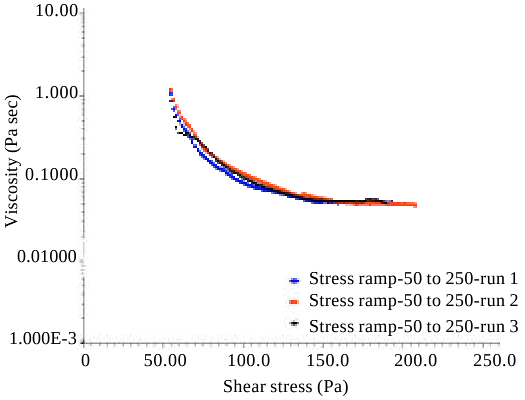

Fig. 6: Vane technique-stress ramp test

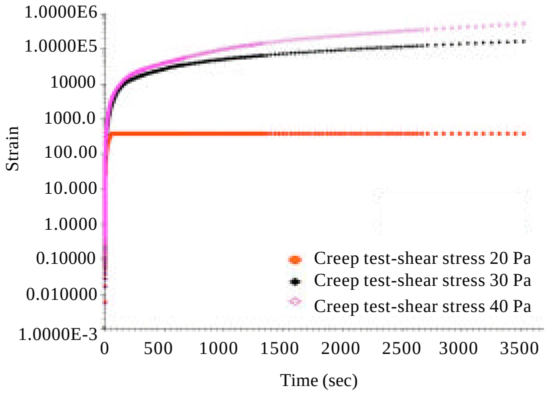

Fig. 7: Vane technique-creep test

Figure 5 shows the results for the stress ramp. As can be observed, the rapid decrease of viscosity can be considered at shear stress 50 Pa. However, it can be accurately considered as the yield stress point because the experiment started at 50 Pa and if the values are lowered perhaps a better representation of data can be observed but the rheometer is not able to achieve the shear stress lower than 50 Pa where the results are not obtained at all.

Figure 6 shows the results for the stress ramp test and Fig. 7 shows the results for creep test.

From Fig. 6, the sudden decrease of viscosity is observed at 50 Pa but it can’t accurately be considered at the yield point due to the limitation of the rheometer where it can’t achieve shear stress lower than 50 Pa. Therefore, this method is not good enough to represent the yield point for the waxy crude oil.

As can be seen in Fig. 7, the results are represented as the average of three runs for each shear stress. For shear stress 20 Pa, the strain becomes constant at 100 sec and for both shear stress 30 and 40 Pa, the strain becomes constant at 250 sec. Therefore, this method is not reliable enough to determine the yield point of the waxy crude oil.

In conclusions, all the above method’s results obtained from this measurement were not reliable and the repeatability was very poor. Therefore, a new method was derived for the yield stress measurement which defines the static yield stress and dynamic yield stress.

For dynamic yield stress, it was determined by increasing the shear rate and taking the minimum shear stress obtained from each shear rate. It was then extrapolated with shear rates versus shear stress and the value of dynamic yield stress is at the point where shear rate is zero. To ensure repeatability, the experiment was repeated three times and the results presented are the average of it.

Figure 8 shows the results of the dynamic yield stress of each cooling rate for static cooling. For cooling rate 1°C min^{-1}, the dynamic yield stress is 23.04 Pa, 3°C min^{-1} is 18.53 Pa, 5°C min^{-1} is 22.02 Pa, 7°C min^{-1} I 17.45 and 10°C min^{-1} is 26.04 Pa. Hence, we can’t conclude the trend on how the cooling rates affect the dynamic yield stress for static cooling.

As can be seen in Fig. 9, the effect of cooling rate on the dynamic yield stress for dynamic cooling is not that clear. The results obtained do not show any obvious trend on the dynamic yield stress can be affected by the cooling rate. The dynamic yield stress decreases from 35.12 to 24.08 Pa with increasing cooling rate from 1 to 3°C min^{-1}. Then, at increasing cooling rate from 5, 7 to 10°C min^{-1} and the dynamic yield stress increases from 24.66, 34.61 to 35.48 Pa.

The effect of shear rate on dynamic yield stress for dynamic cooling was studied using the following shear rates which are 5, 10, 20 and 50 sec^{-1}. From Fig. 10, it can

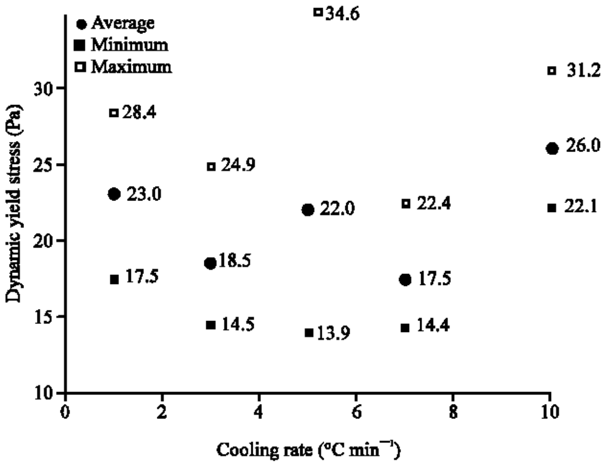

Fig. 8: Effect of cooling rate-dynamic yield stress-static cooling

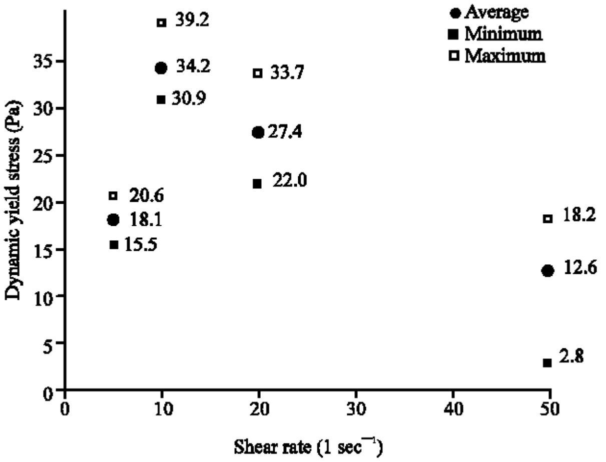

Fig. 10: Effect of shear rate-dynamic yield stress-dynamic cooling

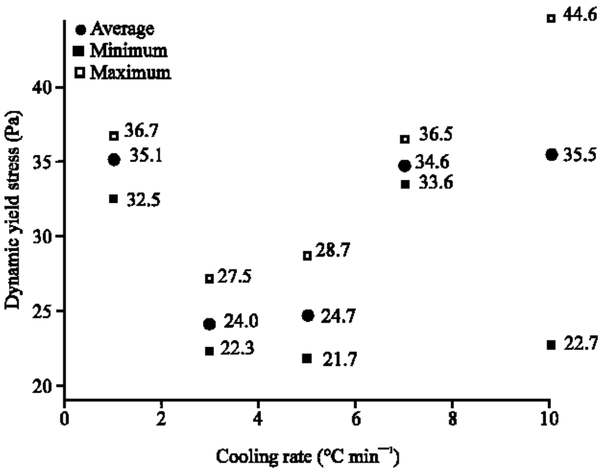

Fig. 9: Effect of cooling rate-dynamic yield stress-dynamic cooling

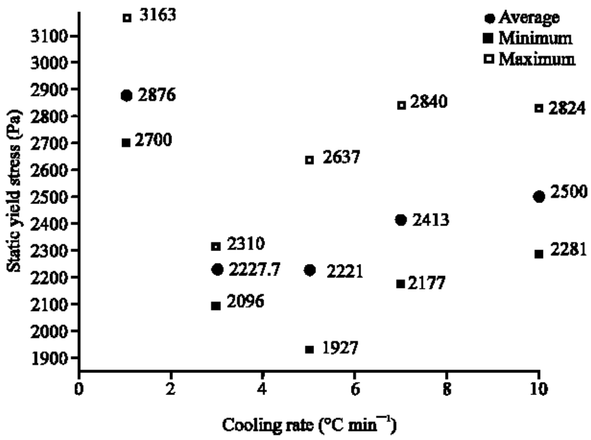

Fig. 11: Effect of cooling rate-static yield stress-static cooling

be seen that at low values of shear rate which is 5 to 10 sec^{-1} and the dynamic yield stress increased from 18.09 to 34.12 Pa. Thereafter, the yield stress decreased from 27.4 to 12.59 Pa with increasing shear rate of 20 to 50 sec^{-1}. The results produced are in agreement with the work done by Venkatesan *et al*. (2005) in one of his research. A maximum in the dynamic yield stress was observed at the shear rate of 10 sec^{-1}. The maximum value of the dynamic yield stress is due to the differences in the wax crystal structure formed under the various shear rates. The initial increase of yield stress is due to the aggregation of the wax crystals which will results in larger crystals and stronger network. Further increased of the shear rate will break down the structure which will result in smaller crystals and weaker network. Hence, the yield stress decreases.

Static yield stress was determined by conducting the strain sweep step from 0.01 to 100% with fixed angular frequency of 1rad sec^{-1}. The point where it deviates from the linear of the stress-strain relationship, it considered as the static yield stress. The effect of cooling rate on the yield stress was studied for both static and dynamic cooling. The effect of shear rate on the yield stress was studied only for dynamic cooling. The results shown in the following graphs are the average of three runs of each experiment with different cooling rates and shear rates to ensure the repeatability.

Figure 11 shows the result of the average static yield stress of each cooling rate for static cooling. With an increasing cooling rate of 1, 3 and 5°C min^{-1}, the value of the static yield stress decreases from 2876, 2227.7 and 2221 Pa. As the cooling rate increases further from 7 to

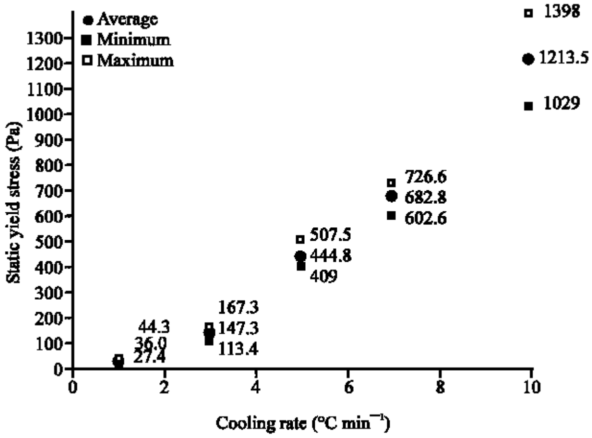

Fig. 12: Effect of cooling rate-static yield stress-dynamic cooling

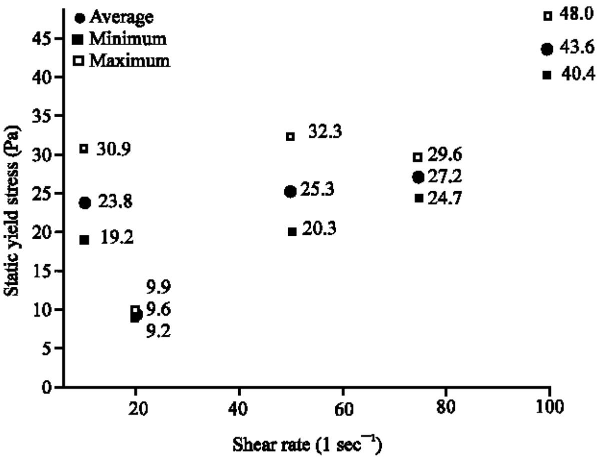

Fig. 13: Effect of shear rate-static yield stress-dynamic cooling

10°C min^{-1}, the static yield stress increases from 2413 to 2500 Pa. However, we can't see a specific trend on how the cooling rates affects the static yield stress for static cooling.

The effect of cooling rate on the static yield stress is more obvious for dynamic cooling. The results are shown in Fig. 12. The value of static yield stress increased with increasing cooling rate. For cooling rate 1°C min^{-1} is 36.01 Pa, for 3°C min^{-1} is 147.43 Pa, for 5°C min^{-1} is 444.8 Pa, for 7°C min^{-1} is 682.77 Pa and for 10°C min^{-1} is 1213.5 Pa. The possible reason for this is that the shear stress will break the crystal network as it forms. Thus, when the cooling rate is low, the waxy crude oil is exposed to the shear stress for a longer time before the sample gels up. Due to this, the gel built up is weaker. When the cooling rate is high, the sample gel up faster and the waxy crude oil is exposed to shear stress for a shorter time. This will results in stronger gels therefore the stress needed to break this gel is greater. Therefore, the value of yield stress increases with increasing shear rate.

The effect of shear rate on the static yield stress was studied using various shear rates ranging from 5, 10, 20, 50, 75 and 100 sec^{-1} under the fixed cooling rate of 1°C min^{-1}. Figure 13 shows the variation of yield stress with the shear rates. It can be seen that when the shear rates increases from 5, 10 to 20 sec^{-1} and the static yield stress decreased from 27.36, 23.75 to 9.57 Pa. However, with further increment of shear rate from 50, 75 to 100 sec^{-1} and the static yield stress increases from 25.32, 27.28 to 42.28 Pa. The minimum in the static yield stress is due to the differences in the wax crystal structure formed under the various shear rates.

At low values of shear rates (<20 sec^{-1}), the yield stress decreases with an increasing shear rate as it breaks down the structure even more and smaller crystals are formed which indicates the crystal network is weaker. Whereas for high values of shear rate (>50 sec^{-1}), the yield stress increases with an increasing shear rate as it breaks down the structure lesser and larger crystals are formed which indicates the crystal network is stronger. Possible explanation for this can be that at higher shear rates the aggregations of wax crystals are larger than the breakage. This scenario is been observed with using cooling rate of 1°C min^{-1}. The result might show a different trend with using higher or lower cooling rates.

CONCLUSION

Yield stress is the rheological property that determines the strength of the waxy crude oil. The knowledge of the yield stress is crucial in estimating the force required for pigging the waxy crude oil or the pressure needed to restart the completely clogged oil pipelines. Due to the poor repeatability and not reliable results obtained from creep test, stress ramp test and using vane technique, new method was developed for the yield stress measurement.

The results of static yield stress were produced by performing oscillatory test and dynamic yield stress was produced by extrapolating from high shear rate to the zero shear rates in the controlled stress test. The static yield stress can be applied to the design for predicting the restart pressure where else the dynamic yield stress can be used in the calculation of the pressure-flow relationship when the oil is flowing after fracture. All results produced shows good repeatability and it was presented as the average of three runs. The effect of cooling rate on the static and dynamic yield stress for sample cooled statically is not that clear. There was no obvious trend that can be concluded. However, when the gel is formed under dynamic cooling, the yield stress increases with increasing cooling rate for static yield stress but for dynamic yield stress the trend is unclear. Experimentation with a range of shear rates have revealed that, at a fixed cooling rate, the yield stress reaches a maximum point at a moderate value of shear rates. This scenario is observed for dynamic yield stress. The initial increase of yield stress is probably due to the aggregation and entanglements of the wax crystal. The subsequent decrease in yield stress at high values of shear rate is due to the breakage of the wax network. For static yield stress, the results obtained are the opposite of it. At moderate shear rates value, the yield stress reaches a minimum point. Therefore, the initial decrease of yield stress might be due to the breakage of the wax network and the subsequent decrease is due to the aggregation of the wax crystal is greater than the breakage.

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