EFFECTS OF DRILLING MUD CONTAMINATION ON THE PROPERTIES OF WAXY CRUDE OIL

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ABSTRACT

Contamination of crude oil with drilling mud normally occurs during drilling process. The contamination may change the properties of crude oil leading to inaccurate assessment of the first extracted crude oil. Incorrect prediction of crude oil properties and behavior will subsequently lead to under- or over-designing of the upstream and midstream facilities. Therefore, the work aims to investigate the effects of drilling mud contamination on waxy crude oil properties such as pour point temperature, wax appearance temperature (WAT) and the yield strength of the waxy crude oil gel. Measurements conducted on the drilling mud contaminated mixture show no effect on the pour point, minimal changes to the WAT and considerable increase to the viscosity and yield stress values. Bottle test results indicate that drilling mud free oil layer which should be used in characterizing the crude oil exist only after 24 hours of gravity settling.

Keywords: Drilling mud contamination, waxy crude oil properties, yield stress.

INTRODUCTION

Waxy crude oils are aliphatic hydrocarbon having a high molecular weight paraffin with carbon number ranging from C18 to C65 [1]. The solubility of paraffin waxes is dependent on the temperature; it decreases with decreasing temperature. At reservoir conditions where the temperature ranges around 70°C and 150°C with a pressure range of 50MPa to 500MPa, the solubility of the solid paraffins in the crude oil is adequately high [2]. The wax molecules fully dissolve in the crude oil mixture resulting in a single-phase crude oil and in the absence of other components and contaminants the crude oil behaves predominantly Newtonian with low viscosity [3]. Once the crude oil leaves the reservoir and flows through cold pipelines placed on the seabed with temperature normally ranging from 20°C down to 5°C, the crude oil temperature begins to drop dramatically due to the heat loss to the surroundings, resulting in the crude oil being subjected to phase transformation from a liquid state, showing Newtonian behavior, to the extreme of a gel-like structure exhibiting non-Newtonian characteristics.

When the temperature of crude oil decreases below the Wax Appearance Temperature (WAT), the solubility of the high molecular weight paraffin in the crude oil also decreased. The saturation limit will be reached, causing instability and precipitation of the wax molecules out of the liquid solution. A paraffin deposit will form as the molecules precipitated in the form of solid crystals. At this stage, the viscosity of the crude oil increases due to the presence of the wax crystals and the crude oil behaves as a non-Newtonian fluid [2]. These solid crystals will adhere to the cold surface of the pipeline walls, which is normally at the lowest temperature compared to the core of the pipeline. The radial temperature gradient results in the wax mass flux towards the wall of the pipelines [4]. If no mitigation strategies are taken to increase the pipeline temperature, the paraffin deposits will further thicken over time reducing the effective diameter of the pipeline, throughput and the flow efficiency. Build-up of wax deposits will also increase the pressure drop as well as increase the severity of corrosion and scale as the precipitated wax encourages adhesion of bacteria bio-films and mineral scale [5].

Further wax gelation is induced when the temperature of the crude oil drops below the pour point temperature of the crude beyond which, the crude oil flow ceases completely. These very severe flow restrictions will eventually lead to plugged pipeline, a costly problem in the oil industry. To avoid such problems, it is essential to predict the crude oil flow behavior by characterizing the hydrocarbon fluid. Especially for new fields, proper sampling procedure is required, such that representative sample could be preserved. Sampling could be conducted at the surface or downhole of a well. For exploration wells, most of the samplings are conducted down hole. One of the issues with the bottom hole sampling is drilling mud contamination. If the sample is contaminated, the physical properties of the hydrocarbon fluid may change. The study is intended to analyze the effects of the drilling mud at concentrations ranging from 10 to 20% by wt. on a waxy crude oil at stock tank condition. The waxy crude oil properties such as the viscosity, WAT and yield strength as well as the pour point with and without drilling mud contamination will be analyzed.

EXPERIMENTAl DETAILS

Working fluid

The crude oil utilized throughout the study is a waxy crude oil (dead oil sample) produced in the South East Asia. The samples were extracted from the separator at the corresponding temperature and pressure after the choke and subsequently stored in IATA compliance sample containers. The WAT, as measured using a rheometer, pour point temperature (ASTM 5985-96), API gravity (ASTM D1250) and other properties of the waxy crude oils are tabulated in Table 1. Conditioning of the crude oil upon received involves heating at ~20°C above the WAT for 8 hours in a large water bath. The crude oil is then poured into smaller containers (~20mL) to ensure minimal sample variations due to light and heavy components, and allowed to cool at room temperature.

As thermal history or heat treatment to which the crude oil samples are subjected to influences, especially the flow behavior of the sample, it is important to standardize the pre-conditioning treatment to ensure repeatability of the measurements. Prior to measurements the crude oil in the smaller container is soaked again in a water bath at ~20°C above the WAT for 2 hours to dissolve any wax crystals present ensuring homogeneity and promote a stable composition during testing. The drilling mud sample is supplied by PETRONAS Carigali Sdn Bhd (PCSB) with a density of 1.402 g/ml.

Table 1 Properties of waxy crude oil and drilling mud

Properties	Waxy crude oil	Drilling mud
Density (g/mL)	0.855	1.402
API gravity	33.7	N/A
Pour point (°C)	39.0	
WAT (°C)	39.3	
Yield stress (Pa)	5.79	

Sample preparation

Six samples are prepared for the study with three different drilling mud concentrations; 10%, 15% and 20% w/w, mixed at two different mixing temperatures. Detail information on mass and volume fraction of drilling mud and crude oil, and the mixing temperatures as well as the total mass and volume of the samples are presented in Table 2. S1, S2 and S3 samples are prepared for bottle test and pour point temperature comparisons with that mixed at a higher mixing temperature. Detailed characterization is conducted only on samples S4, S5 and S6 as the mixing temperature is close to that experienced in the field.

Mixing of the crude oil with the drilling mud is conducted under an overhead stirrer at 400 rpm for 60 minutes at 45°C which is roughly 5°C above WAT as well as at 80°C (close to the temperature of the well). Constant speed and time is utilized throughout the mixing process to minimize possible factors affecting the accuracy and repeatability of the experiments.

surface showed that the gelled crude failed within the sample indicating no slip on the upper roughened surface as well as on the smooth lower surface. Hence, the authors believed that an upper roughened surface is sufficient to minimize the slip effect. The waveforms produced at the same % strain for different gap settings have also been analyzed. The similar waveforms produced at different gap settings confirmed minimal slip effects as proposed by [7].

The sample in liquid form is loaded onto the peltier plate which is set to a temperature above the WAT to avoid any 'thermal shock'. Upon loading, the head is lowered gradually to avoid 'spilling and squeezing' out

Table 2 Fraction of drilling mud, crude oil and total mass and volume of the samples

Sample	Drilling mud fraction			Crude oil fraction			Total		Mix. Temp. (°C)
	(% wt.)	(g)	(mL)	(% wt.)	(g)	(mL)	Mass (g)	Vol. (mL)	
S1	10	4.29190	3.06127	90	38.62710	46.93907	42.92	50.00	45
S2	15	6.57945	4.69290	85	37.28355	45.30641	43.86	50.00	
S3	20	8.97020	6.39815	80	35.88080	43.60181	44.85	50.00	
S4	10	4.29190	3.06127	90	38.62710	46.93907	42.92	50.00	80
S5	15	6.57945	4.69290	85	37.28355	45.30641	43.86	50.00	
S6	20	8.97020	6.39815	80	35.88080	43.60181	44.85	50.00	

Equipment and procedures

A TA Instrument controlled stress rheometer, AR-G2 with magnetic bearing (minimal friction), is utilized for all theological measurements, including the WAT and yield stress for samples S6, S7 and S8. The operating torque range of the rheometer is between 0.01 μN.m up to 200 mN.m with 0.1 nN.m of torque resolution. The geometry (parallel plate) is a 40 mm cross hatched plate with the groove roughness higher than 10 μm [6] to minimize wall slip phenomena where complete adhesion is violated. Visual observations are performed to verify any presence of slip on smooth surface geometries. When both smooth upper and lower geometries are utilized, the smooth upper surface practically cleans after what is thought to be an apparent yielding of the sample while the sample remained intact on the smooth lower surface. However, yielded sample using the roughened upper

the sample and hence, minimizing the 'mechanical shock' experienced by the sample on the peltier plate. As the waxy crude oil system is known to be thixotropic, steps have also been taken to 'standardize' the shear history prior to any testing procedure. The sample is pre-sheared at $10s^{-1}$ at a temperature of 50°C, higher than WAT, for 3 minutes and subsequently left for 2 minutes to rest. The viscosity of the samples is measured via a temperature ramp from 50 (C down to 20°C at a shear rate of $10s^{-1}$ and a cooling rate of 1°C/min. WAT is measured under oscillatory condition at a small strain of 0.01% to minimize disturbance to the sample with the temperature ramped from 50°C down to 20°C. A strain sweep test is then performed on a gelled waxy crude oil from 0.01 to 100% at a temperature of 20°C with an angular frequency of 10 rad/s to measure the yield strength.

A gap setting of 800 µm is used throughout all the rheological measurements to provide freedom for the waxy crude crystals to move. A solvent trap is used to minimize evaporation of the light end components from the sample for all the rheological measurements and subsequently the stability of the sample composition is assumed to be maintained. All the sample measurements conducted in this study are repeated at least 3 times to confirm repeatability.

The pour point temperature is measured using a pour point tester PPT 45150 supplied by PSL Systemtechnik. This instrument is a compact set-up with precision up to 0.1°C and complies with the rotational method used by ASTM D5985 [8].

Microscopic observations are conducted on the wax crystal structure of the crude oil with and without the presence of drilling mud with 10 times magnification. The instrument used in this observation is a cross polar microscopy (Olympus BX 53) equipped with a video camera and a recorder. Prior to observations, the samples are brought to a temperature well above the WAT for 30 minutes using a water bath to ensure complete dissolution. The small amount of the sample is then transferred on a slide glass, covered with a thin cover glass and subsequently placed onto a hot stage for controlling cooling and heating. The microscopy observations are conducted from a specified high temperature well above WAT down to the room temperature (24°C) at a cooling rate of 1°C/min.

RESULTS AND DISCUSSIONS

Bottle test study on crude oil mixed with drilling mud

A bottle test observation of the crude oil samples mixed with the drilling mud is conducted to observe the separation behavior due to the different densities. It is observed that all samples formed 3 layers after 24 hours. The drilling mud with the highest density of 1.402 g/ml settles at the bottom of the tube while the crude oil which has the lowest density of 0.83 g/ml stays at the upper layer. In between these two layers is a mixture of crude oil and drilling mud as shown in Figure 1.

Figure 2(a) shows the sedimentation process of drilling mud and the mixture for S1 containing 10% of drilling mud (roughly equivalent to 3.0 ml) mixed at 45°C. The separation of drilling mud from the mixture only occurs after 15 minutes. The amount of drilling mud settling increases until 2 hours of observations where it is maintained constant at 0.8 mL. Even after week 4 of observation, only 0.8 ml of drilling mud separates from the mixture. Figure 2(b) depicts the volume over time of the crude oil, drilling mud and the mixture for S4 containing 10% of drilling mud mixed at 80°C. 0.1 mL of drilling mud sedimentation is observed after 5 minutes and the amount increases and plateaus at 1.0 mL after 4 hours. Similar to the sample mixed at lower temperature, the crude oil separation for the sample mixed at 80°C is only observed after 4 hours. All other samples are observed to separate in a similar trend.

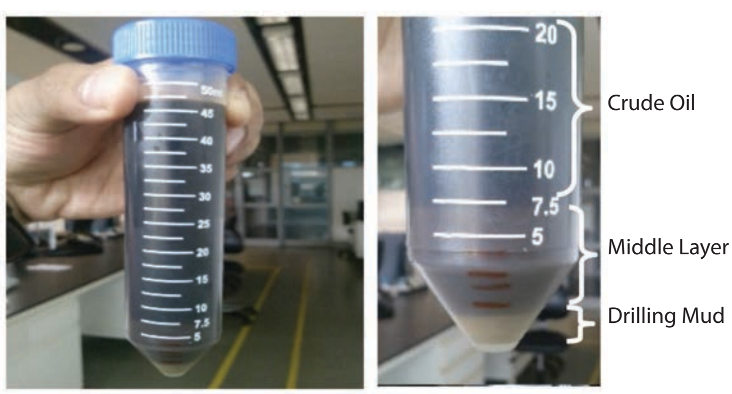
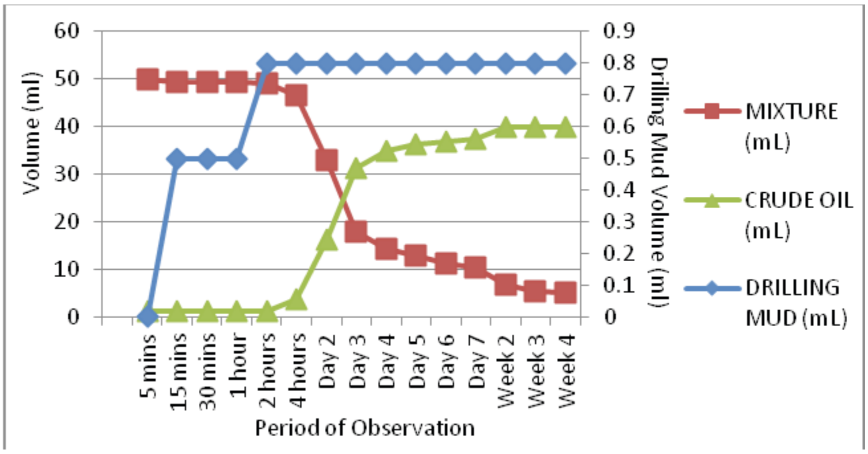
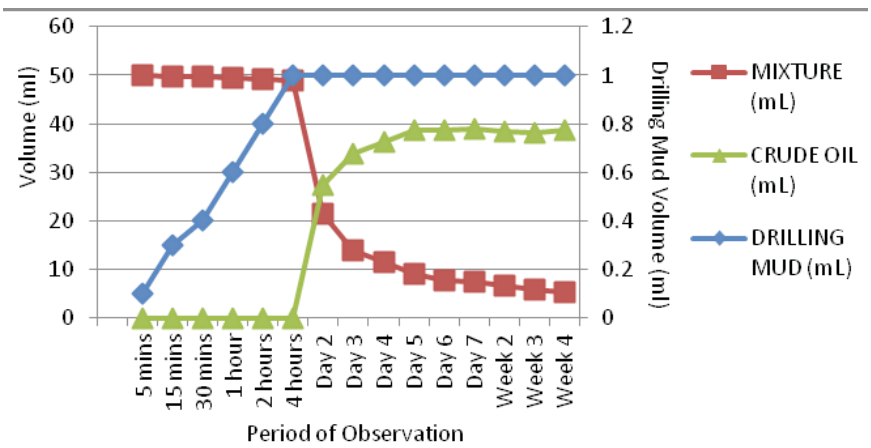


Figure 1 Three layers formed during bottle test



(a)



(b)

Figure 2 Bottle results, a) S1 and b) S4

Pour Point Temperature measurement

Table 3 tabulates the pour point results for all the samples. Generally, there is no significant change in the pour point temperature of the waxy crude oil that can be detected with the addition of drilling mud to the sample compared to that of pure waxy crude oil (as shown in Table 1).

Table 3 Pour point temperature of the samples

Sample No.	Pour Point (°C)
S1	39
S2	39
S3	39
S4	39
S5	39
S6	39

Rheology Test

The viscosity measurements conducted on the samples with and without drilling mud addition mixed at 80°C indicate that the drilling mud increases the viscosity of the crude oil mixture by approximately 10 times (maximum). The viscosity increase, however, is independent of the drilling mud concentration as shown in Figure 3. The observation indicates that the drilling mud has a direct tendency to create a more viscous solution, especially at temperatures near to the WAT and may increase the pressure drop in flowing conditions.

Figure 4 shows the data for WAT determination for S4, S5 and S6. These samples are retrieved immediately after mixing with the drilling mud. It can be seen that at a critical temperature, the elastic modulus G' and the loss modulus, G'' increase significantly. The critical temperature is thought to be that of WAT. Above this temperature, the data show scatter typical of a very low viscosity sample. Within this region, the instrument inertia (raw phase angle is >150°C) is dominant resulting in the data scatter. Similar measurements are also conducted on the middle unresolved layer retrieved from the bottle test after 3 days of separation.

The elastic limit yield stress is determined by conducting the strain sweep step from 0.01 to 100% with a fixed angular frequency of 10 rad/s. A low angular frequency is chosen as tests conducted at an angular frequency of 10Hz (62.8 rad/s) showed that the gelled waxy crude has a very strong consistency with the elastic and loss moduli to be close to a million at low % strain, hence indicating that the selected initial frequency is too high for such a strong material. The stress value at which the oscillation stress deviates from the linear stress-strain relationship, i.e. point of deviation from ideal Hooke's Law, is considered as the elastic limit yield stress following the definition provided by [9] which state that the strong interlocking network in the gelled crude oil behaves more like a solid structure prior to yielding beyond which the Hookean behavior fails. Figure 5 shows the raw data from the strain sweep procedure. The point at the start of the linear deviation is taken as the point of yield. The waveforms retrieved (not shown here) showed deviation from the sinusoidal behavior starting from the point of yield. A sinusoidal waveform would indicate that the sample is exhibiting viscoelastic features while a non-sinusoidal response would indirectly indicate a yielded sample. Similar to the viscosity measurements, the procedures are repeated on the middle unresolved layer retrieved from the bottle test after 3 days of separation.

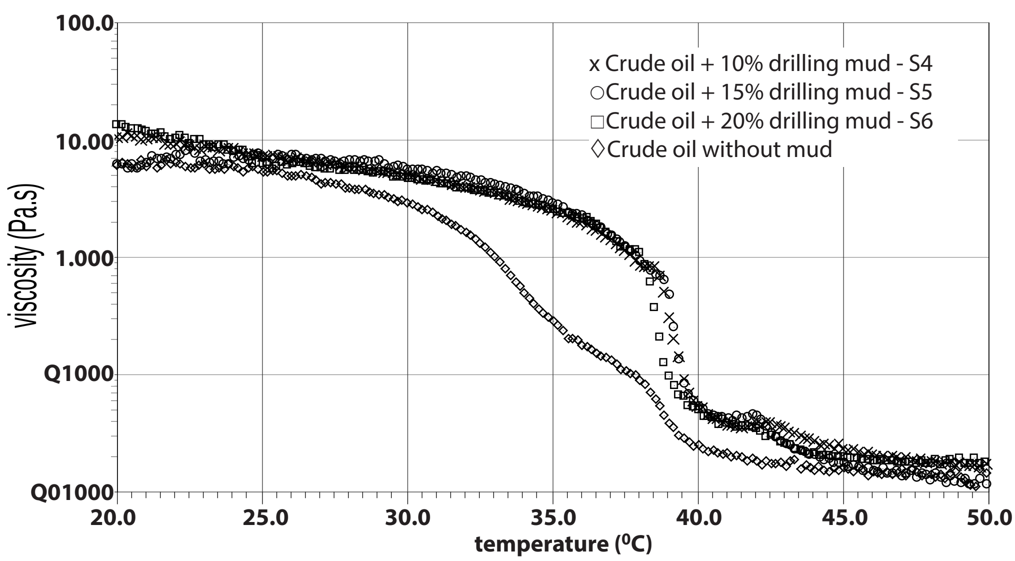
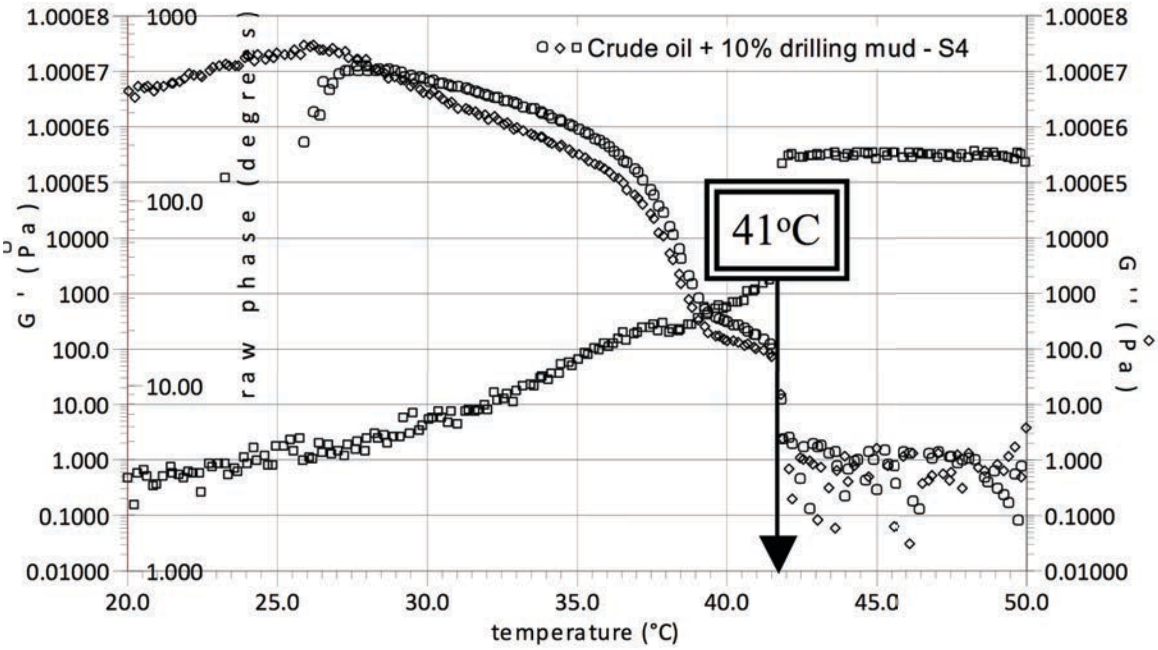
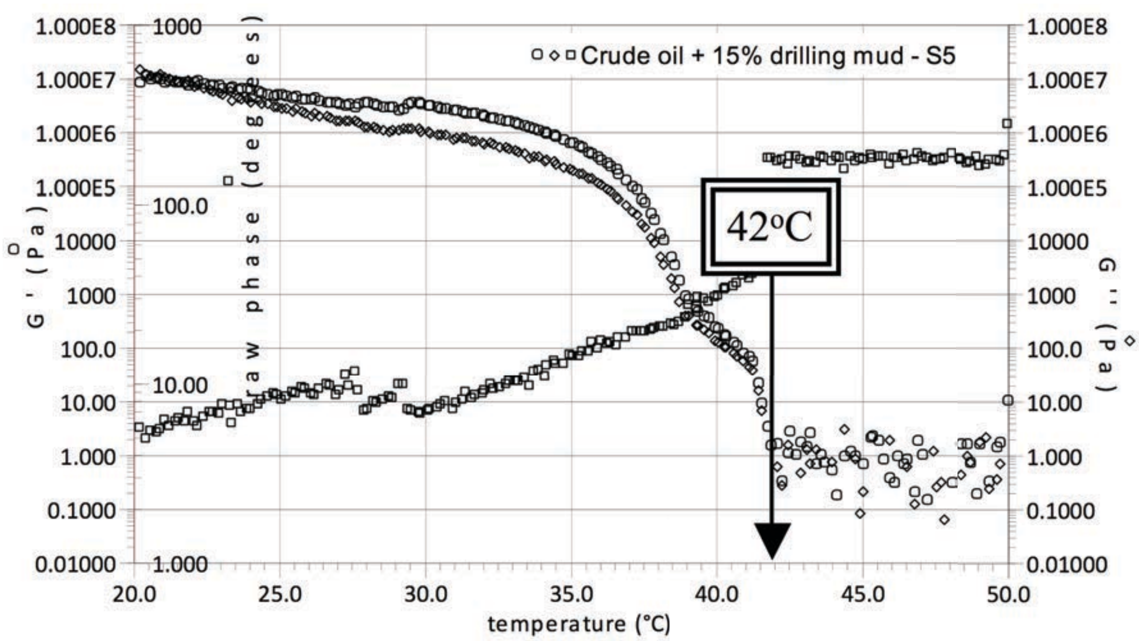


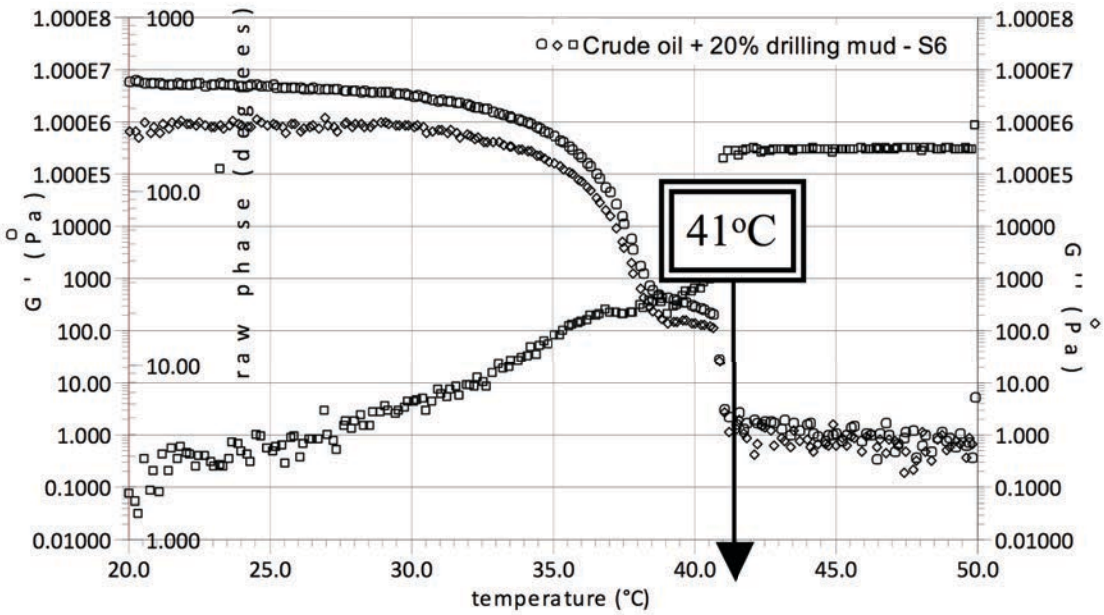
Figure 3 Viscosity data of crude oil samples with and without drilling mud addition


Crude oil + 10% drilling mud - S4
41°C
temperature (°C)
G' (Pa)
raw phase (degrees)
G'' (Pa)

(a)

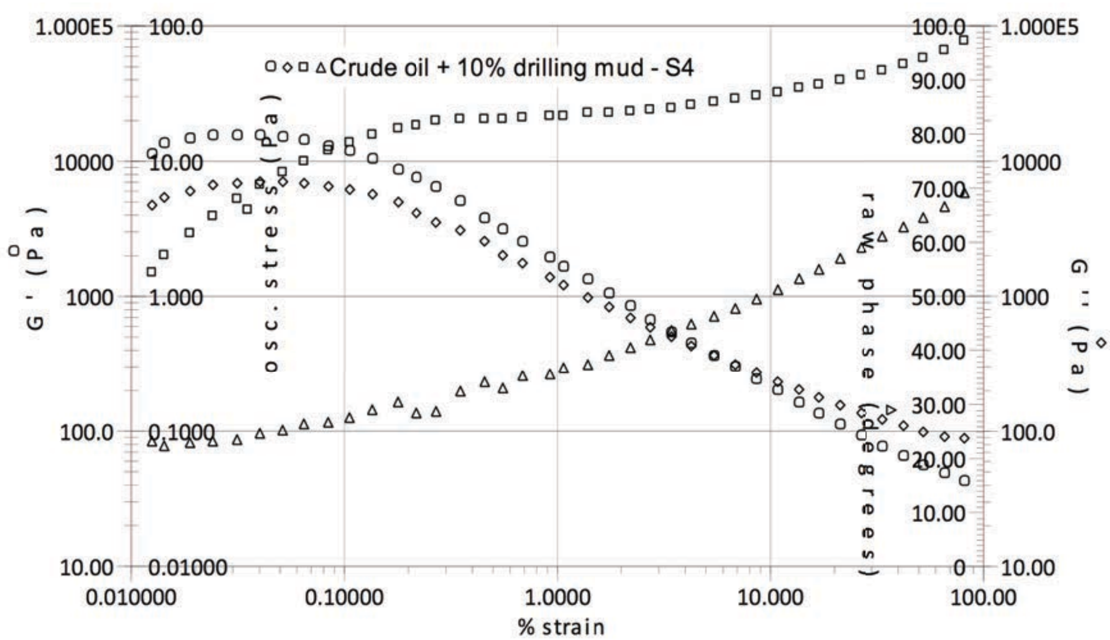

Crude oil + 15% drilling mud - S5
42°C
temperature (°C)
G' (Pa)
raw phase (degrees)
G'' (Pa)

(b)

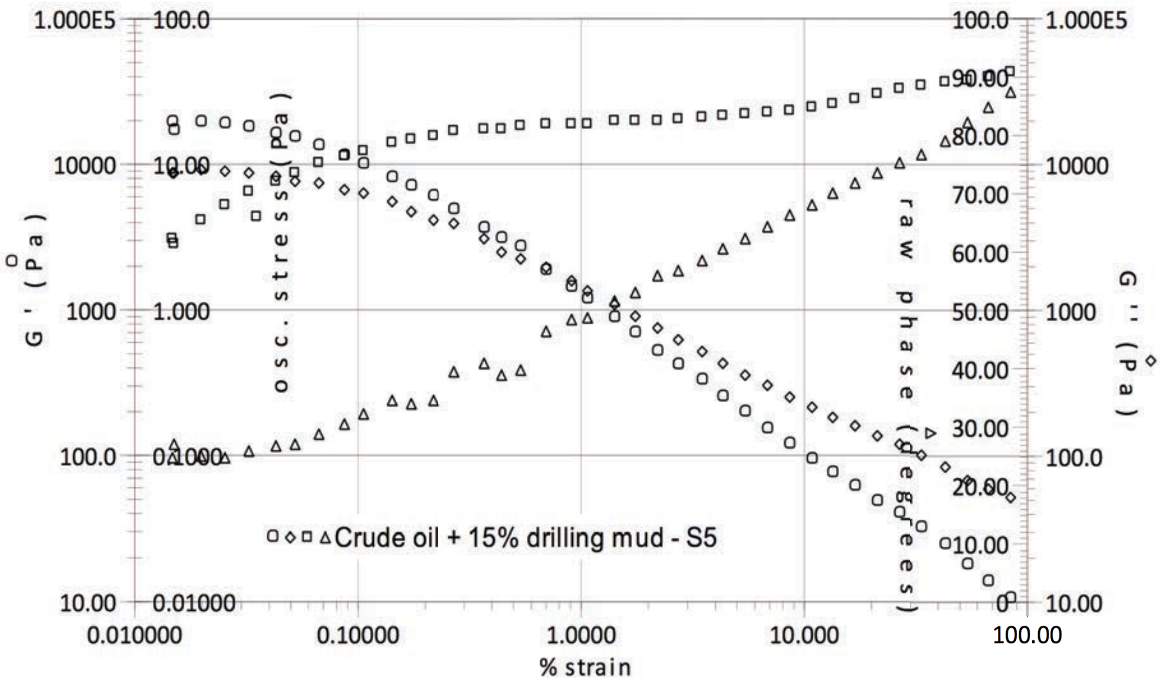


(c)

Figure 4 Temperature dependent behavior of, a) S4, b) S5 and c) S6 under oscillator condition



(a)



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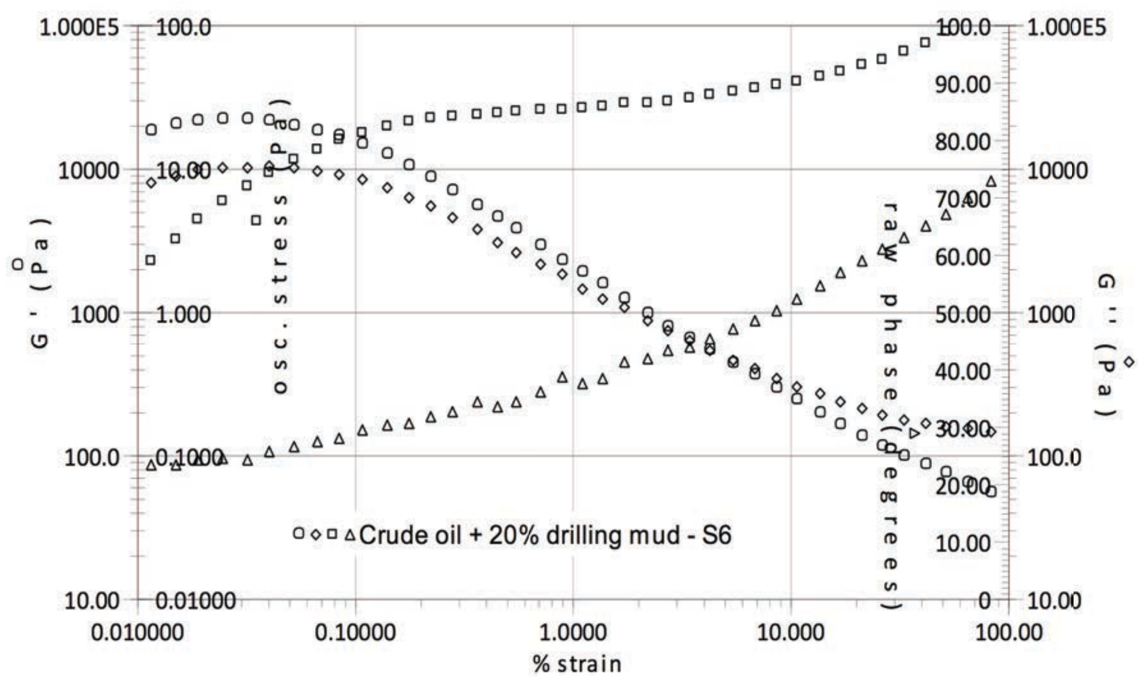

(c)

Figure 5 Strain sweep test results of, a) S4, b) S5 and c) S6 for the determination of elastic limit yield strength

Table 4 summarizes the WAT and yield strength values of both mixture and middle layer of samples from S4, S5 and S6. The mixture represents the samples immediately after mixing (before bottle test) with the drilling mud while the middle layer represents the middle layer after 3 days of bottle test.

Table 4 Result of rheology test for waxy crude oil with different percentage of drilling mud mixed at 80°C

Sample Details	Wax Appearance Temperature (°C)		Yield Stress (Pa)	
	Mixture	Middle Layer	Mixture	Middle Layer
Waxy Crude Oil + 10% DM (S4)	42	42.5	8.0	0.12
Waxy Crude Oil + 15% DM (S5)	42	42.0	10.0	15.0
Waxy Crude Oil + 20% DM (S6)	41	41.2	10.5	30.8

The WAT data shows minimal effects of the drilling mud to the wax appearance temperature of the crude oil. A similar trend is also observed for WAT from cross polarized microscopy observations. The yield strength data, however, show a tendency of the drilling mud to increase the gelling property of the sample and hence exhibiting increased yield strength. Note that the yield stress of crude oil alone is 5.79 Pa. Significant increase in yield stress value is especially observed for the middle layer. The results implied that there may be an interaction between the drilling mud constituents and the crude oil wax crystals, which strengthen the gel network.

Microscopic Observations

Microscopic observations are conducted at a room temperature of 24°C (well below pour point temperature) to observe the wax crystals of the samples mixed with drilling mud and subsequently retrieve the volume fraction of the crystals. The volume fraction is extracted using an image processing code developed in house by performing a 2D image processing on the image collected from cross polar microscope (CPM) using a threshold technique [2]. There are 34 wax samples representing low to high threshold values are first taken from the image and plotted to map the distribution of threshold values. General power law model is utilized to fit the sample data and the fitting is accepted if the R^2-value is equal to or above 0.8. The threshold value can then be deduced from the fit. Based on the threshold value, an algorithm is developed to determine the wax area fraction (volume fraction). The detailed procedure is available in [2].

Figure 6, 7, and 8 shows the microscopic observation of waxy crude oil with 10%, 15% and 20% of drilling mud respectively, mixed at 80°C. The white color region shows the wax crystals within the crude oil. Greater amount of wax crystals is observed at lower drilling mud concentrations with the volume fraction to be decreasing from 0.170, 0.137 and 0.102.

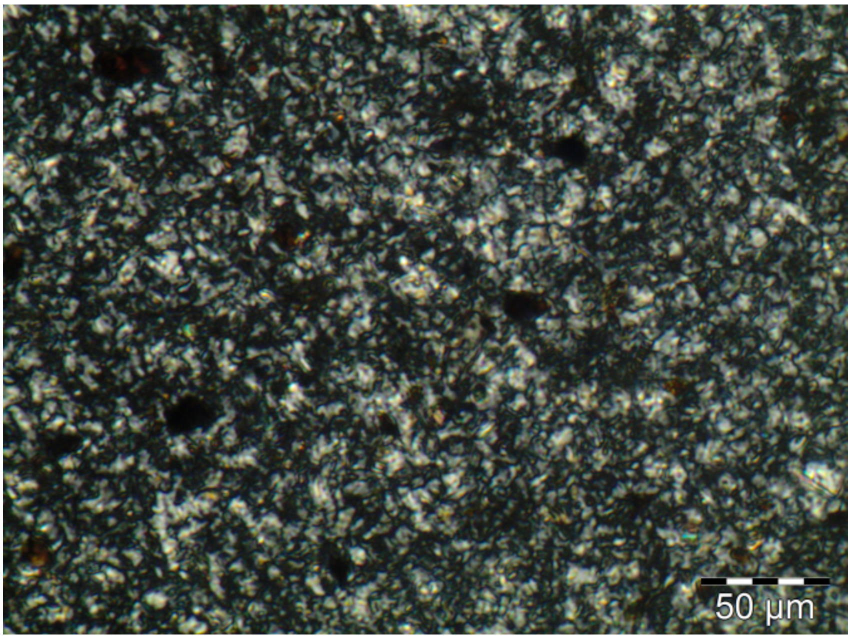

Figure 6 Microscopic observations of waxy crude oil with 10% drilling mud

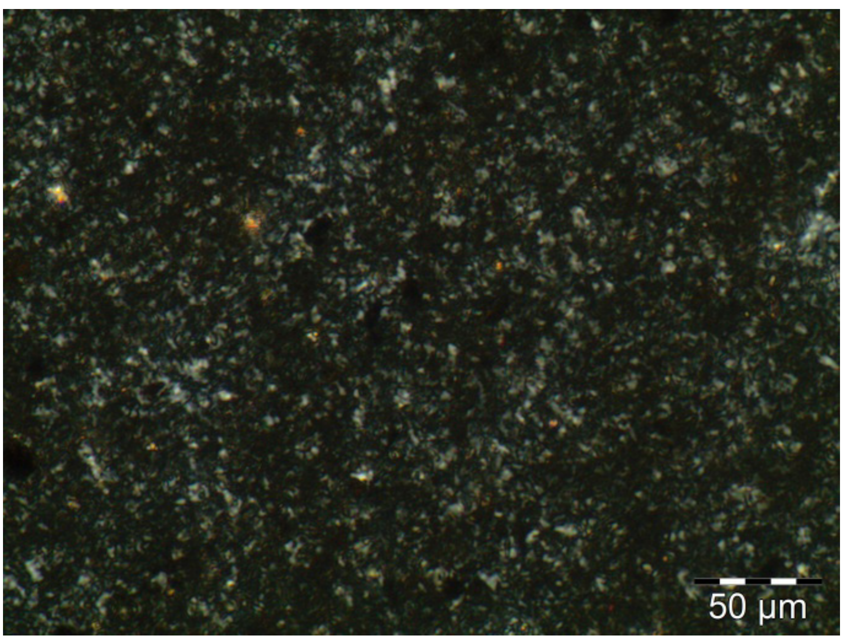

Figure 7 Microscopic observations of waxy crude oil with 15% drilling mud

These observations indirectly indicate that the increasing yield strength of the gelled waxy crude oil observed with the addition of the drilling mud is due to the property of the mud itself and not solely due to the interlocking of the wax crystals forming the gel. The drilling mud is believed to interfere with the crystallization process resulting in a lower number density of the wax crystals. However, the WAT and pour point remains unchanged by these interactions.

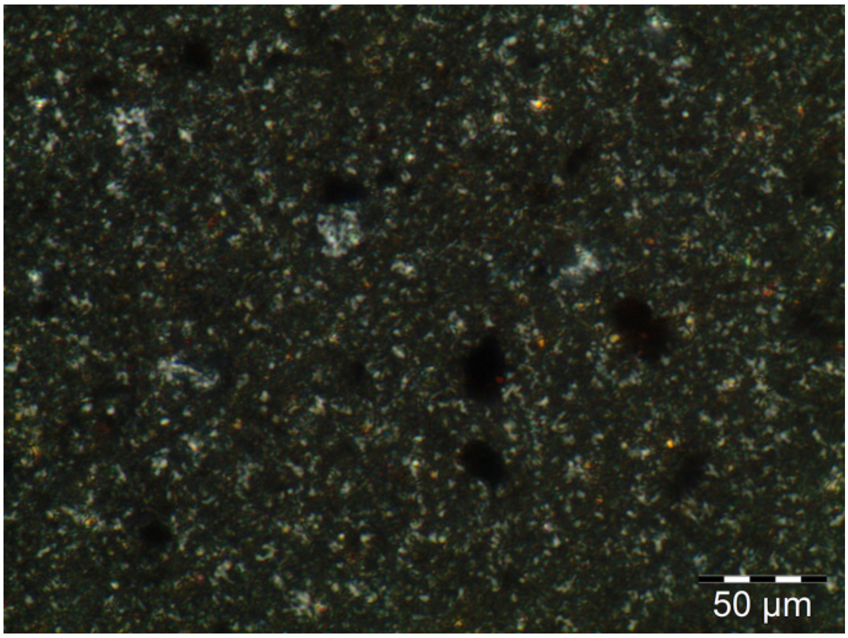

Figure 8 Microscopic observations of waxy crude oil with 20% drilling mud

CONCLUSIONS

Effects of drilling mud contamination on the properties of a waxy crude oil are analyzed. The drilling mud concentration is varied from 10 to 20% by wt. and mixed with the crude oil at two mixing temperatures of 45°C and 80°C. Mixtures show tendency to separate into three layers at different rates depending on the mixing temperature. Drilling mud free oil layer which should be used in characterizing the crude oil exist only after 24 hours of gravity settling. Observations at Day 3 onwards after mixing on all samples indicate that less than 40% of drilling mud settles at the bottom while the remaining is trapped in a mixture with crude oil within the middle layer. The drilling mud contamination shows no effect on the pour point temperature and slightly changes the wax appearance temperature but is still within the experimental uncertainty. The viscosity and the yield strength are significantly increased with mud concentration, especially the middle layer. The results also indicate that if the sample is contaminated with drilling mud, the duration post sampling and the sampling point of the retrieved sample affects the viscosity and the yield stress measured.

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