Depositional Behaviour of Highly Macro-Crystalline Waxy Crude Oil Blended with Polymer Inhibitors in a Pipe with a 45-Degree Bend

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Abstract

Production, transportation and storage of highly waxy crude oil is very challenging. This is because they are usually characterised by high content of macro-crystalline waxes, predominantly consisting of n-alkanes (C_{18} to C_{36}) that which could cause costly deposition within the wellbore and production equipment. The accumulation of deposited wax can decrease oil production rates, cause equipment breakdown, and clog the transport and storage facilities. Currently, different polymeric inhibitors have been utilised in the oil and gas field to mitigate and prevent wax deposition. However, as of today, there is no distinctive wax inhibitor that could work effectively for all oil fields. One of the objectives of this work is to study the efficacy of a blended commercial wax inhibitor - pour point depressant on wax deposition mitigation in a flow rig designed with 0 and 45-degree bends in the pipeline.

Standard laboratory techniques using high-temperature gas chromatography (HTGC), rheometer rig, polarized microscope and elution chromatography were employed to obtain n-paraffin distribution, oil viscosity, WAT, pour point and SARA fractions. Series of experimentation were carried out with and without the inhibitor in a straight pipe test section. The severity of wax deposition in the pipeline built-in with a 45-degree bend is compared with a straight pipe. The blended inhibitor was tested at concentrations of 500, 1000, and 1500-ppm, under laminar and turbulent conditions. The crude oil sample was found to be naturally waxy with wax content of 19.75wt%, n-paraffin distributions ranges from C_{15}-C_{74}, WAT and pour point of 30°C and 25°C respectively. The severity of wax deposition in the test section is 43% higher in 45-degree bend compared to straight pipe. However, the severity of the deposition was reduced to 12.3% at extremely low temperature and flow rate. Nonetheless, better inhibition performance was achieved at 25 and 30°C. The wax thickness was reduced from $\delta_{wax} \approx 0.36$mm at 5 l/min to $\delta_{wax} \approx 0.132$mm at 7 l/min at constant coolant temperature ($25°C$) and 1500-ppm, whereas, no wax deposition was observed at 11 l/min. Mechanisms such as molecular diffusion due to frictional pressure losses, shear dispersion and gravity settling due to momentum change and hydrostatic, alongside with thermal difference are the main drivers for wax deposition in both straight and bend pipe. Whereas, the interaction mechanisms such as the nucleation, alongside with adsorption, co-crystallization, and solubilisation between the new blended inhibitor and the wax crystals provide an improved inhibition performance in the system even at extreme cases.

Keywords: Wax Deposition, 45-Degree bend pipe, Wax Inhibitor, Mechanism

Introduction

Waxy crude oils are characterized as highly viscous and low-degree API gravity fluid composed of macro-crystalline and micro-crystalline aggregates of hydrocarbon molecules (Lee, 2008; Li *et al.*, 2018; Zheng *et al.*, 2016). The macro-crystalline paraffin wax comprises of low molecular weight straight-chain n-alkanes (C_{16} to C_{36}), which crystallizes in needle or platelet shape crystals at low temperature (Lee, 2008; Li *et al.*, 2018) and usually can be found in the production and transportation systems (Al-Safran and Brill, 2017; Hao *et al.*, 2019). Whereas, the micro-crystalline wax consists of high fraction of branches and cyclo-alkanes with a high molecular weight (C_{30} to C_{60}) and usually precipitates in form of needles and mal-crystalline shapes (Kok and Saracoglu, 2000; Mansoori, 2009; Stephan John, 2013; Zheng *et al.*, 2016). Micro-crystalline is mostly found at the bottom of sludge tanks (Al-Safran and Brill, 2017). For many decades, the wax content in the crude oil has been one of the major issues affecting the production, transportation and storage of waxy crude oil. Typically, the precipitation of wax molecules during the transportation process causes an increase in viscosity of the flowing fluid that leads to the increase in pressure drop along the pipe. Therefore, if there is no any prevention or management strategy in place, the precipitated solid could lead to the formation of wax deposit on the surface of the facilities, which could consequently decrease the oil recovery, and increase pumping cost. In the worst-case, the facilities could completely be blocked, causing significantly economic losses that may even lead to abandoning the wells (Adeyemi and Sulaimon, 2012; Matzain *et al.*, 2002; Rashidi *et al.*, 2016; Rittirong *et al.*, 2015; Sanjay *et al.*, 1995; Singh *et al.*, 2000). This particularly occur in a low temperature environment such as the offshore production (Halstensen *et al.*, 2013; Li *et al.*, 2019). Usually, the deposition occurs within the flow lines, surface equipment, topside facilities, downstream refineries and in some of the extreme case occurs in the tubing (Chala *et al.*, 2018; Giacchetta *et al.*, 2018; Guan, 2016; Lee, 2008). Staffia oil field in the UK operated by Lasmo Oil Company is one of the typical fields abandoned due to severe and frequent wax deposition problems. The field recorded over $100,000,000 loss after four years of production (Nguyen *et al.*, 2001; Singh *et al.*, 2000).

Indeed, several wax prevention and remediation methods have been developed and tested in the oil field that include (1) mechanical method, using devices known as pigs, scrapers or cutters (2) thermal method, such as inductive heating, (3) chemical treatment using inhibitors (Al-yaari and Fahd, 2011; Chi *et al.*, 2016; El-Dalatony *et al.*, 2019; Kok and Saracoglu, 2000; Li *et al.*, 2018) and (4) microbial method, which uses microorganisms to produce by-products that act as surfactants or solvents (Etoumi, 2017; Sakthipriya *et al.*, 2017). Other methods tested with limited success are (1) electromagnetic treatments (Balakirev *et al.*, 2007; Coto *et al.*, 2014; Towler and Rebbapragada, 2004), (2) piezoelectric energy (Sulaiman *et al.*, 2011), and (3) vacuum-insulated tubing (Singh *et al.*, 2007). Similarly, Kasumu, (2014) reported cold flow as another emerging technology to control wax deposition. However, according to several researchers (Aiyejina *et al.*, 2011; Coto *et al.*, 2014; Li *et al.*, 2018) these techniques are influenced by efficiency, cost and environmental concerns. Among these methods, pipeline pigging and chemical treatment are conventionally ranked as the top in the field practice for wax remediation and prevention (Aiyejina *et al.*, 2011; Galta, 2014; Li *et al.*, 2019; Zheng *et al.*, 2013). Despite of its effectiveness, pigging can lead to costly operations, particularly in extreme deep production (Wei, 2015). Similarly, Li *et al.* (2019) revealed that the technique depends on the rules of thumb due to the poor understanding of wax removal mechanism (Tolmasquim and Nieckele, 2008). On the other hand, chemical treatment is used as the preferred prevention technique, especially for uninterrupted production, suitable for long-distance pipeline/flow lines (Aiyejina *et al.*, 2011; Majeed *et al.*, 1990) and widely employed to reduce restart pressure and yield stress (Aiyejina *et al.*, 2011; Majeed *et al.*, 1990). The chemicals usually offer an improved rheology properties by disrupting the orderly aggregation of the growing crystals, which decrease the viscosity, pour point and the WAT (Kelland, 2009). Equally, the

use of chemical treatment has practically increased in the petroleum industry over decades, this is because many chemicals having the required wax inhibition properties (Coto *et al.*, 2014) have been discovered.

In reality, none of the techniques mentioned above could be employed as a stand-alone method to provide a hundred percent fit for purpose wax deposition management and remediation (Coto *et al.*, 2014; Fan and Llave, 1996; Li *et al.*, 2018; Yang *et al.*, 2015). In other words, a complete mitigation or prevention of wax is not usually achieved (e.g. using chemical inhibitors only). In some cases, the technique is combined with either heating or pigging method to achieve incredible treatment (Kelland, 2009) and to reduce cost, energy consumption and environmental pollution caused by mechanical and thermal treatments (Mahto and Kumar, 2013; Yang *et al.*, 2015). Unfortunately, the application of chemicals is usually limited to a specific oil well due to the varying properties of the crude oils and origin. This implies that there is no single inhibitor developed to equally be effective in all crude oil well types (Adeyanju and Oyekunle, 2014; Halim *et al.*, 2011; Kang *et al.*, 2014). Consequently, it is typical to discover an inhibitor that is effective in a specific well but less effective or even fail in a different well of the same basin (Coto *et al.*, 2014; Towler and Rebbapragada, 2004). As a result, a chemical is normally designed to fit specific properties of crude oil and environment conditions (Jennings and Breitigam, 2010). It is therefore paramount to appropriately evaluate the performance and the efficacy of chemical inhibitors with different crude oil prior to the applications in the flow loop experiment and oil field operation. This offer the opportunity to make advancement in this area. In this work, four commercial polymeric chemical inhibitors were studied using rheometer and other techniques to understand their performance and influence on waxy crude oil properties. It was found that two of the inhibitors presented a better performance than the others. As a result, a blend of eight inhibitors was created at different fractions. Similarly, blend A produced greater inhibition in comparison with the original products. Therefore, this work aims to study the behavior and performance of the blend A inhibitor on wax deposition, using a pipeline flow rig that incorporates straight and 45° test sections. In previous studies, similar works were carried out in Chi *et al.* (2016) and Coto *et al.* (2014). However, this work investigated the inhibition performance of a new blended inhibitor with a different flow rig design. Additionally, the composition and properties of the sample crude oil was characterized, which is paramount in any field development and prior to experimental studies.

Classification of Chemical Inhibitors
Over many years, different inhibitors have been synthesized to mitigate the deposition problem. F**our** main group of chemical inhibitors reported in the literature include **(i) Wax crystal modifiers, (ii) Solvents, (iii) Dispersants, and (iv) Surfactants** (Adeyanju and Oyekunle, 2018; Coto *et al.*, 2014; Daraboina *et al.*, 2016; Pedersen and Rønningsen, 2003). These chemicals are usually employed to prevent deposition or to mitigate the deposited wax. For instance, the crystal wax modifiers otherwise known as pour point depressants are chemicals with similar molecular structure to the wax molecules (Pedersen and Rønningsen, 2003). Typically, these chemicals co-precipitate or co-crystallize with wax crystals by replacing wax molecules on the crystal lattices; thus, preventing the wax molecules from networking to form a lattice structure that enrich the deposition of the precipitated wax crystals (Kang *et al.*, 2014; Pedersen and Rønningsen, 2003; Woo *et al.*, 1984) as clearly illustrated in Figure 1.
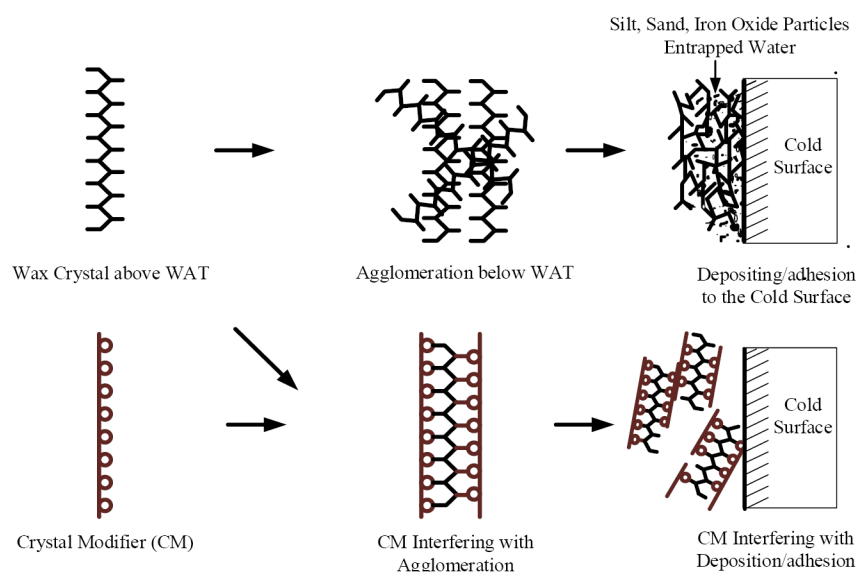
Figure 1—Influence of Inhibitor - Crystal Modifiers on Wax Deposition Process (Adapted from Allen and Roberts, 1978; Sousa *et al.*, 2019).

Some of the wax crystal modifiers currently employed include polyethylene, copolymer esters, ethylene/vinylacetate copolymers, olefin/ester copolymers, ester/vinyl acetate copolymers, polyacrylates, polymethacrylates and alkyl phenol resins (Dobbs, 1999; Kang *et al.*, 2014). Holder and Winkler (1965), Irani and Zajac (2007), Knox *et al.*, (2007), Pedersen and Rønningsen (2003) reported an observation as whether the chemicals cause large, small crystals, and/or aggregate structure. However, the precise way in which these chemicals operate is still under study (Pedersen and Rønningsen, 2003). Interestingly, as of today, polymeric based polymer has been proven to be efficient (Hoffmann and Amundsen, 2013; Perez *et al.*, 2015). Hoffmann and Amundsen (2013) showed that polymer-based inhibitors can successfully reduce about 60%-90% of wax deposit. Nonetheless, their performance differs based on molecular weight, which influenced the agglomeration. For example, shorter or lower molecular weight polymers may cause less disruption to the wax crystal agglomeration and growth, whereas longer and higher molecular weight polymers can interact more with the wax crystals structures. The interaction has great influence on the rate of wax formation, which leads to the formation of softer wax that easily flows along the bulk fluid (Adeyanju and Oyekunle, 2014; Al-Sabagh *et al.*, 2009; Jang *et al.*, 2007).

On the other hand, some solvents can be used as inhibitors to treat solid wax deposit and remediate formation damage. The solvents increase the solubility of wax molecules in the crude oil and dissolve already deposited wax crystals (Kang *et al.*, 2014). The most commonly used solvents as revealed by Kang *et al.* (2014) include xylene, toluene, benzene, carbon tetrachloride, trichloroethylene, perchloroethylene, carbon disulfide, white or unleaded gasoline and pine-derived terpenes. Whereas, the dispersants such as polyesters and amine ethoxylates are usually employed as surface-active agents (Pedersen and Rønningsen, 2003). Normally, dispersants keep the pipe surface water wet, minimizing the tendency of the wax to adhere. These chemicals also help to disperse wax crystals into produce oil, thereby keeping the wax nuclei from agglomerating (Lee, 2008; Nguyen and Fogler, 2005). Nevertheless, the ability to maintain such a surface for an extended period is one of the operational problems with dispersants. Surfactants are used for the cleaning of pipelines and other part of the system where wax may have deposited. Usually, surfactants promote the formation of stable oil and water – emulsion, which is beneficial to pipeline transportation (Nguyen *et al.*, 2001; Singh and Fogler, 1998). Also, similar to dispersants; they could be adsorbed on the pipe surfaces and decrease the adhesion of waxes to the surfaces, and possibly change the wettability of the pipe surface. In other words, surfactants create an environment in which wax crystals are easily sheared off or adsorbed onto the wax crystals surface, which prevent sticking (Nguyen *et al.*, 2001; Singh and Fogler, 1998).

Understanding the severity of wax deposition in pipe curvature is significant in oil field. Experimental study by Makwashi et al. (2019) has shown that at different seabed conditions, the severity of wax deposition is significantly higher in bend pipelines. According to the literature, bends in pipes cause hideous problem in a typical fluid flow; apart from flow restriction, bends in pipe significantly affect the global and local flow parameters such as pressure drop, advection and particle interaction (Wang et al. 2004; Yadav et al. 2014), which directly could affect wax deposition mechanisms. As suggested by Bern et al. (1980), Burger et al. (1981), Majeed et al. (1990), Brown et al. (1993), Singh et al. (1999), Azevedo and Teixeira (2003) and Aiyejina et al. (2011) the mechanisms include; molecular diffusion, Brownian diffusion, shear dispersion, gravity settling and shear stripping. Although, the actual relevance of all the mechanisms and their impact on flow characteristic and dynamics flow behaviour on wax deposition is still under scrutiny. However, studies by Burger et al. (1981), Brown et al. (1993) and Singh et al. (2000) have clearly revealed the existence of molecular diffusion in a straight pipe, where friction is the main dominant force and the Fickian diffusion as the driver for deposition of wax crystals in a laminar sublayer. Whereas the effect of Brownian diffusion, shear dispersion, and gravity settling has been elucidated by Bern et al. (1980), Bott and Gudmundsson (1977), Majeed et al. (1990), Singh et al. (2011) and Venkatesan (2004).

Interaction mechanisms of Chemical Inhibitors with wax crystals
One of the benefits of adding pour point depressant is to reduce the force of interaction among the wax molecules (Goia and Boccaleri, 2016; Li et al., 2018). As of today, the exact interaction mechanism of the wax molecules and the inhibitors such as polymeric pour point depressants are still under scrutiny (Chi et al., 2016). Nevertheless, studies by Chi et al. (2016), Yang et al. (2015) and Li et al. (2018) have revealed some of the mechanisms that include nucleation, adsorption, co-crystallization, and solubilization interactions (Al-yaari and Fahd, 2011; Atta et al., 2011; Pedersen and Rønningsen, 2003; Yang et al., 2014). During the nucleation process, as wax molecules precipitate below the WAT, a crystalline nucleus of a subcritical size is formed, leading to the formation of a larger compound wax crystal in Figure 1. Simultaneously, the high-molecular weight polymeric inhibitor precipitates and acts as crystalline nucleus for the wax crystals (Li et al., 2018). Eventually, this leads to the formation of a more subcritical nuclei of micelle-like aggregates. Subsequently, this inhibits the crystal growth and decreases the supersaturation, resulting in the formation of smaller wax crystals that are stable in the oil phase, implying improved flowability (Hao et al., 2019; Yang et al., 2015). Whereas, once the oil temperature drops below the WAT, the precipitated wax molecules are adsorbed on the polymeric inhibitor nuclei with similar chemical structure, through co-crystallization to modify the morphology of the wax crystal (from long stick-like or large plate-like to small spherical-like), which delays the formation of new crystals (Hao et al., 2019;Li et al., 2018). According to Li et al. (2018) the changes of wax crystals reduce the specific surface area and decrease the surface energy, which make it difficult to form three-dimensional network structure (Wang et al. 2014). Following the adsorption and co-crystallization processes, as the concentration of the inhibitor increases in crude oil, there will be progressive formation of small spherulitic-like crystals in the crude oil system (Hao et al., 2019;Li et al., 2018). The crude oil (liquid) and precipitated wax crystals (the solid) act as disperse and continuous phase along the system. However, when there are no pour point depressants in the system, the solid-liquid interface between wax crystal and liquid phase as well as the energy of the system are large, resulting in an unstable system (Li et al., 2018;Yang et al., 2015). In order to minimize the energy of the system, the solid-liquid interface has to be reduced, by doing that it would cause a union of the wax crystals, as a consequence, larger crystals will form and will create three-dimensional network structure, thus, the addition of pour point depressants improves the dispersion of small wax crystals (Li et al., 2018).

Nonetheless, studies by Li et al. (2018), Manka and Ziegler (2001), and Yang et al. (2015) revealed some key factors that influence the effectiveness of the inhibitors during the interaction between wax molecules and the inhibitors. These factors include composition of crude oil, molecular structure of polymeric wax inhibitors, length of the pendant chains (alkyl side chains), polymer molecular weight, and the carbon

number of alkane chain in the crude oil. Similarly, external conditions are considered as another factor (Kumar et al., 2015;Li et al., 2018). In this work, these factors are carefully considered in elucidating the performance of the inhibitor investigated. As established in literature, WAT and pour point of crude oils are mainly governed by the composition of the hydrocarbons. Basically, the systems of waxy crude oil exists as a complex mixture of crude oil, wax content, and usually, dense organics such as asphaltene and resin. Study by García (2002) have shown that when the number of C in n-paraffins is higher than 24, the paraffin inhibitors become effective. In another study (García,2002), revealed that high concentrations of cyclo +branched alkanes could effectively improve the activity of wax crystal modifier. Also, studies have shown that there is a significant improvement on the performance of wax inhibitors in the presence of asphaltenes (García,2002; Yang et al. (2015). Similarly, polymer molecular weight plays a key role in interacting with wax crystals. According to Manka and Ziegler (2001), polymers with low molecular weight may not have enough molecular volume to disrupt the crystals as it deposits and agglomerates. It may also be the case that if the polymer has very high molecular weight may lead to self-interaction instead of interacting with the paraffin crystals of the crude oil, thus, initiating the paraffin precipitation, raising the pour point of the crude oil as a result.

Experimental Analysis

Crude oil Characterization and blending of Inhibitors. Detailed descriptions and procedure of each standard technique used in the work are reported in the early work by Makwashi et al. (2019). The wax content was determined from HTGC (Singh et al., 2011) with a modified UOP 46-64 method which was slightly modified from Burger et al. (1981). The pour point was determined using a slightly modified ASTM Standard D97-08 and from viscometric plot, similar to the study by Adeyanju and Oyekunle (2013) and Theyab and Diaz (2016). A modified ASTM D2549-02 (elution chromatography) method was used to obtain the Saturates, Aromatics, Resins, and Asphaltene (SARA) fraction and the modified ASTM D2007-80 was used to de-asphalten the sample. A summary of the results of crude oil properties is presented in Table 1.

Table 1—Properties of this crude oil sample and their standard methods of Measurement.

Crude Oil Properties	Unit	Values
Density	g/cc	835
API @ 60°F	°API	35
Wax Content	wt%	19.7
Pour Point	°C	25.5
WAT at 120 1/s	°C	30
WAT at 10 1/s	°C	35
Saturates Fraction	wt%	73.25
Aromatics Fraction	wt%	21.2
Resins Fraction	wt%	5.14
Asphaltene Fraction	wt%	0.41

On the other hand, this work provides more details of the HTGC study. The analysis was done using a proprietary commercial method known as T-SEP® "thermal separation technique". The technique is co-developed by the Kernow Analytical Technology Laboratory and the University of Plymouth. An Agilent 6890 gas chromatograph equipped with flame ionization detector (FID) and a Varian Vf–5ht Ultimetal column (15m × 0.25mm × 0.1μm) was used. The oven is programmed to run from 40 to 435 °C at 10 °C/min ramp before holding at 435°C for 10 min. Helium was used as carrier gas at a flow rate of 5 mL/ min. Initially, the chromatograph of the solvent blank (cyclohexane), compound in C_{10}-C_{60} standard and

Polywax 655 external standard are workout. A 'Whole' and 'Topped' oil samples were prepared, in which the n-Paraffin distribution and wax content. This method is very reliable, reproducible and effective than the simple conventional techniques employed by most laboratories. Figure 2 shows the Chromatograms of the whole and toppe oil samples.
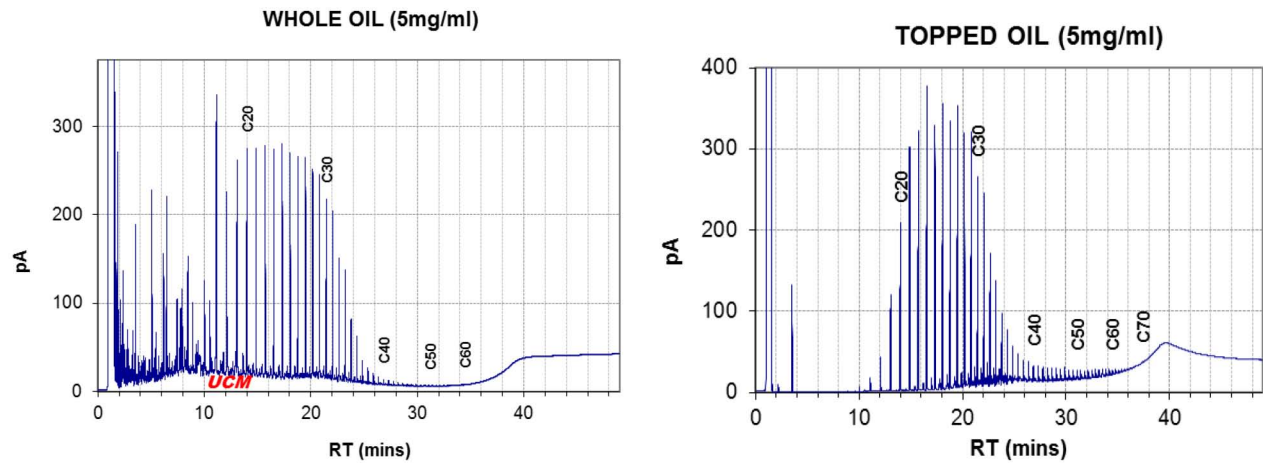

Figure 2—HTGC Chromatograms of Whole oil and Topped Oil Sample obtained from T-SEP® technique.

Topping the sample allows all unresolve complex mixture (UCM) found in the whole oil chromatograms to be removed. Similarly, as shown above, the peaks of n-alkanes fractions above C_{30+} are more pronounce and the peaks around C_{15}-C_{29} are intensified in the topped sample. This means that in the whole oil the n-alkanes fraction above C_{30} are considered to be below the detection limit of HTGC (KAT, 2009). Additionally, the n-alkanes fraction below $<nC_{15}$ are eluted in the topped oil, as these fractions do not contribute to wax deposition process. Therefore, based on this technique (KAT, 2009), the data of 'whole' and 'topped' oil were merges in order to produce a new "adjustable value" which represents the actual n-paraffin distribution in the oil sample. The data for these analyses is formulated in Table A.

On the other hand, the compositions of four commercial chemical inhibitors supplied by Roemex Oilfield Service Company are shown in Table 2. As previously mentioned, out of these samples, W2001 produces better performance, followed by W2003 (see Figure 6). Similarly, eight samples were prepared by blending 70, and 50% of W2001 chemicals with 30 and 50% of W2003, W2004 and W2005. The mixing criteria was based on the inhibitor's performance and composition. From this new samples, blend A (50% W2005 and 50% W2003) produces an improved inhibition performance compared to the original inhibitors (Figure 7).

Table 2—The chemistry of chemical inhibitors used in this study.

Inhibitors	Inhibitors Chemistry		
	Compositions	Percentage %	CAS
W2001	Hydrocarbons, C_{10}, Aromatics, >1% Naphthalene (HAN)	30% – 60%	n/a
	Olefinic polymer derivative	30% – 60%	1269781-05-6
W2003	Solvent naphtha (Petroleum), Heavy Aromatic	7% – 13%	64742-94-5
	Distillates (Petroleum), Hydrotreated light	7% – 13%	64742-47-8
	Naphthalene	1% – 3%	91-20-3
W2004	Alkenes, C_{20} -C_{24} Alpha-, Polymers with maleic anhydride, C_{18}-C_{22} alkyl esters	60% – 100%	n/a
	Solvent naphtha (Petroleum), Heavy Aromatic	7% – 13%	64742-94-5
	Distillates (Petroleum), Hydrotreated light	7% – 13%	64742-47-8
	Naphthalene	1% – 3%	91-20-3

Inhibitors	Inhibitors Chemistry		
	Compositions	Percentage %	CAS
W2005	Solvent naphtha (Petroleum), Heavy Aromatic	7% – 13%	64742-94-5
	Distillates (Petroleum), Hydrotreated light	7% – 13%	64742-47-8

Experimental Flow Rig and Test Configurations

The details of the experimental flow rig design including the parameters of straight and bend pipeline test section, velocity trend profile along the longitudinal cross-section around the straight and 45^0 bend, and the capacity of the flow rig are reported in a study by Makwashi et al. (2019). The flow rig was designed based on the work by Adeyanju and Oyekunle (2013) and Theyab and Diaz (2016) for the straight pipe, whereas, studies by Xing et al. (2013) and Rashidi et al. (2016) were used to design the bend pipe test section with modifications. The straight pipe test section is jacketed in a copper pipe material, while, the bend pipe is installed in an open polypropylene feed and expansion cistern tank (40 gallons capacity).
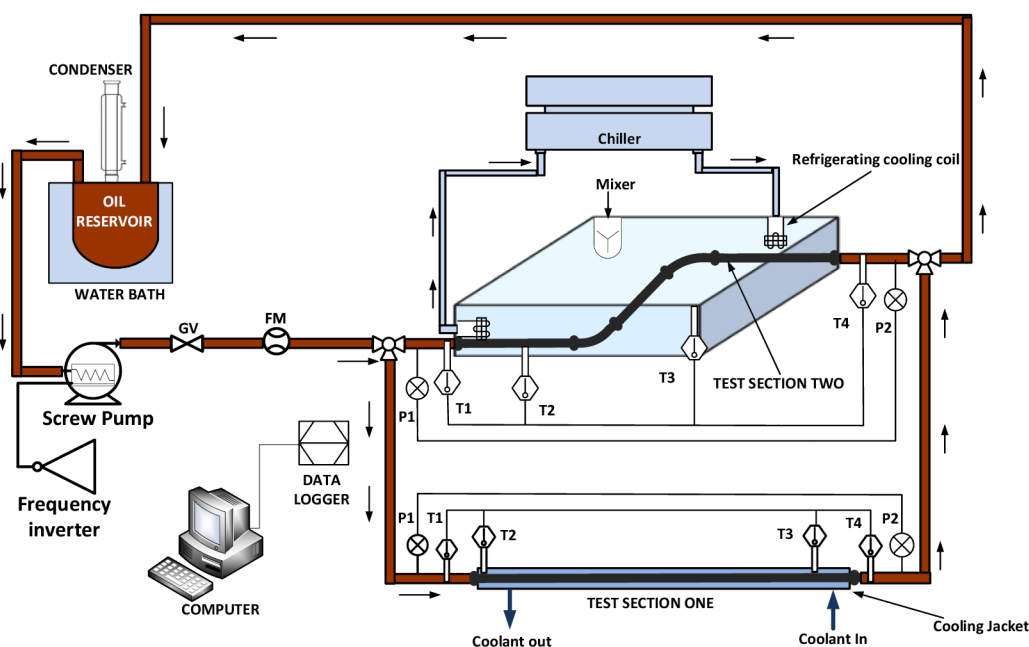

Figure 3—Wax deposition Flow Rig Facility designed with Straight and 45O Bend Pipeline. Where;
FM: flow meter, P1 and P2: inlet and outlet pressure of the test section, T1 and T4: inlet and
outlet oil temperature, T3 and T2: the inlet and outlet coolant (water-glycol mixture) temperature.

The experiment was conducted in a single-phase flow. The straight pipe test-section is horizontally positioned throughout the experiment, whereas, the 45° bend pipeline test section was studied in horizontal and inclined positions. Further details, such as running the test sections, removal for sampling the wax deposit can be found in Makwashi et al. (2019).

Initially, a based case experiment was carried out with pure waxy crude oil according to the test matrix in Makwashi et al. (2019). The crude oil entered the test section at the temperature above WAT (45°C). The varied parameters included cooling temperatures (15, 20, 25, 30 and 35°C) and flow rates (5, 7, 9 and 11 L/m). The testing procedure detailed in Makwashi et al. (2019) consists of Start-up (without cooling), Wax deposition testing and Deposit thickness analysis. The concentrations of the inhibitor used for the straight pipe tests were 500, 1000, 1300, 1500 and 1800-ppm. This provides an opportunity to determine the optimal concentration that gives higher inhibition efficiency. However, in flow rig with 45° bend the concentration used was 500, 1000 and 1500-ppm. In both tests, weight correlation method was used to determine the

thickness of deposit wax by accurately measuring the deposit in the test section. According to previous studies such as Hoffmann and Amundsen (2010); Panacharoensawad and Sarica (2013); Theyab and Diaz (2016), the pressure drop technique, one of the most reliable methods for wax thickness quantification in single-phase flow, produces approximately the same results with the weight correlation method. Similarly, the work by Makwashi et al. (2019) establishes the same behaviour.

$$\delta_w = R - \sqrt{R^2 - \frac{M_{dep}}{L\,\pi\rho_{dep}}} - \delta_0 \tag{1}$$

Where: δ_w is the average thickness of the wax deposited, δ_0 is the residual oil in the test section, R and L are the inner (empty) pipe radius and the length of the removable test section respectively. While, ρ_{dep} is the density of the wax and M_{dep} mass of the deposited wax in grams.

Furthermore, during the deposition study, as the deposit mass and wax content are considerably affected by the added amount of inhibitor, it is necessary to take both parameters into account to evaluate the inhibition efficiency at the different conditions (Bello et al., 2006; Chi et al., 2017; Wang et al., 2003). Therefore, in this study, wax inhibition efficiency (WIE) was evaluated using Eq. 2 shown below:

$$\text{WIE}\,(\%) = \frac{W_f - W_t}{W_f} \times 100 \tag{2}$$

Where W_f is the amount of wax deposit without inhibitor (in g) and W_t is the amount of wax deposit with inhibitor treatment at different concentrations.

Results and Discussion

Composition and Properties of Crude Oil Sample
Characterization of wax crude oil is one of the crucial stages prior to the production in any oil field. The data obtained play a significant role in handling wax formation and deposition and predicting its severity for strategic prevention, management and control during production, transport, refining, and storage (El-Dalatony et al., 2019). Composition and properties such as wax content, n-paraffin distribution, SARA fraction and even morphology of the crude are critical parameters in the design of the oil field production plan. Globally, many oil fields experience considerable amounts of wax content (2~44%) (Holder and Winkler, 1965; Kasumu, 2014; Rehan et al., 2016). High temperature gas chromatography (HTGC) and UOP methods were used in this work to determine wax content and n-paraffin (macro and micro-crystalline) wax. Both methods produced roughly the same result of wax content of about 19.7 and 19.4 wt%. These results suggested that the crude oil has wax content that can cause serious depositional problem. Studies have shown that crude oil containing as little as 2% by mass of wax can potentially cause deposition problem in the flow lines, pipeline, and separators at suitable conditions that favor the process (Holder and Winkler, 1965; Kasumu, 2014). The wax content of a crude oil has a linear relationship with the aromatic and saturate fractions of the sample. It is revealed that the higher wax content of crude oil is directly related to the higher saturate and lower aromatic fraction of the sample and vice-versa (Ekaputra et al., 2014). As observed from Table 1, the saturate and aromatic fractions of 73.25 and 21.2wt% further proved that the crude oil is highly natural waxy crude.

On the other hand, through the HTGC analysis, a wide range of n-paraffin distribution between C_{15} to C_{67} is discovered (Table A, appendix). According to França et al. (2018), molecules from C_7 to >C_{100} could easily be detected from typical HTGC analysis. The wax (n-paraffin) in the crude oil sample falls into macro and micro—crystalline as shown in Figure 4. The distribution was generated based on the integration of chromatograms of the "Whole and Topped" oil sample using T-SEP® technique. As reported by França et al. (2018) "Macro- and micro-crystalline waxes have different functional properties (including viscosity and melting point) due to the differences in their hydrocarbon content (linear or branched)". Accordingly, as

stated in the literature, micro-crystalline wax is smaller and thinner than that of macro-crystalline waxes and usually crystallizes at a higher temperature compared to macro-components where stronger gels are formed. As shown in Figure 4, the crude oil sample used in this work have high concentration of *n*-paraffin (i.e. the macro-crystalline component), which implies possible depositional problem. The deposition of macro-crystalline wax usually occurs in the production and transportation systems, unlike the former (i.e. micro-crystalline wax) that commonly deposits during refining process and at the bottom of sludges tanks. It is observed that from the distribution n-paraffin composition, there was a proportion of macro-crystalline wax fraction from C_{15} to C_{27}. Subsequently, the n-paraffin gradually became rich in higher molecular carbon component of higher molecular weight with decreasing fraction. This is in accordance with other research findings (García *et al.*, 2000; El-Dalatony *et al.*, 2019; Japper-Jaafar *et al.*, 2016).
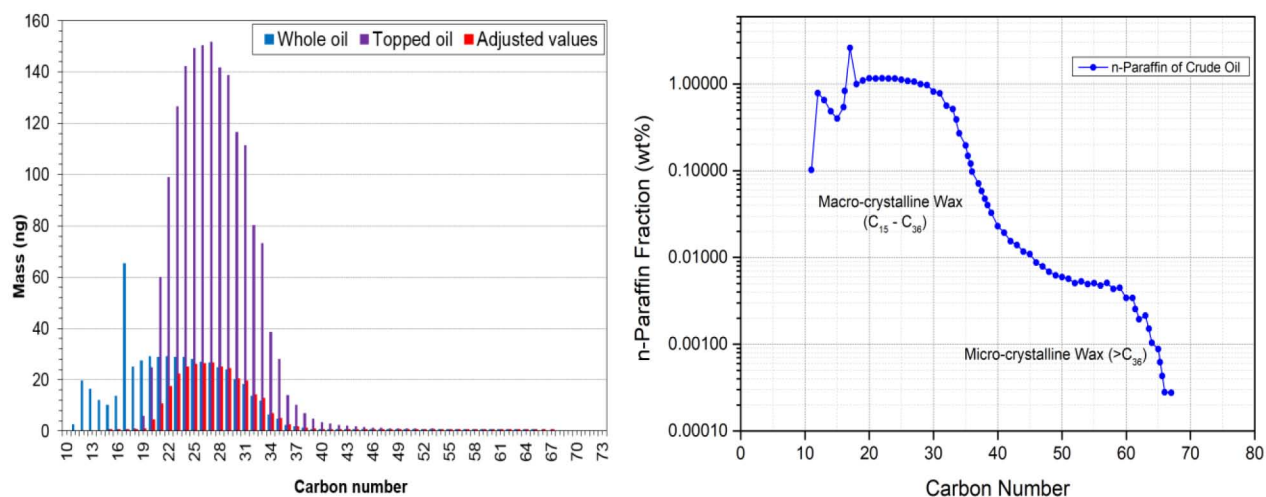

Figure 4—Left: Distribution of n-Paraffin. Left: Merged whole and topped oil with adjustable value as the actual distribution of the sample. Right: The actual n-Paraffin distribution of the crude oil.

Overall, in this study, the macro-crystalline wax (C_{15}-C_{36}) in the sample varies between 0.196-1.161wt%, with exception of a unique peak around C_{17} (2.31wt%). From the normal GC-MS analysis (not included in this work), it is found that some fraction of pristane ($C_{19}H_{40}$) colludes nC_{17}, particularly as their boiling point, is approximately the same, which give rise to such behaviour. The inability to resolve these two compounds using HTGC (T-Sep technique) is a limitation of this method. However, this will not affect the accuracy of the results, in fact, the component of nC_{17} exhibits considerably less effect on wax deposition problem compared to other heavier components. On the average, each fraction macro-crystalline wax (C_{15}-C_{36}) is around 0.92wt%. Compared to micro-crystalline wax (C_{30}-C_{60}) that exists in small quantity, the fraction varies from 0.0003-0.097, and on average, each fraction is 0.014wt%.

On the other hand, the microscopy observation of the crude oil is in agreement with previous studies by Coto *et al.* (2014), Lee *et al.* (2008), Li *et al.* (2018), Yang *et al.* (2014), where the pure crude oil (without inhibitor) displays a particular shape or structure that are considered to be needle-like or rod-like particles (Figure 5, microgram A). However, as observed in the Figure 5 (microgram A*), the presence of blend A wax inhibitor affects the structure of the wax crystals, which is transformed from needle-like or rod-like to agglomerate to small particles dispersed in the oil matrix. Hence, the rheological properties are improved due to morphological modification of wax crystals by doping with 1000-ppm of blend A inhibitor. Therefore, in this case, the inhibitors disrupt the strong interactions of the wax crystals around the microsphere through steric hindrance effect.
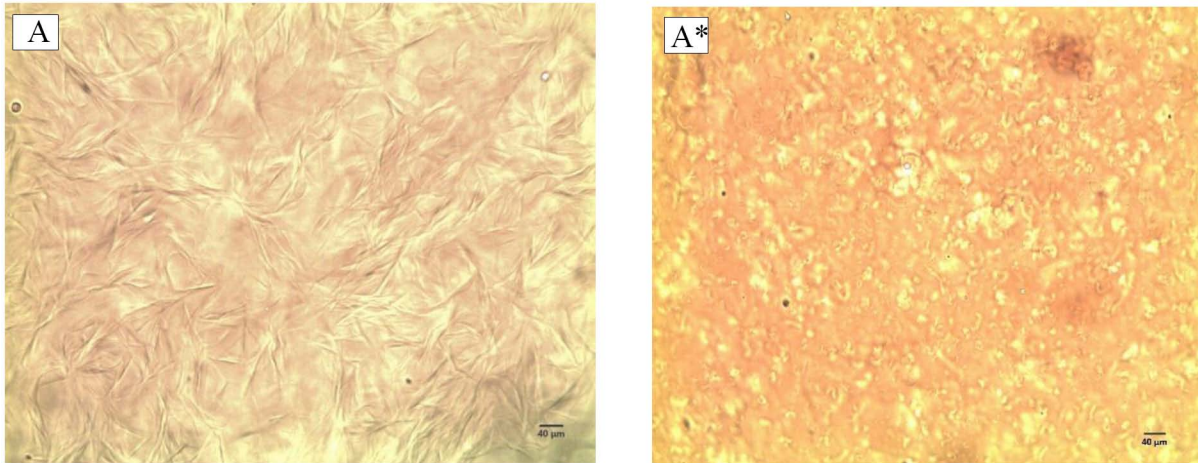
Figure 5—Effect of adding Blend A inhibitors in waxy crude oils. (A) Wax Crystals morphology with undoped (A*) Changing Wax Crystals Morphology with 1000-ppm Doped crude oil.

Effect of blended wax inhibitors on Deposition

Prior to the study in the flow rig, the rheology of the oil sample was obtained with the mixture of crude oil and inhibitors at 0, 500, 1000 and 1500-ppm. The details of the undoped oil (i.e. at 0-ppm) can be found in the study reported by Makwashi *et al.* (2019). It is established that the rheological properties of crude oil were strongly affected by the W2001, W2003, W2004 and W2005 inhibitors at 1500-ppm, compared to the lower concentrations (such as 1000 and 500-ppm). However, apart from the concentration, the performance of these inhibitors is influenced by its composition. Similar behaviour are reported by Kuzmić *et al.* (2008), Hoffmann and Amundsen (2013), Pedersen and Rønningsen (2003). In this study, it was found that wax inhibitor W2001 provided the best performance in reducing viscosity (Figure 6), pour point and WAT of the crude oil. The inhibitors performance decreases in the order of W2001>W2003>W2005>W2004.

For example, during the control cooling experiment using rheometer, at 5°C and 120 1/s, the viscosity of the pure crude oil reduced from 5 to 3 Pa. s at 500 ppm concentration for W2004 and W2005 inhibitors. The viscosity further reduced to 1.76 and 1.5 Pa. s for sample W2001 and W2003 at the same concentration. Generally, as seen in Figure 6, the inhibitor W2001 has demonstrated better performance. Similar behaviour are observed with other parameters measured such as WAT and PP.
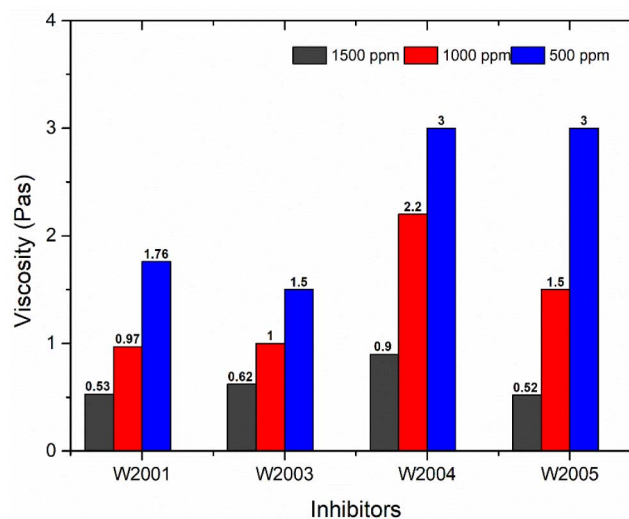

Figure 6—Effect of chemical inhibitors on viscosity of crude oil at 5°C and shear rate of 120 1/s.

As mentioned in this work, the synergy of the four commercial inhibitors was initially investigated. In the Figure below, the eight prepared inhibitors show great inhibition performance, however, it was observed that at 1500-ppm concentration, blend A provides much better performance. Hence, it was noticed that the viscosity of crude oil further reduces from 5 to 0.33 Pa. s at 5°C at the above condition.
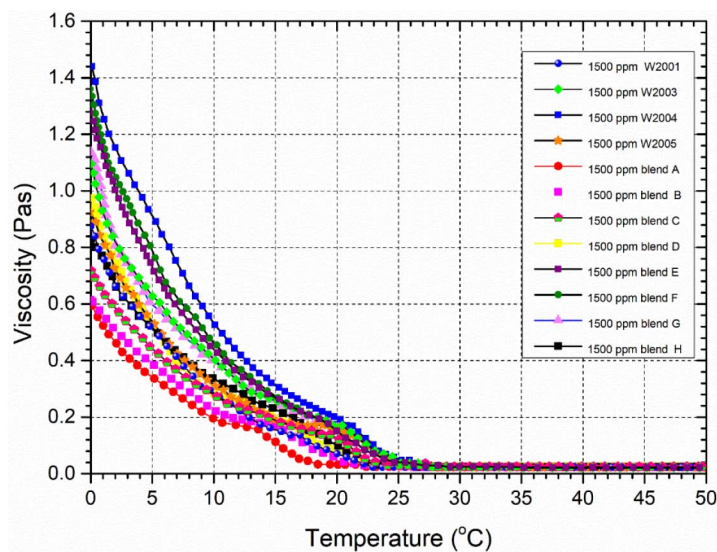

Figure 7—Effect of blend A inhibitor compared to other inhibitors at different temperature and constant shear rate of 120 1/s.

Effect of blend A Inhibitor on the Wax Thickness and Deposit Mass

In the course of flow rig study, only blend A inhibitor was used to mitigate wax deposition in terms of deposit thickness, mass and volume. Based on the work by Makwashi et al. (2019), the severity of wax deposition in 45° bend is 14% higher in laminar flow regime at lower flower rate (5 l/min) and at the temperature of 15°C, compared to the straight pipe test section at the same conditions. The severity is yet substantial even as the flowrate increases in a turbulent flow region (11 l/min) at the same temperature. The study revealed that around 8% more wax would be inherited as changing from straight to pipeline with 45° bend. Similarly, an increase in wax deposition was observed at the test section when the bend is introduced in the pipeline switching from horizontal to an inclined flow (Makwashi et al., 2019). As stated in the literature, higher bend radius in pipe encourages flow separation. The fluids immediately separated after the bend. Consequently, horizontal flow with bend may result in more dominated molecular diffusion within the region and shear dispersion mechanisms at lower temperature (Burger et al., 1981; Kok and Saracoglu, 2000). Unlike in a straight pipe, where pressure drop due to the frictional forces dominates the system. Burger et al. (1981), Brown et al. (1993) and Singh et al. (2000) have shown that molecular diffusion is the only main mechanism responsible for the deposition. In an inclined flow with in a pipeline with bends, it is reported that the increase in deposition may result in additional gravitational settling effect, as a consequence of the effect of bend parameters and especially at lower temperature and laminar flow conditions (Makwashi et al., 2019). In contrast to the study by Burger et al. (1981), no change in the deposition rate was observed when a straight pipe test section was switched from horizontal to vertical flow.

Through this study, an optimal concentration was determined in a straight pipe by doping the crude oil with 500, 1000, 1300, 1500, and 1800-ppm. As mentioned in the previous section, parameters such as the inhibitor concentration, oil flow rate and cooling rate affect the inhibition performance. Figure 8 illustrates such behaviour in a straight pipe. As clearly demonstrated in the figure, the performance of the inhibitor was tested and the results are illustrated in three different graphs (at constant cooling temperature; 15, 25, and 30°C), each at four different flow rates; at 5 and 7 l/min (within laminar flow regime) and 9 and 11 l/min (turbulent flow regime). The three cooling temperatures (1) 15°C, considered as one of the

extreme cases (below the pour point and WAT), (2) 25°C, around the pour point and (3) 30°C, at wax appearance temperature. However, the analysis above the WAT is detailed in study by Makwashi et al. (2019). From the Figure below, at 5°C cooling temperature, it can be seen that at lower concentration (500-ppm) and 5 l/min, the wax deposit thickness decreases substantially from $\delta_{wax} \approx 1.93$ mm for crude oil without inhibitor to $\delta_{wax} \approx 1.19mm$ at 500-ppm. Further decrease in the deposit thickness was observed as the concentration of the inhibitor increases at the same conditions, i.e. more wax is reduced from the system. At 1500-ppm the thickness further decreases to $\delta_{wax} \approx 0.73$ mm. Therefore, without the inhibitor, it is found that 30.8% of the oil will deposit in the pipeline, however, by doping the crude with 1500-ppm inhibitor only 11% of the oil could be deposited at these extreme conditions of temperature and flow rate. This could be due to the adsorption and co-crystallization processes of the inhibitor, which progressively drives the formation of small (spherulitic-like crystals) in the system, as a result of increasing the concentration of inhibitor in crude oil (Li et al., 201; Hao et al., 2019). Furthermore, at higher concentration, the inhibition performance becomes increasingly better around the turbulent flow regime due to the shearing effect (Bern et al., 1980; Makwashi et al., 2019). Therefore, more reduction of wax deposit thickness was observed as the flow increases from the laminar flow regime to turbulent flow regime at the same cooling temperature and concentration.
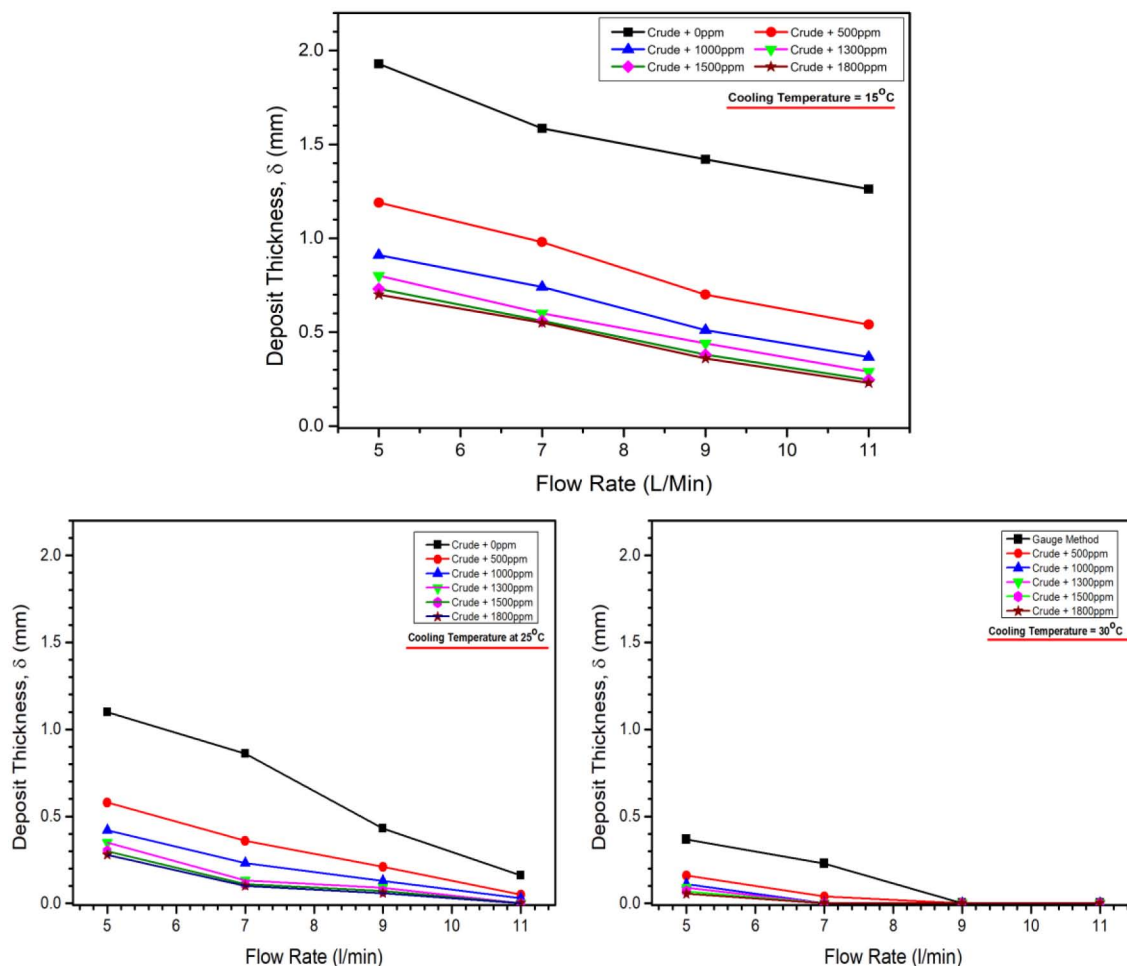

Figure 8—Influence of Blended Crystal Modifier on Wax Deposit Thickness at 15°C Cooling Temperature – Straight Pipeline Test Section.

As the cooling temperatures increases to 25 and 30°C (as shown above), the observed wax deposition with both pure crude and mixture of crude with inhibitor decreases rapidly especially at WAT (30°C). At

this temperature it is found that there is zero deposition particularly at turbulent flow regime i.e. at 9 and 11 l/min, $\delta_{wax} \approx 0\ mm$. Similarly, even at lower flow rate (5 l/min) the observed deposition without inhibitor at higher temperature (30°C) is very small ($\delta_{wax} \approx 0.36\ mm$). Whereas, the corresponding deposit thickness with inhibitor at 500 and 1500-ppm is $\delta_{wax} \approx 0.16\ mm$ and 0.07 mm respectively. According to study by Bern *et al.* (1980), at higher temperatures and high heat flux conditions, molecular diffusion is expected to dominate the deposition process, however, at lower temperatures and low heat fluxes shear dispersion is the dominant mechanism. One interesting phenomenon observed from these results is that the inhibitor concentration at 1500 and 1800-ppm produced approximately the same inhibition performance in both conditions. This implies that under these experimental settings the optimal concentration that produces higher inhibition performance in the flow rig is 1500-ppm. However, at 1300-ppm a similar inhibition behaviour to 1500-ppm was observed, but based on the percentage efficiency, the later (1500-ppm) concentration is still better as optimum value (as shown in the Figure below).
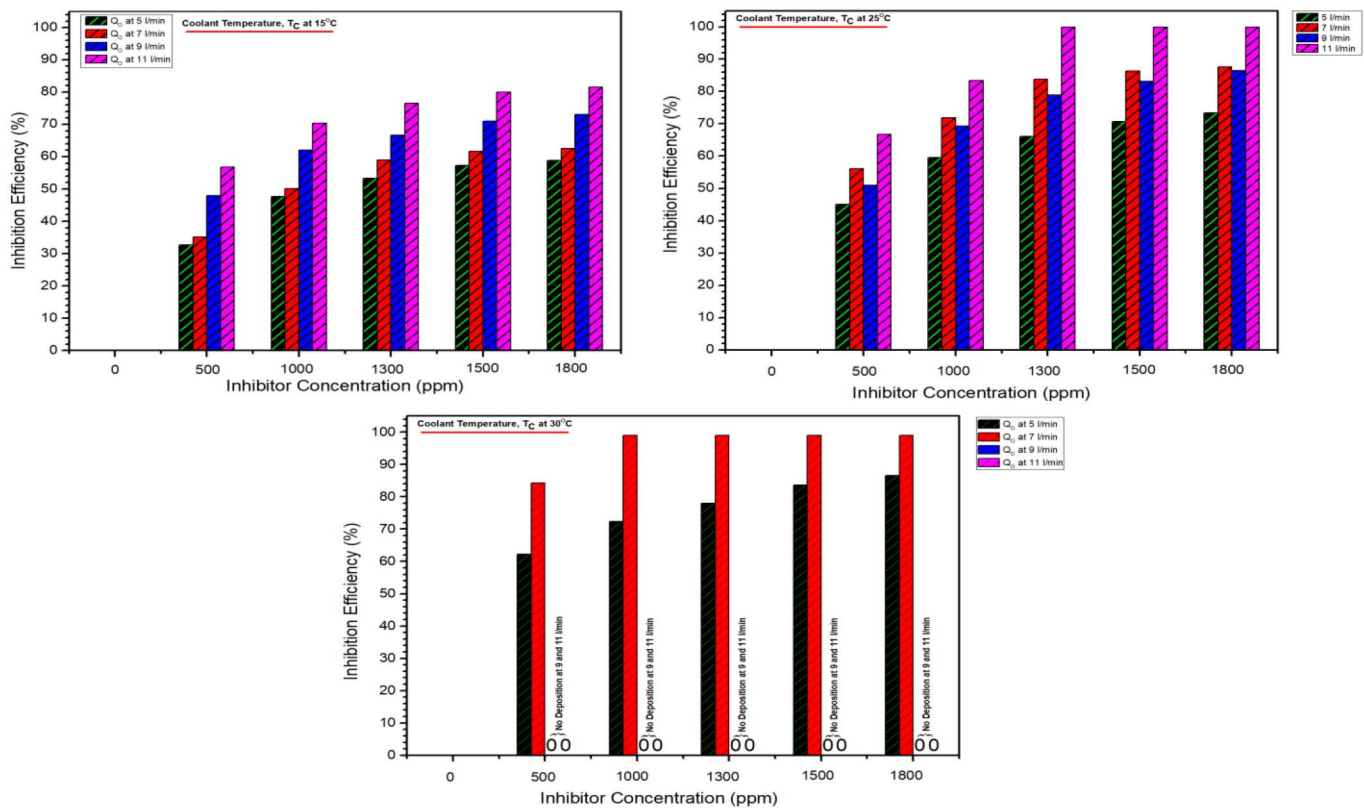

Figure 9—Efficiency of blended crystals modifiers through straight pipeline test section.

It is crucial to improve the understanding of the inhibition behaviour and its effect in varied cooling temperature at different settings. As shown in Figure 10 (a, b, and c) the effect of flow rate was examined at different coolant temperature (15, 20, 25, 30 and 35°C). The performance as observed in Figure 8 was also detected in the Figure 10, meaning at higher concentration, higher flow rate and cooling, the inhibition performance was achieved. However, it is remarkable to mention that at turbulent flow regime of $Q_{oil}=$ 11 l/min and around the pour point temperature (below WAT) at $T_{cool}=$ 25°C (in Figure 10b), there is zero deposition observed at 1000, 1500 and 1800-ppm, whereas, the deposit thickness with 0-ppm is $\delta_{wax} \approx 0.18$ mm at the same conditions. At this point, it is believed that the polymeric inhibitor precipitates and acts as crystalline nucleus for the wax crystals (Li *et al.*, 2018), which eventually leads to the formation of a more subcritical nuclei of micelle-like aggregates. The aggregate particles are transported along with the

bulk of crude oil due to high shear rate and sometimes the particles inhibit the deposit wax by stripping effect in the system.
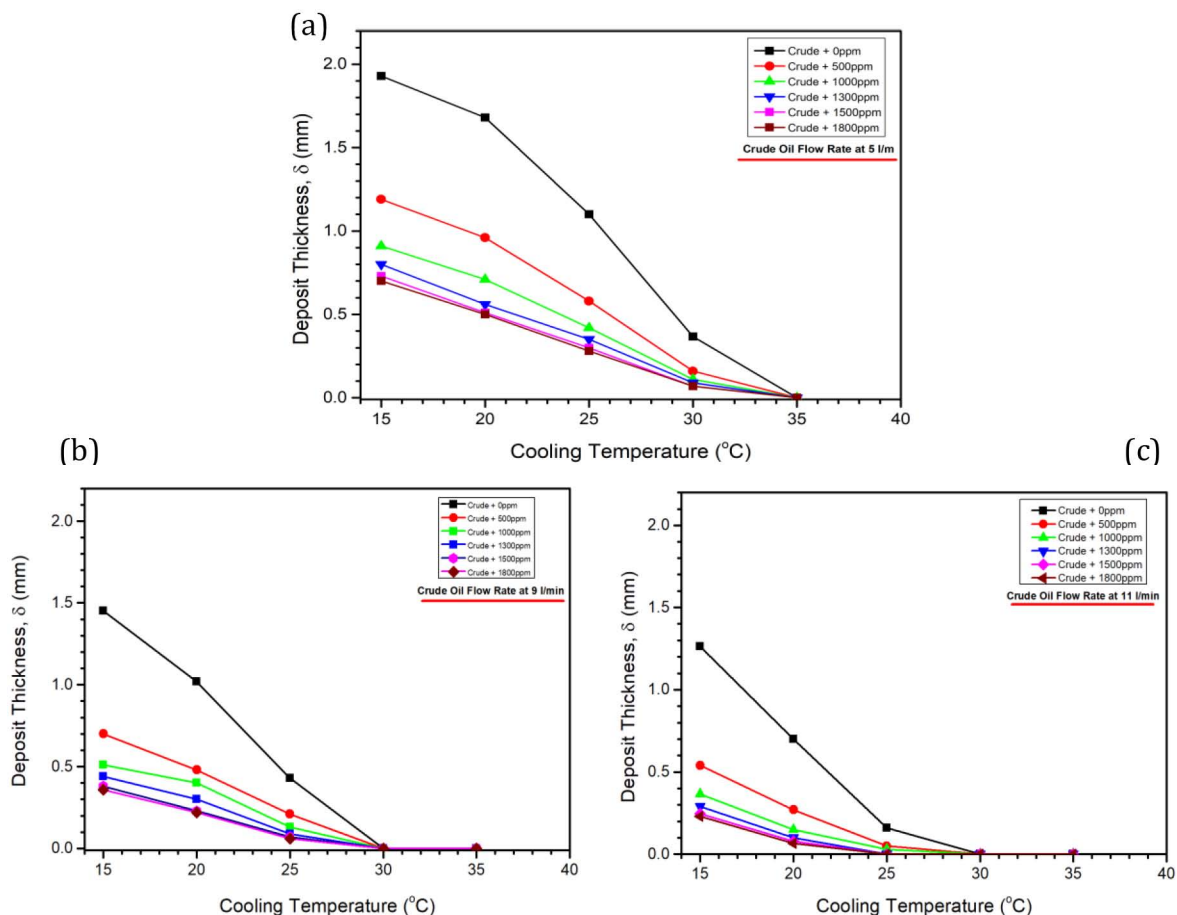

Figure 10—Effect of varied cooling temperature on Wax Deposit Thickness
at turbulent flow (9 and 11 l/min) - Straight Pipeline Test Section.

On the other hand, the inhibition performance was studied in horizontal flow with 45° bend pipeline test section (Figure 11), which produced severe deposition scenario compared to the straight pipe. Similarly, the 45° bend pipeline test section was switched to an inclined flow (Figure 12) in order to understand the settling effect of particles based on the gravitational mechanism and other influential factors (e.g. bend parameters, momentum and inhibitor concentration etc.). It has been demonstrated that wax deposition in horizontal straight pipe is highly controlled by molecular diffusion mechanism only (Burger et al., 1981). Whereas, in the pipeline with bends, it is revealed that apart from the molecular diffusion, shear dispersion and stripping, gravity effect play a major role as the deposition mechanism. This could be due to the forces acting in the system (such frictional, momentum and gravity) and flow separation around the bend. However, it is clearly understood that shear stripping opposed the deposition process. Makwashi et al. (2019) revealed that as the fluid experiences isolation during separated flow, a rapid thermal difference from the bulk fluid could result in the swift molecular diffusion process particularly at lower temperature (Burger et al., 1981; Kok and Saracoglu, 2000).

Figure 11(a, b and c), shows the result of inhibition performance in a horizontal pipe with 45° bend at different flow rate and concentration. Figure 11a, the extreme case (i.e. at T_{cool}= 15°C), higher wax thickness, $\delta_{wax} \approx 3.08$ *mm* was seen at low flow rate (5 l/min) at 0-ppm. This severe deposition was inhibited by doping the crude oil with 500, 1000, and 1500-ppm. At these concentrations, low deposition was achieved without altering the operation extreme condition. At 500 and 1000-ppm, the thickness was reduced to $\delta_{wax} \approx 1.8$

and 1.4 *mm*, whereas, 0.9 *mm* thickness was measured at 1500-ppm. Hence, in percentage, the deposition was reduced from 43% to 25.3, 19.1 and 12.6% at 0, 500, 1000 and 1500-ppm. However, as the coolant temperature increases to 25 and 30°C (Figure 11, b and c) inhibition performance increases considerably. At 25°C cooling temperature, which is around the pour point, no deposition is observed at higher turbulent flow (11 l/min), while very small amount of wax thickness was seen at low turbulent flow (9 l/min). Unlike, in a laminar flow regime, where the thickness is relatively higher, $\delta_{wax} \approx 0.36mm$ at 5 l/min and $\delta_{wax} \approx 0.132mm$ at 7 l/min respectively. This is because in a laminar flow regime the shear effect and Brownian dispersion are insignificant (Zhu *et al.*, 2008). Thus, the particles are well ordered and could easily deposited on the surface. Hence, in horizontal pipeline with bend, molecular diffusion and shear dispersion (due to momentum change) are the possible dominant mechanisms for wax deposition.

Figure 11—Effect of variation of flow rate, and inhibitor concentration on Wax Deposit Thickness at:
(a) T_{cool}= 15°C, (b) T_{cool}= 25°C, and (c) T_{cool}= 30°C through Horizontal pipeline section with 45° bend.

On the other hand, Figure 12(a, b and c) shows the results in which the horizontal pipe test section with 45° bend was switched to an inclined flow at different flow rate and concentration. A study was reported by Burger *et al.* (1981) where the gravity settling mechanism was tested by switching a straight pipe test section from horizontal to vertical flow, with no effect of gravity settling observed in contribution to wax deposition. However, as shown in the Figure 12a, it is observed that at lower flow rate (Q_{oil}= 5 l/min) in inclined bend test section, the wax thickness at 0-ppm and T_{cool}= 15°C is $\delta_{wax} \approx 4.12 \ mm$. Unlike in horizontal position, $\delta_{wax} \approx 3.1 \ mm$ at the same condition. In both cases, the severity of wax deposition decreases with increasing the flow rate, cooling temperature and concentration. As shown in this work, it is obvious that apart from the molecular diffusion and shear dispersion, it was observed that the effect of 'gravity settling'

is shown as the bend pipes switched from horizontal to an inclined flow. More details can be found in Makwashi *et al.* (2019).

As shown in the Figure 12 b and c, as the cooling temperature and flow rate increase with increasing the concentration, the performance of the inhibitor is enhanced. It is obvious that at 9 and 11 l/min, under both coolant temperatures of 25 and 30°C no wax deposit was measurable. Clearly, it can be seen that at 30°C almost 100% inhibition efficiency was achieved even at lower flow rate and at 1500-ppm.
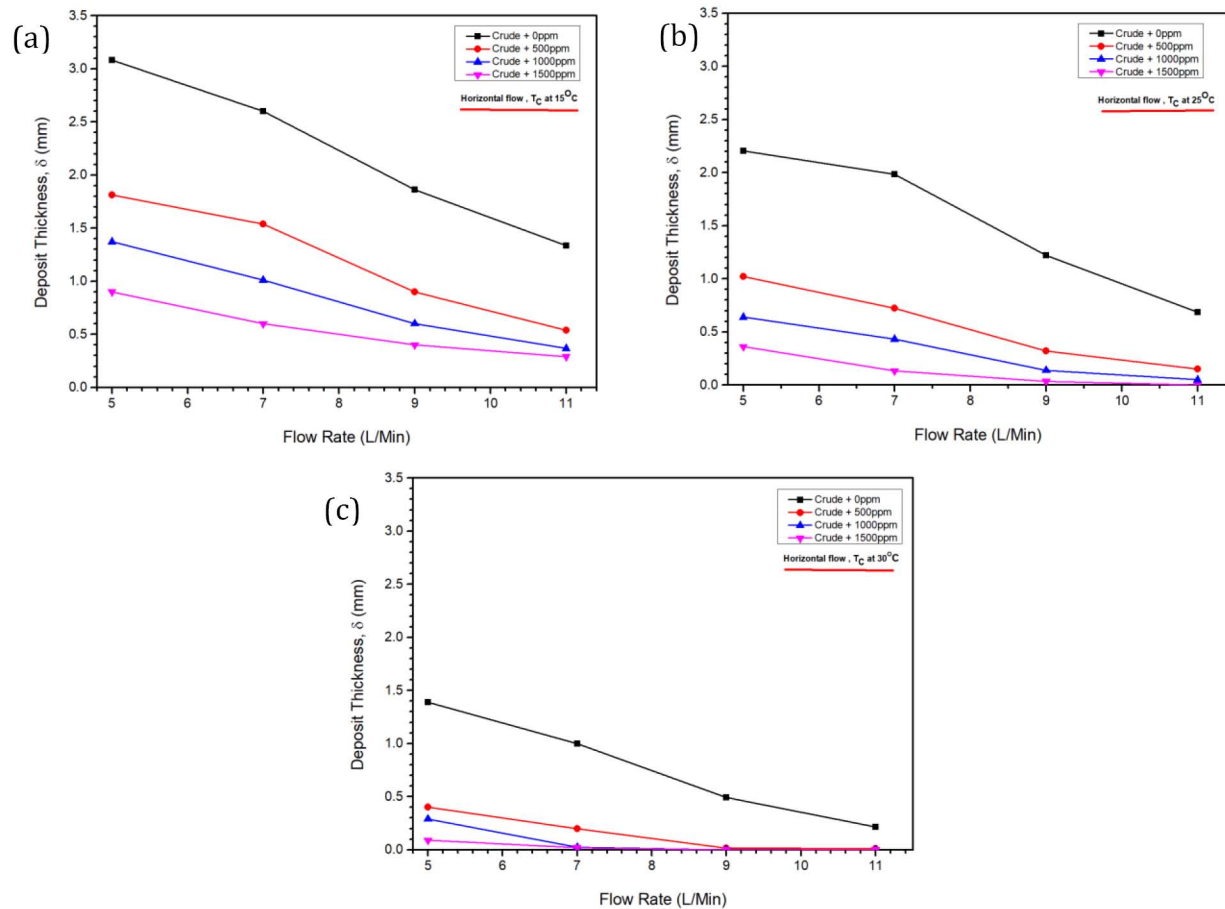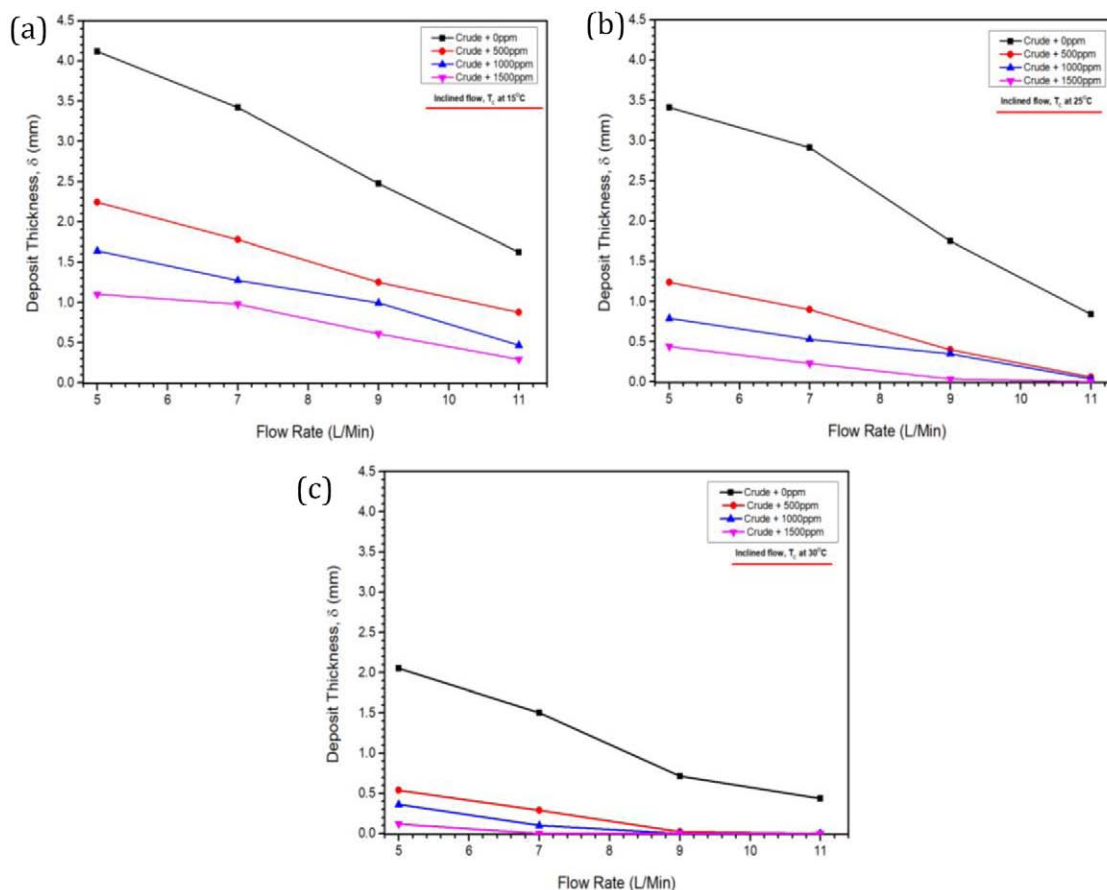

Figure 12—Effect of variation of flow rate, and inhibitor concentration on Wax Deposit Thickness at: (a) T_{cool}= 15°C, (b) T_{cool}= 25°C, and (c) T_{cool}= 30°C through inclined pipeline section with 45° bend.

Interestingly, the combined effect of variations in flow rate, temperature and inhibitor concentration on the deposited wax (in mass) is presented in a surface 3D plot in Figure 13 and Figure 14. First, a general trend in the presented results indicates a reduction in the amount of wax by increasing inhibitor concentration and flow rates at different temperatures of 15°C, 25°C and 30°C. The effect of increasing flow rate tends to increase turbulence in the pipe, thus, reduce the residence time for deposition. Even though wax formation could occur, the deposition process is however, not encouraged due to these shearing effects in the pipe. Hence increasing the oil flow rate from 5 l/min to 11 l/min a reduction in wax deposition for all cases are presented in Figure 13 and Figure 14. As revealed in the previous sections, an optimal inhibitor concentration of 1500-ppm which corresponds to the maximum concentration of inhibitor required to reduce the wax in the straight and bend pipes reduces deposition to nearly zero. This was the case for all scenarios as presented for all results.

Moreover, Figure 13a shows initial mass deposit of wax of 79 g at 15°C, compared to that for a higher temperature in Figure 13b and 13c at 25°C and 30°C for 39.5g and 15.9g respectively. These reductions with increasing temperature were expected and further indicative of the conditions above the

wax appearance temperature (WAT). In summary, it is imperative for operators to consider these combined effects to minimize wax deposition in the pipe line. In a practical approach, the control of temperature and concentration of the inhibitor in the pipeline could be modelled and implemented to reduce wax deposition, since the flow rate is a set deliverable.

Figure 13—Combined Effect of varied flow rate, cooling temperature and inhibitor concentration on Deposited Wax (in mass): (a) at T_{cool} = 15°C, (b) at T_{cool} = 25°C, and (c) at T_{cool} = 30°C – Through Pipeline Test Section

Figure 14—Combined Effect of varied flow rate, cooling temperature and inhibitor concentration on Deposited Wax (in mass): (a) at T_{cool}= 15°C, (b) at T_{cool} = 25°C, and (c) at T_{cool}= 30°C. Through Pipeline Test Section with 45° bend.

Conclusion

The wax content, *n*-paraffin distribution, wax appearance temperature (WAT), and pour point, which defined the crude oil as naturally waxy oil are measured via HTGC. Similarly, the effects of different chemical inhibitors on the crude oil rheological properties are obtained. The flow rig experiments were conducted to investigate the effect of different influential parameters and geometry on wax deposition with and without the presence of blended wax inhibitor, and under variable coolant temperatures, flow rates, and inhibitor concentrations.

For wax deposition study without inhibitor, it has observed that the deposition is always higher in the bends pipeline compared to the straight pipe. The performance of blended inhibitor is much better as related to other tested inhibitors. The performance slightly reduced at low concentration (e.g. 500-ppm) compared to a high dosage. This is due to the adsorption and co-crystallization processes of the inhibitor. Nonetheless, as the concentration increases above 1500-ppm, no improved behaviour was observed. Therefore, the actual optimal inhibitor dosage is a huge concern in the oil field due to cost and environmental aspects. However, in this study, 1500-ppm was considered as the optimal concentration, which corresponds to the maximum dosage of inhibitor required to provide maximum inhibition experience. From the study, a conclusion can be made that the efficiency of the inhibitor decreases with decreasing concentration, lower coolant temperature and flow rate for both flow conditions. Therefore, it is imperative for field operators to consider these combined effects to minimize wax deposition in the pipeline. In a practical approach, the control of temperature and concentration of inhibitor in the pipeline could be modelled and implemented to reduce wax deposition, since the flow rate is a set deliverable.
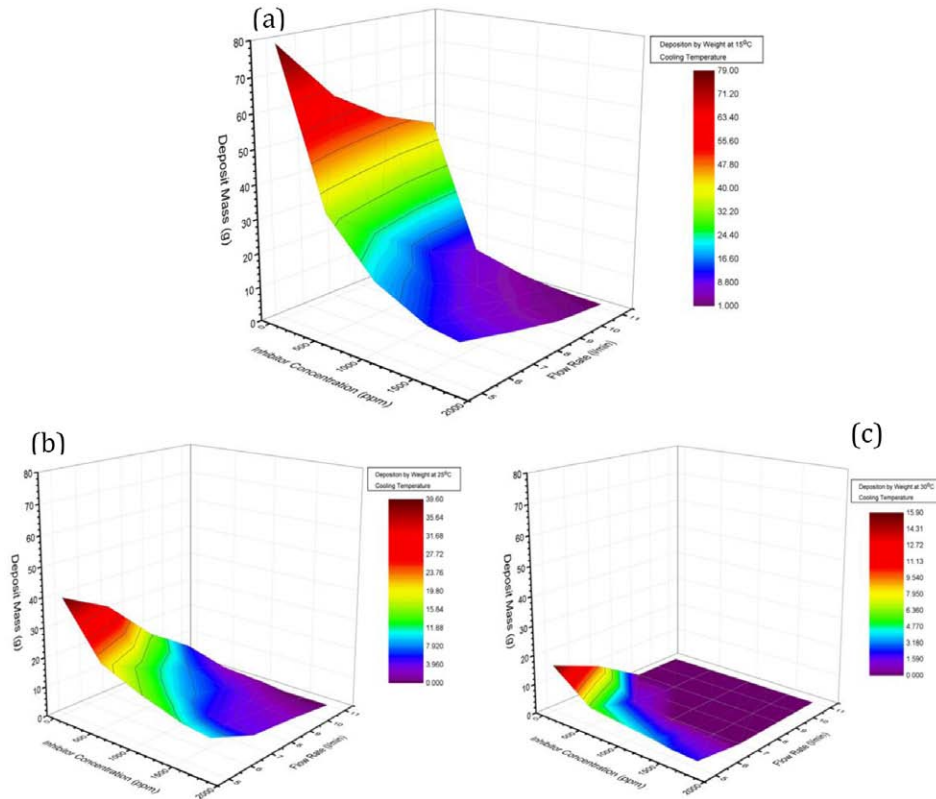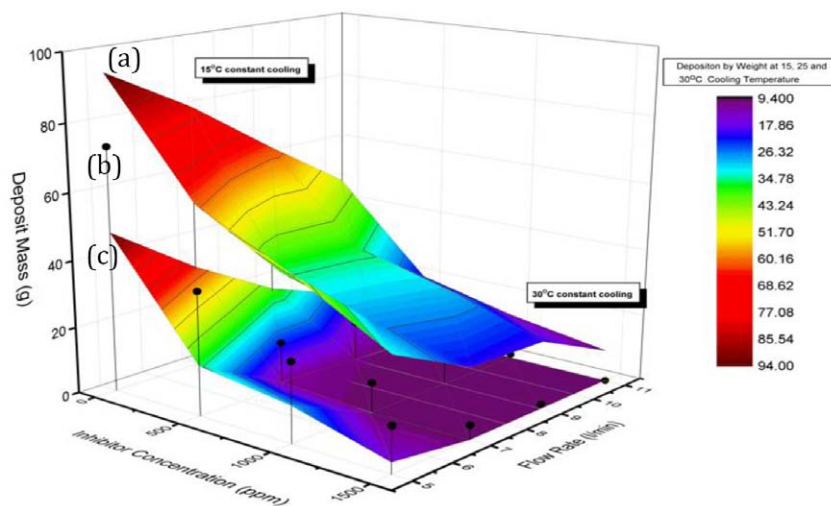
Acknowledgements

Nomenclature

d	diameter of the pipeline
c and n	constant parameters
f	frictional factor
ΔP_f	pressure drop
L	length of pipe section
Q	volumetric flow rate
ρ	fluid density
δ_w	average thickness of the wax deposit
M_{dep}	Mass of wax deposit
ρ_{dep}	Density of wax deposit
W_f	amount of wax deposit without inhibitor
W_t	amount of wax deposit with inhibitor treatment at different concentrations.
WIE	Wax inhibition efficiency

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Appendix

Table A 1—Table A: HTGC Processing Data for the Crude Oil Sample

| Carbon Number | Sensitivity (pA*s/ng) | WHOLE OIL | | | TOPPED OIL | | | MERGED DATA | | |
		RT (mins)	Area (pA*s)	Mass (ng)	RT (mins)	Area (pA*s)	Mass (ng)	Adj. Mass (ng)	Mass (ng)	n-Paraffin Wt %
10	16.47									
11	16.89	4.26	43.1	2.55					2.6	**0.102**
12	18.38	5.08	340.3	18.52					18.5	**0.786**
13	18.56	6.18	289.0	15.57					15.6	**0.651**
14	18.74	7.93	219.5	11.71					11.7	**0.483**
15	18.92	8.92	185.3	9.79	8.97	0.1	0.01	0.00	9.8	**0.398**
16	19.11	10.03	257.3	13.46	10.05	0.7	0.04	0.01	13.5	**0.541**
17	19.29	11.14	1269.5	65.81	11.12	5.8	0.30	0.05	65.8	**2.310**
18	19.47	12.11	495.0	25.42	12.11	16.6	0.85	0.15	25.4	**0.996**
19	19.66	13.07	555.5	28.26	13.08	117.7	5.99	1.03	28.3	**1.094**
20	19.84	14.00	601.9	30.34	14.02	512.7	25.84	4.45	30.3	**1.161**
21	20.40	14.88	603.9	29.61	14.92	1254.2	61.49	10.58	29.6	**1.153**
22	20.48	15.73	614.8	30.01	15.79	2092.6	102.16	17.58	30.0	**1.163**
23	20.53	16.54	615.1	29.97	16.62	2703.2	131.70	22.66	30.0	**1.152**
24	20.57	17.32	621.7	30.23	17.41	3066.5	149.10	25.65	30.2	**1.153**
25	20.61	18.08	608.4	29.52	18.17	3247.4	157.57	27.11	29.5	**1.118**
26	20.65	18.80	592.5	28.69	18.90	3305.4	160.06	27.54	28.7	**1.079**
27	20.69	19.50	589.8	28.50	19.61	3361.6	162.45	27.95	28.5	**1.064**
28	20.74	20.18	552.7	26.66	20.28	3173.2	153.03	26.33	26.7	**0.995**
29	20.78	20.83	538.6	25.92	20.94	3130.5	150.67	25.92	25.9	**0.973**
30	20.82	21.46	456.2	21.91	21.56	2654.7	127.51	21.94	21.9	**0.817**
31	23.86	22.07	414.2	17.36	22.17	2525.0	105.82	18.21	18.2	**0.781**
32	23.84	22.66	306.5	12.86	22.75	1808.4	75.86	13.05	13.1	**0.562**
33	23.85	23.24	263.6	11.05	23.33	1644.9	68.96	11.87	11.9	**0.514**
34	23.86	23.79	140.1	5.87	23.86	859.6	36.02	6.20	6.2	**0.27**
35	23.88	24.33	101.4	4.25	24.39	619.4	25.94	4.46	4.5	**0.196**
36	23.89	24.86	50.3	2.11	24.90	306.8	12.84	2.21	2.2	**0.097**
37	23.90	25.37	37.2	1.56	25.41	223.2	9.34	1.61	1.6	**0.071**
38	23.92	25.87	23.6	0.99	25.91	147.9	6.18	1.06	1.1	**0.047**
39	23.93	26.36	16.1	0.67	26.40	101.5	4.24	0.73	0.7	**0.0327**
40	23.94	26.84	11.9	0.50	26.87	70.9	2.96	0.51	0.5	**0.023**
41	22.07	27.30	9.1	0.41	27.33	59.3	2.69	0.46	0.5	**0.0192**
42	22.07	27.76	7.6	0.34	27.79	47.4	2.15	0.37	0.4	**0.0154**
43	22.07	28.20	6.2	0.28	28.23	42.8	1.94	0.33	0.3	**0.0139**
44	22.07	28.64	5.7	0.26	28.66	36.0	1.63	0.28	0.3	**0.0117**
45	22.07	29.06	4.4	0.20	29.09	33.5	1.52	0.26	0.3	**0.0109**
46	22.07	29.47	3.4	0.15	29.50	26.8	1.22	0.21	0.2	**0.0087**
47	22.07	29.88	3.1	0.14	29.90	24.1	1.09	0.19	0.2	**0.0079**

Carbon Number	Sensitivity (pA*s/ng)	WHOLE OIL			TOPPED OIL			MERGED DATA		
		RT (mins)	Area (pA*s)	Mass (ng)	RT (mins)	Area (pA*s)	Mass (ng)	Adj. Mass (ng)	Mass (ng)	n-Paraffin Wt %
48	22.07	30.27	2.7	0.12	30.30	20.9	0.95	0.16	0.2	0.0068
49	22.08	30.66	2.9	0.13	30.69	19.0	0.86	0.15	0.1	0.0062
50	22.08	31.04	2.2	0.10	31.07	18.2	0.82	0.14	0.1	0.006
51	33.99	31.42	2.2	0.06	31.44	17.0	0.50	0.09	0.1	0.0057
52	34.00	31.78	2.0	0.06	31.80	14.9	0.44	0.08	0.1	0.0051
53	34.17	32.14	1.8	0.05	32.16	15.3	0.45	0.08	0.1	0.0053
54	34.33	32.49	1.5	0.04	32.51	13.9	0.40	0.07	0.1	0.0049
55	34.50	32.83	1.5	0.04	32.86	14.0	0.41	0.07	0.1	0.0051
56	34.67	33.17	1.3	0.04	33.19	12.8	0.37	0.06	0.1	0.0047
57	34.84	33.50	1.3	0.04	33.52	13.5	0.39	0.07	0.1	0.0051
58	35.01	33.82	0.7	0.02	33.85	11.2	0.32	0.06	0.1	0.0043
59	35.18	34.14	0.8	0.02	34.16	11.3	0.32	0.06	0.1	0.0045
60	35.35	34.45	0.6	0.02	34.48	8.4	0.24	0.04	0.0	0.0034
61	17.34	34.77	0.3	0.02	34.78	8.5	0.49	0.08	0.1	0.0034
62	17.34	35.08	0.6	0.04	35.09	4.8	0.28	0.05	0.0	0.0019
63	17.34	35.37	0.3	0.02	35.38	5.3	0.30	0.05	0.1	0.0021
64	17.34	35.70	0.4	0.03	35.67	2.6	0.15	0.03	0.0	0.001
65	17.34	35.98	0.3	0.02	35.96	2.2	0.12	0.02	0.0	0.0009
66	17.34				36.25	0.7	0.04	0.01	0.0	0.0003
67	17.34				36.53	0.7	0.04	0.01	0.0	0.0003
Total Wax content (C$_{15}$-C$_{67}$)										19.4