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Yield Stress Measurement and Thixotropic Behaviour of Waxy Crude Oil from the Malay Basin.

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ABSTRACT

Predicting the restart pressure, specifically the yielding point, for any 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 crude. In pipeline transportation of crude oil it is also important to characterize the thixotropic behaviour of the crude in order for the flow characteristics to be well predicted and understood. Various methods have been proposed to quantify thixotropy, including the well-known "thixotropic-loop" tests. As for the yielding behavior, i.e. the yield stress, existing method defines two yield stresses, static and dynamic. While static yield stress is more difficult to measure since most occur at very low stresses beyond the measurement capability of some testing equipments and that the measuring methods are still subjected to debates, dynamic yield stress is relatively easier to be obtained and requires linear extrapolation of viscosity data at low shear rates. In this study, the thixotropic behaviour of a waxy crude oil from the Malay basin is explored together with the effects of temperature. Measurements for static yield stress were also conducted via oscillatory testing while the dynamic yield stress through the steady state peak hold method for the waxy crude oil, cooled statically and also dynamically to produce the gel. All measurements were performed utilizing a rheometer with minimized wall slip effects. Data showed the extent of thixotropy can be quantified above and below the Wax Appearance Temperature of the crude oil. For the yield stress measurements, interesting findings show that static yield measurement is possible using the proposed method with relatively higher repeatability. The static yield stresses obtained are compared to the dynamic yield stresses measured using standard method available in the literature. The outcome of the study could then be used to fully understand the nature of the gelled crude.

Keywords: Waxy crude oil, static yield stress, dynamic yield stress, thixotropic.

1.0 INTRODUCTION

In the oil and gas fields, about 20% of the petroleum reserves produced and pipelined are crudes containing large proportions of high molecular weight paraffin compounds which are also known as waxy crude oil [1]. These crude oils are light or intermediate crude containing as low as 2% of paraffin wax [2, 3]. These wax molecules are dissolved at reservoir temperatures (70-150°C) and pressures (50-100MPa).

One of the primary interests in dealing with the transportation of the waxy crude is the issue of restarting the flow. During temporary shutdown of production operation at platforms which is usually performed for maintenance or emergency reasons, the crude is static and under such conditions, the temperature within the pipeline is subjected to external factors and drops significantly, especially for subsea installations and in the arctic regions. The temperature decrease beyond the wax appearance temperature (WAT) will result in formation of paraffin crystals [4], which is absent above WAT when the crude oil behaves mainly Newtonian. Below WAT, non-Newtonian behaviour is increasingly significant with the decrease in temperature due to the increase in the amount of wax crystals [5]. As the temperature drops further the crude oil transforms into gel-like structure of which the yield stress and thixotropic behaviour are observed and further exaggerated with aging. During the process, the waxy crude undergoes thermal shrinkage where gas voids appear resulting in supposedly, compressible gel.

Waxy crude could also gel under flowing conditions. Once the crude oil leaves the high temperature and pressure reservoir conditions and flows through the pipelines within low temperature surroundings, the crude oil temperature will begin to drop due to the heat loss to these surroundings. Further temperature decrease will have the same effect as that under static conditions but with different wax crystal structure [5]. Gel structure will form if the temperature decreases low enough eventually leading to the cease of flow. Thermal shrinkage and gas void formations are also anticipated under such conditions.

Figure 1 shows the cross sectional view of a cut-away segment of a pipeline filled with gelled crude oil [8]. As can be observed, the area available for the crude oil flow is drastically reduced due to buildup of wax crustals with decrease of temperature. If the conditions do not improve but further aggravated due to absence of corrective measures or failure of measures such as pigging, the pipeline will eventually clog. Under such circumstances, the pipeline that is totally clogged would have to be replaced. This would further increase the operational costs. According to the US Department of Energy, replacing sections of pipeline in water depths of about 400m can cost up to $1million/mile [8].

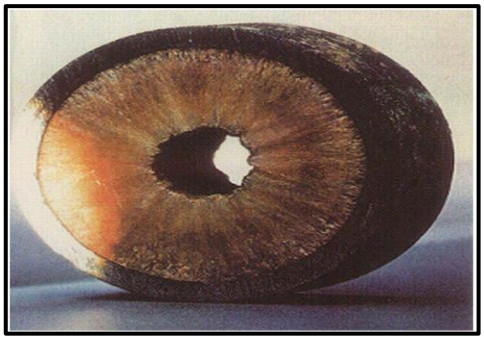

Figure 1: Cross-sectional view of a plugged pipeline [8].

There are two stages in pipeline restart; the first stage is to apply large pressure to mobilize the gelled crude. The time required for this procedure would depend on the crude yield stress, length of the pipeline and the compressibility of the gelled crude. The second stage involves injecting fresh warm oil or any other low viscosity Newtonian fluid such as seawater or diesel at the inlet to flush the remaining gelled crude out of the pipeline [1].

Current industrial practice determines the restart pressure using a conservative relation $\Delta p=4\tau_y L/D$ where τ_y is the yield stress, L is the pipe length and D is the pipe diameter. The yield stress used in this relation is measured conventionally by ????. However, this method is also subjected to debates. Additionally, the relation neglects presences of the gas and water in the crude oil as well as its thermal shrinkage and the rheological behaviour when gelled. This will lead to an extremely over-predication of the restart pressure causing higher capital expenditure when designing the facilities [9].

1.1 Thixotropic behaviour of the waxy crude

Thixotropic is one of the oldest known rheological phenomena and, to date, there is even remaining confusions to the definition of thixotropy [10] as the definition in rheological context has changed over the years. The American Society of Rheology as quoted by Reiner and Scott-Blair [ref??] has defined thixotropy as "that property of a body by virtue of which the ratio of shear stress to viscosity is temporarily reduced by previous deformation" [11]. Later on thixotropy was defined as "a comparatively slow recovery, on standing of the consistency lost through shearing" [12]. Recently, thixotropy has been defined by Barnes, Hutton and Walters [13] as the "decrease of viscosity under constant shear rate followed by a gradual recovery over time when the shear rate is removed".

The changes in the microstructure are the fundamentals of thixotropy which is often very complex and still poorly understood. As a result, a general rheological model has not yet been developed to understand fully the features of thixotropy. Nonetheless, this phenomenon is very usual in industrial and natural systems [12].

The characterization of thixotropic properties of liquids can be classified and mainly based on [14]:

i). Measuring the flow curve from zero to a pre-determined maximum shear rate, holding at a certain time in order to maximize the breakage of built structure and measuring a flow curve back from that shear rate to zero. If hysteresis exists between the increasing and decreasing curve it will be considered as thixotropic and the area under the curve described the extent of it.

ii). Measuring the regeneration of the structure. The sample is sheared continuously for a pre-determined shear rate with the aim to break the built structure. The shear rate is then dropped to a lower value. The subsequent increased of shear stress is tracked as a function of time. The lowest possible shear rate must be chosen so that straining of the rebuilt of structure will be minimized.

iii). Determining the shear stress of fluid τ (t) with time at constant shear rate.

The research work presented here focuses on the hysteresis loop technique to assess the degree of thixotropy. When transient data are plotted as shear stress versus shear rate, a thixotropic sample will describe a hysteresis loop. The area and shape of the loop can differ strongly according to the material [15].

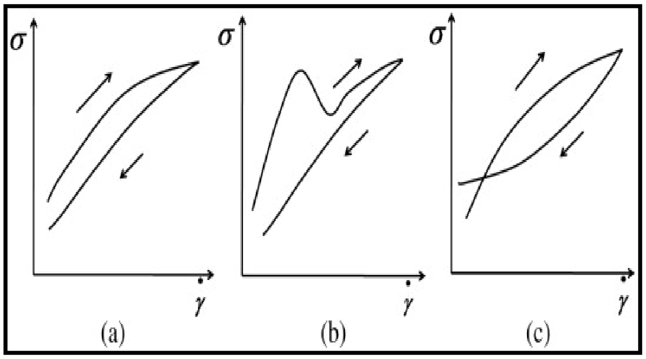

Figure 2: Possible shapes of hysteresis loop [15].

A simple hysteresis loop is shown in Figure 2(a). Figure 2(b) represents a case where breakdown of the initial structure after starting up dominates the time evolution of the stress which results in a stress overshoot. A reduction of stress with increasing shear rate can cause shear banding and heterogeneous shear rate distribution in the sample. When the shear rate is relatively low it induces structure formation and the hysteresis loop can then be represented as in Figure 2(c) [15].

1.2 Yield stress measurement

In order to restart a flow, the respective structure, in this case the gelled crude oil, 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 [8].

A yield stress is the stress corresponding to the transition from elastic to plastic deformation [2]. Existingly, there are two types of yield stresses, a static yield stress and a dynamic yield stress. The static yield stress (τ_s) is taken as 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 an estimated parameter which can be used in defining the oil properties at the final sheared state and therefore it is not a true material property relating to the yielding process [6, 17]. 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 start 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 to initiate flow and ensure pipeline restart, engineers are keen to use the static yield stress, the stress value when the breakage occurs [17]. Supporting this, Frigaard et. al. [1] also proposed that the static yield stress is useful in the pipeline system design stage with the information of the time scale of the measurement. The dynamic yield stress is more crucial in the calculation of the pressure-flow rate relationship when the oil is flowing in an equilibrium state after fracture [1].

Cheng Chang and Petter [16] has conducted a stress ramp experiment for a waxy crude oil known as DH19 at a low temperature [6]. Utilizing a controlled stress rheometer and a cone and plate geometry, the authors discovered a unique structural breakdown at low shear rates shown in Figure 3. For the crude oil utilized, the authors found that the 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. The shear stress at the point of fracture is the most important value and normally taken as the yield stress [16].

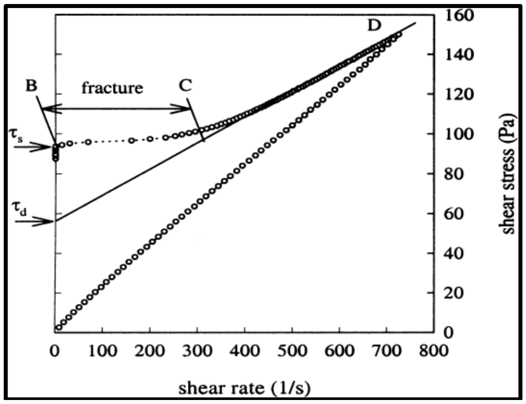

Figure 3: Differences between static and dynamic yield stress for waxy crude oil (Controlled stress test. Stress sweep was performed from 0 to 150Pa to 0) [6].

The authors further discussed the three stages of the yielding behavior; an elastic response, a creep response and a fracture, as illustrated in Figure 4, a typical curve recorded for a waxy crude oil. These data can be obtained by conducting an oscillatory test with a gradual increase of controlled stress at a fixed low frequency.

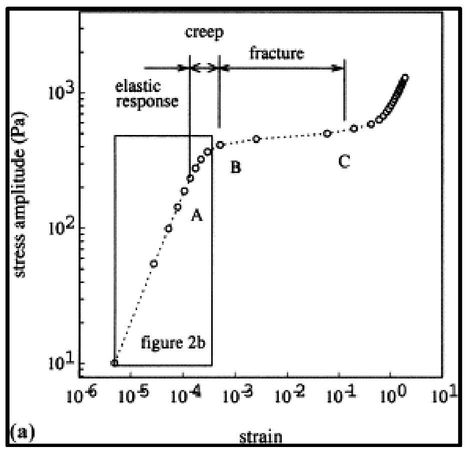

Figure 4: Yielding process of waxy crude oil (DH19) [16].

Prior to point A, the initial linear regime represents an elastic response where the strain increases linearly with shear stress. Creep occurs after point A with the stress-strain relationship slowly deviating from a linear relationship. Fracture starts at point B, showing the breakage of the oil microstructure where 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 [16].

Venkatesan [8] 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 a product of Exxon. The WAT of the 5% wax in oil mixture was 28.3°C. Figure 4 shows the rheometric responses with varying shear stress.

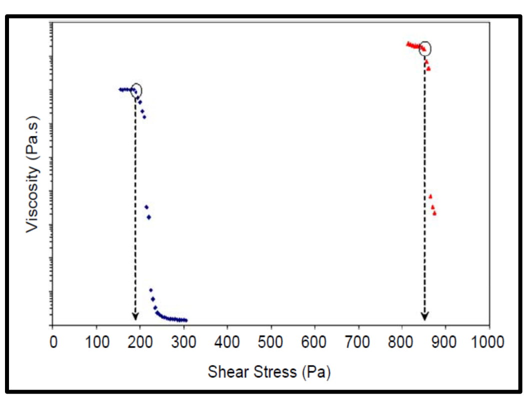

Figure 4: Transient plot of typical rheological experiments [8].

A creep response is observed initially when the gel is subjected to loadings. At about 190Pa for one sample and 850Pa for another, the point of fracture is reached as there is sudden decrease in the viscosity and the gel is considered to yield [8].

Russell and Chapman [ref??] and Chang and Ronningsen [ref???] studied the effect of cooling rate on the strength of the waxy crude oil gels formed under static cooling. However the results by these researches are in contrary. Russell and Chapman [ref??] observed stronger gels produced with larger cooling rate using cone and plate?. Ronningsen and Chang [ref??] however found that smaller cooling rates produced stronger gels with cone and plate geometry??. The possible explanation for the decrease in yield stress, hence weaker gels as proposed by Ronningsen and Chang [ref??], 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, hence stronger. The rate of crystal precipitation is faster for larger cooling rate resulting in limited time for the crystal growth leading to smaller crystals and weaker gel.

The effect of cooling rate on the strength of gel formed under dynamic cooling is studied by Venkatesan [8]. An interesting phenomenon and explanation, albeit opposite behavior compared to statically cooled gel, is observed. The author discovered stronger gels with increasing cooling rate and postulated that the shear stress introduced during cooling tends to break the crystal network even as it forms. When the cooling rate is low, the waxy crude oil is subjected to the shear stress for a longer period before it gels leading to a weaker structure. At higher cooling rate, the gelation happens at a faster rate and the waxy crude oil is subjected to the shear stress for a shorter period of time resulting in relatively stronger gel.

To date, there are various methods utilized and reported in the study of yield stress available in the literature. Nevertheless, there is no standard test for defining the yielding quantity of waxy crude oils that has been be implemented by the petroleum industry due to poor repeatability in any given instrument and poor reproducibility between different experimental setups. The possible reason would be due to the thixotropic nature of the crude itself and hence, suggesting that standard preconditioning treatment is very critical. Another potential problem while testing for the yield stress using a rheometer is the possibility of slip at the surface leading to erroneous data and misleading conclusions. The wall slip can be observed in terms of lower viscosity and yield stress measured if smooth geometries were to be utilized in the measurements [4]. Other possible reasons include instrumental effects such as instrument inertia, damping characteristics of the rotation body and sensitivity to the external disturbances [19].

2.0 METHODOLOGY

The research work discussed in this paper is divided into two areas: thixotropic study on the waxy crude and yield stress measurements for statically and dynamically cooled sample. The measurements were conducted using AR G2 controlled stress rheometer by TA Instrument. The sample investigated is a crude oil from a field in Malaysia. The wax content is reported by Petronas Research Sdn Bhd to be 18wt% with Wax Appearance Temperature (WAT) of 38°C.

The temperature at which the wax crystals appear and starting to dominate the flow behavior can also be confirmed via oscillatory temperature ramp conducted in this research study. Figure 5 shows the evolution of the elastic modulus of the sample, G', representing the solid-like behaviour and the loss modulus, G'', describing the liquid-like behavior, upon cooling from 45°C to 20°C with an imposed shear rate of $10s^{-1}$ and cooling rate of 1°C/min. The gelation temperature, defined from the rheological context to be the point at which the solid-like behaviour of a complex fluid takes predominance over its liquid-like behavior [18], can be deduced from the Figure to be at 38°C. Below the gelation temperature, the sample is dominated by liquid-like behavior above which, due to the precipitation of wax crystals leading to gelation, the solid-like behavior is dominant.

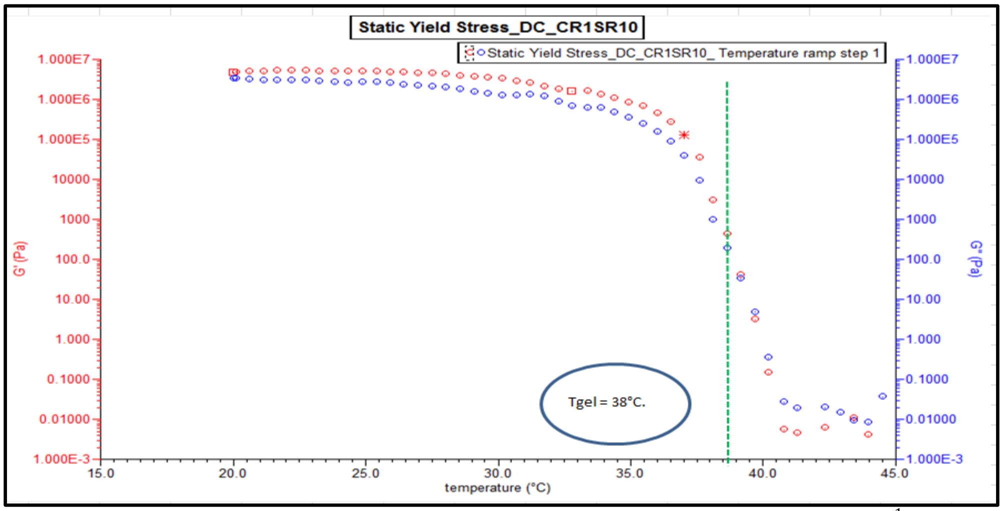

Figure 5: Gelation temperature for cooling rate 1°C/min and shear rate $10s^{-1}$.

Throughout the measurements, the slip effect, as reported in the recent literature [ref??] to be possible for waxy crude oil, is minimized by using a roughened plate geometry. To minimize possible evaporation during the measurement, a solvent trap was also utilized. A standard preconditioning treatment is adopted to enhance repeatability where upon placement of sample onto the peltier plate of the rheometer, the sample was heated at 45°C and sheared at $10s^{-1}$ for 3 minutes to remove any shear history experienced by the sample especially during loading. It was then left to rest for 2 minutes. A temperature ramp step with a constant shear rate of $10s^{-1}$ was then applied with temperature reduced from 45°C to 20°C to produce a gel for dynamic cooling scenario. Zero shear rate is applied for static cooling. For both cooling scenarios, the cooling rate is 1°C/minute with a gap setting of 800 micron. A minimum of three runs were conducted for each measurement with the results shown in the following figures to be the average of all the runs conducted for each measurement setting. (PLEASE PLOT AVERAGE, MINIMUM AND MAXIMUM VALUES FOR THE RUNS SO THAT WE KNOW THE EXTENT OF THE VARIATIONS)

2.1 Thixotropic behaviour of the waxy crude

To achieve the hysteresis loop, the steady state flow step was performed, after the preconditioning treatment, with increasing shear rate from 1 to $1000s^{-1}$ at a constant temperature of 20°C. The sample is then held at a shear rate of 1000 s^{-1} for 30 minutes to maximize the breakage of the built structure. The shear rate is then decreased from 1000 to 1 s^{-1}. The procedures were repeated for 30 and 40°C.

2.2 Yield stress measurement

Prior to any measurements of yield stresses, an oscillatory temperature ramp step was performed after the set of preconditioning treatment under the rheometer with constant strain of 0.5% and angular frequency of 1 rad/s at a cooling rate of 1°C/minute with temperature reduced from 45°C to 20°C. The procedure was undertaken in order to understand the evolution of the gelling process of the waxy crude oil.

Strain sweep was adopted, after the preconditioning treatment, in the measurements of the static yield stress from 0.01 to 100 % at temperature 20°C with angular frequency of 1 rad/s set for the gel-yielding. This method was modified based on the static yield measurement method proposed by Cheng Chang and Petter [16]. Once the strain sweep procedure is completed, the sample was heated at 45°C and left to rest for 2 minutes before being sheared again at $10s^{-1}$ for 2 minutes at 45°C to verify if the viscosity is recovered to its initial state. Steady state peak hold test is adopted in the measurement of dynamic yield stress where, after the set of preconditioning treatment, the gelled sample is subjected to shear of $1s^{-1}$ for 15 minutes at 20°C.

For dynamically cooled sample, the effect of cooling rate and shear rate were studied. For statically cooled sample, the effect of cooling rate was studied. Different cooling rates adopted were 1, 3, 5, 7 and 10°C/minute and was tested with a shear rate at $10s^{-1}$. The effects of shear rate was studied with the shear rates varied at 5, 10, 20, 50, 75 and $100s^{-1}$ and was tested with a cooling rate of 1°C/minute.

3.0 RESULTS AND DISCUSSIONS

3.1 *Thixotropic behaviour of waxy crude oil* *(could you check again the calculation for the area under the curve? The value does not tally with the size shown in the figure)*

The hysteresis in the viscosity data for the waxy crude oil, gelled dynamically, measured at three different temperatures is shown in Figure 6. The low to high indicates increasing the shear rate from 1 to 1000s^{-1} where else the high to low indicates the decreasing shear rate from 1000 to 1 s^{-1}. As shown, thixotropic behaviour in crude oil is more crucial and obvious at temperature below the WAT of the crude which is 38°C. Though it is anticipated that the crude behaves essentially Newtonian above WAT, the hysteresis found at 40°C indicates otherwise. This is possibly due to the presence of a small number wax crystal, as the temperature is close to WAT giving some non-Newtonian characteristics to the flow. The viscosity at this temperature, however, is still lower than that at 20 and 30°C indicating the small number of crystals present compared to the lower temperatures. The extent of thixotropy can further be quantified using the bounded area for the respective temperatures as tabulated in Table 1. The area within the bounded region is approximated using the Simpson's 1/3 rule of approximating integrals:

$$I = \int_a^b f(x)dx$$

where

$f(x)$ is called the integrand,
$a =$ lower limit of integration
$b =$ upper limit of integration

However, no specific trend is seen to the degree of thixotropy below WAT. For statically cooled sample, as shown in Figure 7, the extent of thixotropy is shown to be reducing with temperature as indicated by the area within the bounded region, also tabulated in Table 1.

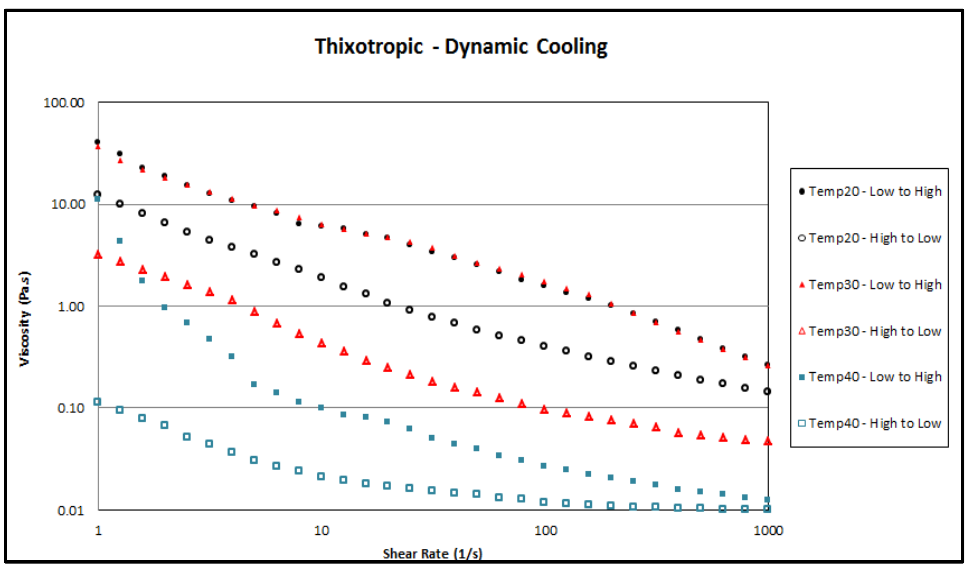

Figure 6: Thixotropic for Dynamic Cooling (PLEASE USE TECPLOT!!)

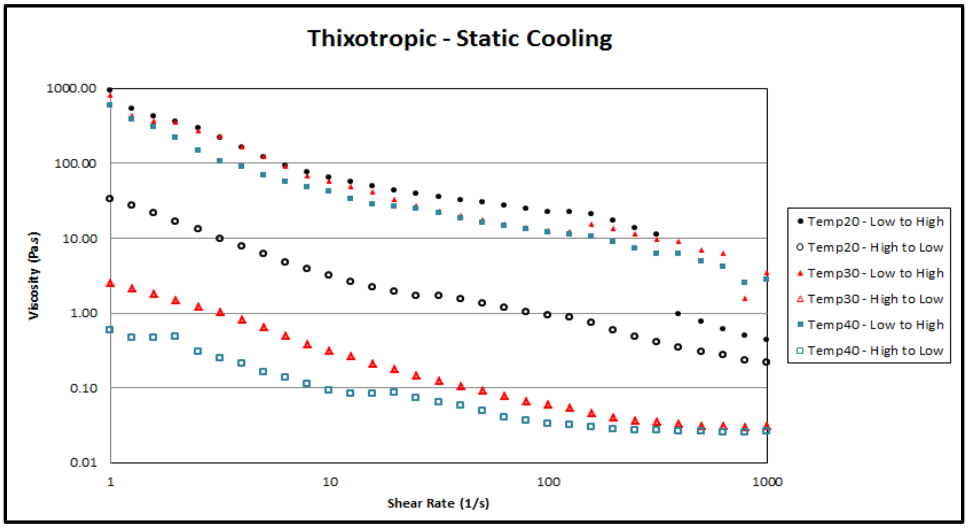

Figure 7: Thixotropic for Static Cooling

Further comparison between the area under the bounded region for both cooling scenarios shows higher values for statically cooled gel. The size of wax crystals formed under the different cooling scenarios is though to highly affect the degree of thixotropy. For static cooling, the crude oil is experiencing no shear upon gelling resulting in the formation of larger crystals as compared to the dynamically cooled gel where the applied stress is simultaneously destroying the crystals as it forms.

Table 1: Comparison of Area under the Curve for Static and Dynamic Cooling

Cooling scenarios	**Temperature (°C)**	**Area (unit^2)**
Static	20	9176.45
	30	12920.12
	40	6827.18
Dynamic	20	587.70
	30	754.76
	40	27.21

3.2 Yield stress measurement

3.2.1 Static Yield Stress

Static yield stress is the stress at which the stress-strain relationship deviates from linearity. Figure 8 shows the static yield stress values at different cooling rates for statically cooled gel. The figure shows an inflection point between 5°C/min and 7°C/min below which the yield stress decreases with cooling rate and above which increases. A totally different trend is observed, however, for the dynamically cooled gel as shown in Figure 9. Increasing cooling rates results in an increase of the static yield stress, i.e. high cooling rate results in stronger gel when cooled dynamically. This is in agreement with that observed by Venkatesan [8] and hence, the explanation provided by the author on the effects of shear to the formation of wax crystals holds.

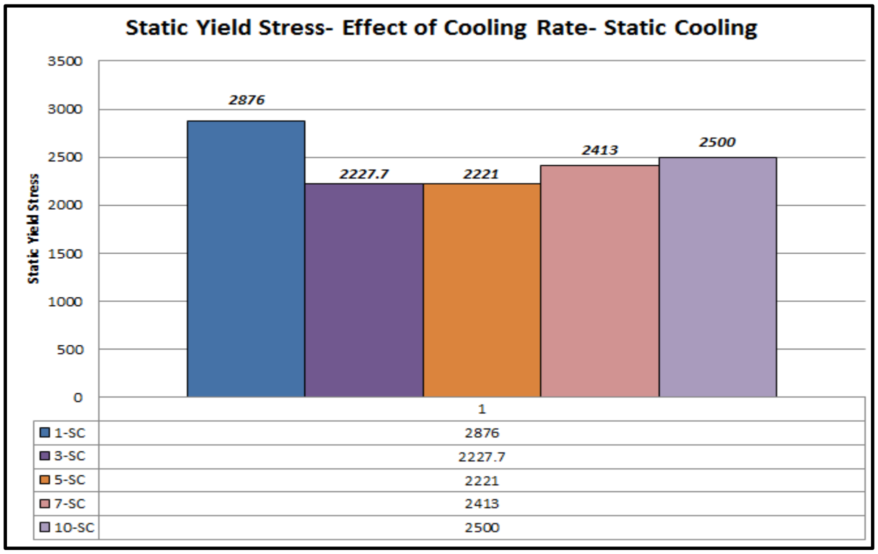

Figure 8: Static yield stress-effect of cooling rate for static cooling.

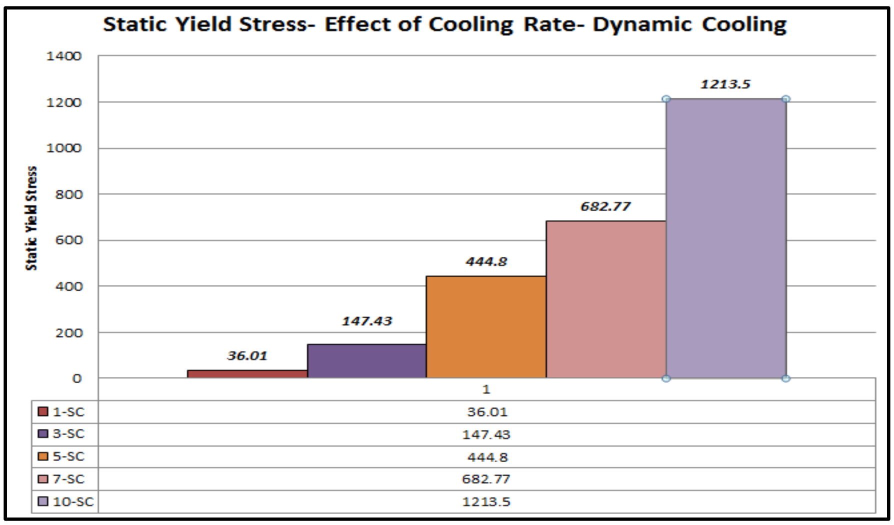

Figure 9: Static yield stress-effect of cooling rate for dynamic cooling.

Figure 10 shows the variation of yield stress with the shear rates for dynamically cooled gel. Decreasing trend is observed on the static yield stress with increasing shear rate below 20 s^{-1}. However, further increment of shear rate above $50s^{-1}$ results in increase of the static yield stress suggesting a possible existence of an inflection point.

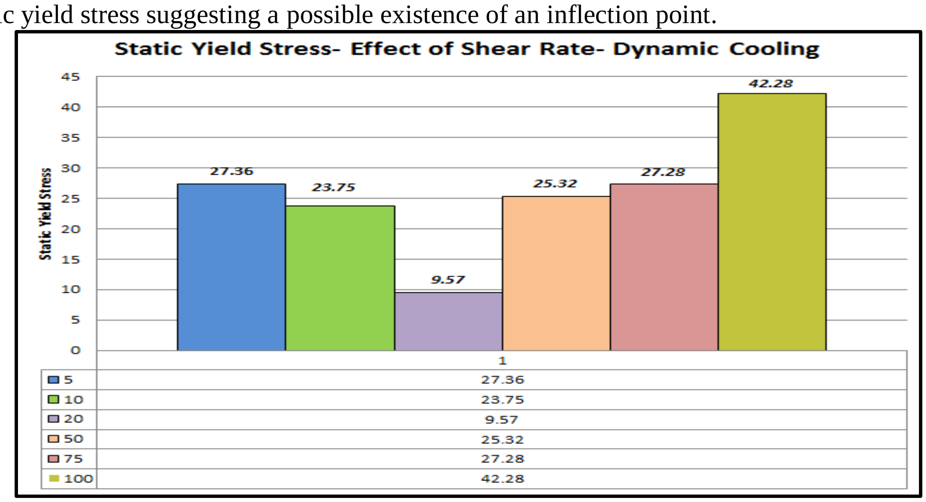

Figure 10: Static yield stress-effect of shear rate for dynamic cooling

3.2.1 Dynamic Yield Stress

Dynamic yield stress is the extrapolated values from the stress-strain relationship. The shear stress value for each strain is the minimum stress exhibited by the sample under the imposed constant shear rate. Figure 11 shows the dynamic yield stress of a statically cooled sample gelled under different cooling rates. However, no specific trend is observed with the cooling rates similarly with that of the dynamically cooled sample under different cooling rates shown in Figure 12.

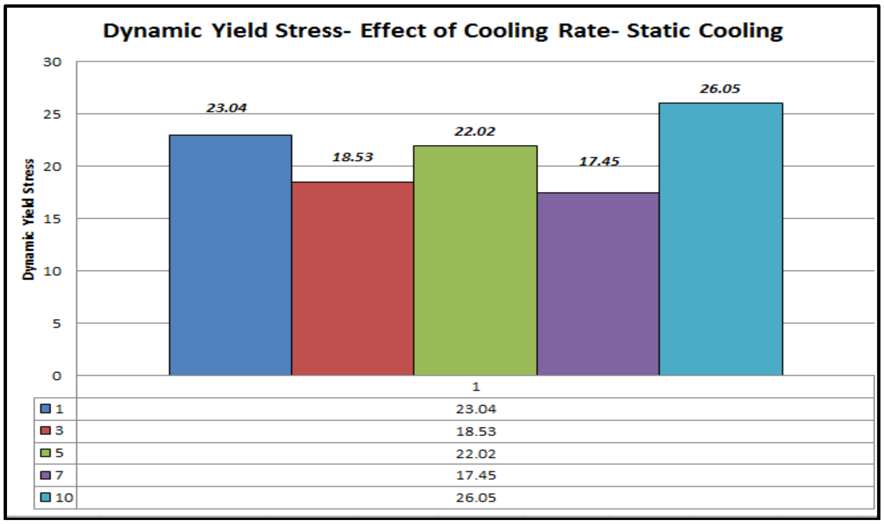

Figure 11: Dynamic yield stress-effect of cooling rate for static cooling.

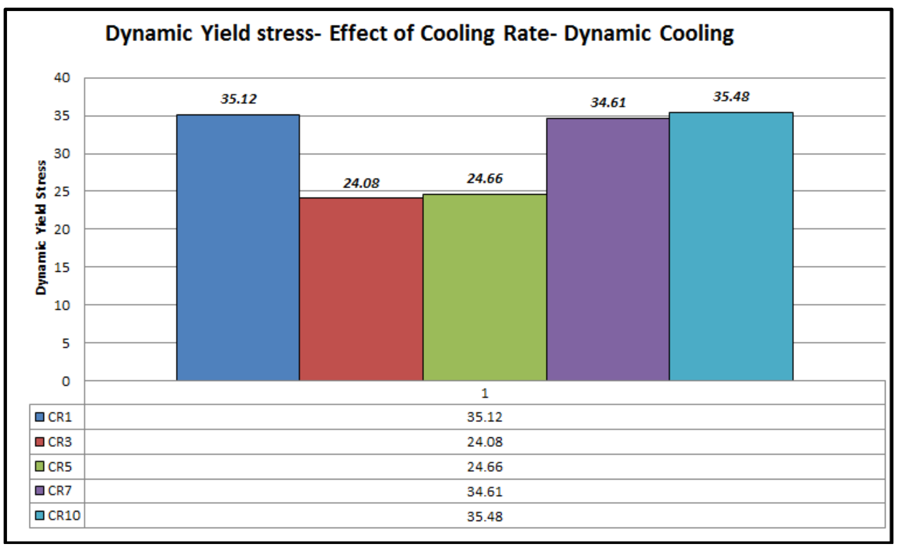

Figure 12: Dynamic yield stress-effect of cooling rate for dynamic cooling.

The effect of shear rate on dynamic yield stress for dynamically cooled sample is shown in Figure 13. The results were found to be in agreement of that by Venkatesan [8] where an inflection point is observed. At low value of shear rates (below 10 s^{-1}) the dynamic yield stress increased from 18.09 to 34.12 Pa but thereafter decreased from 27.4 to 12.59 Pa beyond 20 s^{-1}.

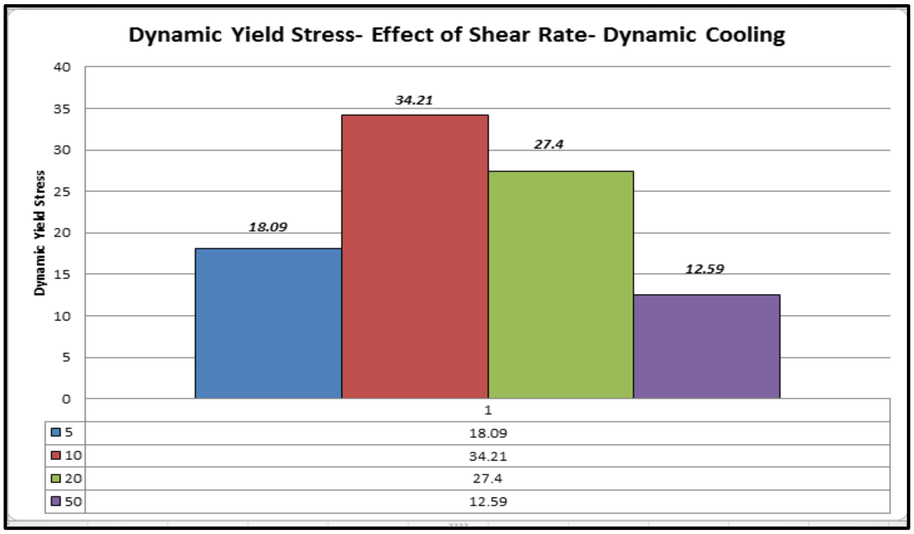

Figure 13: Dynamic Yield stress-effect of shear rate for dynamic cooling.

The summary of the yield stresses obtained for statically cooled gel and dynamically cooled gel is tabulated in Table 2.

Table 2: Static and Dynamic yield stress for statically and dynamically cooled gel

4.0 CONCLUSION

The research work presented in this paper outlined a set of preconditioning treatment essential for testing thixotropic waxy crude oil. Another method of determining static yield stress of the thixotropic waxy crude oil was also proposed and proven to yield greater repeatability. The improved method revealed interesting features such as the possible existence of inflection points with increasing shear rates. The values measured for the static yield stress were also found to be several orders of magnitude larger than the dynamic yield stress, obtained from adopted method in the literature. However, the repeatability of the dynamic yield stresses is relatively low and in some cases, failed to reveal any specific trends.

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