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Experimental Methodology Followed to Evaluate Wax Deposition Process

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Abstract

This study illustrates the methodology used to evaluate wax deposition volume. It outlines the details of the rig used to evaluate the wax deposition process and study the factors that influence on the wax deposition process such as crude oil temperature, flow rate, pressure drop, inlet coolant temperature, oil viscosity, shear stress, time. Also, presents the different chemicals used as inhibitors to mitigate wax deposition and spiral flow as a mechanical method used to reduce wax deposition in the crude oil pipeline. This study, presents the standard analytical and chemical methods to characterise the crude oil. The experimental rig described in more details during this study. In this study, several strategies are followed and illustrated to study its influence on wax deposition process. During this work, four different techniques are used to estimate wax deposit thickness. These techniques are including pressure drop, pigging, heat transfer, and liquid displacement-level detection technique (LD-LD).

After following the methodology to evaluate the wax deposition volume and the mitigation methods to decrease wax deposition, the results indicates that the reduction in wax deposition was 100% after using the effect of bending the spiral flow with the inhibitor W802 at a concentration of 1000 and 2000ppm at different time and flow rates. The reduction in wax deposition was 100% after using the effect of bending the spiral flow with the inhibitor W802 at a concentration of 500ppm at flow rate 4.8 L/min, and the reduction in wax deposition was 94% at the same concentration and flow rate 2.7 L/min.

Keywords: Oil; Hydrocarbons; Pipeline; Crude oil; Paraffin

Inroduction

One of the main fluid flow assurance issues in the oil industry world is wax deposition. Wax deposition in the hydrocarbons pipeline can leads in the restriction of crude oil flow, producing an artificial blockage, pressure abnormalities that reduce the production. In very extreme cases, wax deposition can lead to pipeline or production facility to be abandoned. In the cold environments, when the inner pipe wall temperature drops below the wax appearance temperature, the paraffin components in crude oil start to precipitate and deposit on the cold pipeline wall creating the layer of wax.

Low temperature is considered as the main factor that affects the process of wax deposition, which means that subsea pipelines are especially vulnerable. Thus, the prevention of wax deposition process comes to be very important in deep- water oil production. Several methods have been used to reduce or prevent wax deposition by using one or combination of chemical, mechanical, and thermal remediation methods. Though, with the deep production, offshore drilling and ocean floor completions, the use of thermal and mechanical inhibition methods becomes high-priced economically; therefore, the chemical additives become more prevalent [1]. Selected chemical inhibitor was tested in the current work to study its effect on wax deposition. Furthermore, exams the effect of bending spiral flow with the chemical inhibitor on wax deposition.

This study shows the methodology followed to measure wax deposition volume and the techniques to estimate wax thickness including pressure drop, pigging, heat transfer, and liquid displacement-level detection technique (LD-LD). The impact of various factors on the wax deposition process were studied, such as the inlet coolant temperature 14°C, 24°C and 33°C, circulating time of oil at 2, 3, 4, 5 and 6 hours and the variation of flow rate from 2.7 to 4.8 L/min.

During this work, numerous wax deposition experiments were carried out using the chemical inhibitor Polyacrylate polymer W802 at a concentration of 500ppm, 1000ppm, and 2000ppm, respectively. The new technique of bending spiral flow with W802 at different concentrations presents valuable results comparing with the results of just using Polyacrylate polymer.

The wax deposit volumes at 14°C for each experiment set. At the flow rate of 2.7 L/min, the maximum volumes of wax were found by carrying out the experiment using just crude oil at a different experimental time, while the minimum volumes of wax were found by carrying out the experiment using the effect of bending the spiral flow with 2000 ppm of W802. Increasing the flow rate or increasing the pipe wall temperature obviously reduces the wax deposit volume (Figure 1).

Experimental Methodology

Crude oil characterisation

The examined crude oil is one of the oil field reservoirs that experience waxing problems in the extreme north-eastern part of India. The characterisation of the crude oil was carried out using experimental methods and standard analytical techniques. The characteristics details of the crude oil are shown in Table 1 [2,3].

Specific gravity and API

Specific gravity was calculated using the ratio of the density of crude oil ρ_{oil} to the density of water ρ_{water}. Specific gravity is a dimensionless quantity.

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$$SG = \frac{\rho_{oil}}{\rho_{water}} = \frac{850}{1000} = 0.85 \quad (1)$$

API gravity is a degree used to evaluate how is heavy or light the crude oil compared to water. The formula used to calculate API gravity from specific gravity (SG) is:

$$API = \frac{141.5}{SG} - 131.5 = \frac{141.5}{0.85} - 131.5 = 35 \quad (2)$$

Rheological behaviour

The rheological behaviour of the crude oil during this study was evaluated using a Bohlin Gemini II Rheometer. The principle of the rheometer work is to placing the sample of oil between a lower horizontal plate and an upper rotating plate to measure oil viscosity, pour point and wax appearance temperature at different shear rates, temperatures and shear stresses. The measurements begin by cooling down the crude oil from 55°C to 0°C at a rate of 5°C/min and shear rates of 10, 60, 120 and 180 1/s. The intersection between the viscosity lines at different shear rates represents the wax appearance temperature.

The pour point can be measured from the curve of crude oil viscosity, where, at this point the liquid is converted from a non-Newtonian to a Newtonian liquid. The pour point and wax appearance temperature of the crude oil of this study are shown in Figure 2.

Wax content

The precipitation method was used to determine wax content. 2 ml of crude oil is diluted in 4 ml of n-pentane and stirred for 30 minutes. Acetone (acetone/n-pentane ratio 3:1) is added to the mixture and cooled down to -20.15°C for 24 hours. The precipitated solid phase from the diluted oil was separated by filtration in a Buchner funnel using a glass microfibre Whatman filter N° 934. N-hexane was used to re-dissolve the separated solid phase in order to remove asphaltenes. After solvent removal, the final product is weighed.

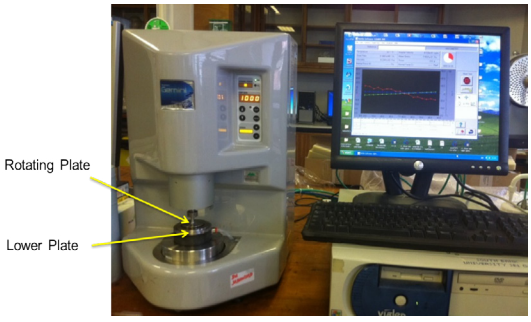

Figure 1: The Rheometer (Bohlin Gemini II) used to measure viscosity, WAT and pour point [11].

Property	Unit	Value	Method
Oil Density	kg/m³ (15°C)	850	Divided mass to volume
Sp. Gravity	60/60 °F	0.85	Calculated
API Gravity	60 °F	34.97	API
Wax Appearance Temperature at shear rate 10 1/s	°C	39	Using Rheometer
Wax Appearance Temperature at shear rate 120 1/s	°C	30	Using Rheometer
Pour Point or cloud point	°C	27.6	Using Rheometer
Wax Content	wt%	20.15	Precipitation Method
Saturates Content	wt%	74.91	SARA
Aromatics Content	wt%	20.44	SARA
Resins Content	wt%	4.26	SARA
Asphaltene Content	wt%	0.39	SARA

Table 1: Crude oil characteristics [8-10].

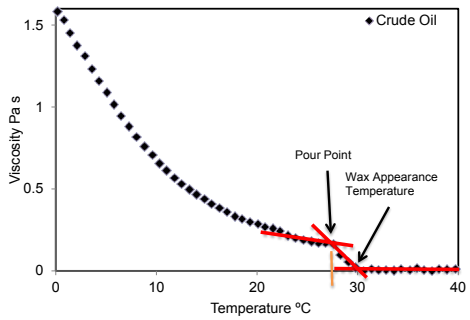

Figure 2: Variation of viscosity of Arunachal crude oil with temperature, indicating the WAT and pour point using the rheometer [11].

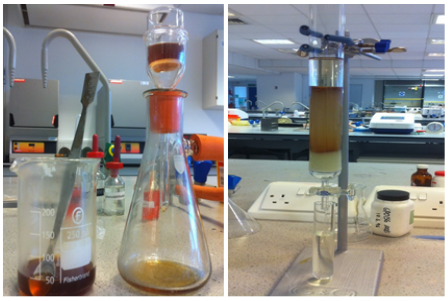

Figure 3: (a) Filtration equipment used during this study to separate asphaltene, and (b) chromatographic column used to separate saturate, aromatic and resin.

SARA analysis

SARA analysis was used to separate the crude oil into four major classes of compounds – saturates, aromatics, resins, and asphaltenes (SARA) – using a chromatographic column, as shown in Figure 3. The purpose of this analysis is to understand what the crude oil contains, which helps in easily dealing with the wax deposition problem.

This method was carried out on a de-asphaltene 10 gram of crude oil with n-heptane; the asphaltene content measured from the weight of the precipitate. The maltene (filtrate) was freed from the solvent to the greatest possible extent, then weighed, and 2 gm of it was added to the chromatographic column with activated silica (100°C) saturated with n-heptane and eluted sequentially with 100 ml of n-heptane (elution of saturates); 100 ml of toluene (elution of aromatics); 100 ml of toluene-methanol solution (50:50) (elution of polars/resins); 100 ml of methanol-chloroform solution (50:50) (elution of polars/resins); 100 ml of chloroform (elution of polars/resins); and 100 ml of acetonitrile (elution of polars/resins) [4].

Elutes were initially collected in separate containers and those from the last four elution sequences were subsequently mixed, stripped of their respective solvents under vacuum and weighed. The saturate, aromatic and resin contents were calculated from the weight percent of the residue in the whole crude [4].

Experimental Rig used to Study Wax Deposition

The experimental rig of Theyab and Diaz was used to study the wax deposition thickness under the single phase (Figures 4 and 5) [1-3].

This rig facilitated a study of the impact of some factors, such as flow rate, pressure drop, inlet coolant temperature, crude oil temperature, oil viscosity, time, shear stress, chemical inhibitors and spiral flow, that influence and control on wax deposition process.

In this study the test flow loop described in more details, where, its consists of test section, copper pipe jacket, pump, crude oil reservoir, hot bath water, Churchill Conair chillier, manometer pressure gauge, thermocouples, condenser, and Pico-Meter.

Test section

The place where paraffin deposition investigation takes place is called the pipe test section. The test section is consisting of a copper 150 cm in length and diameter of 1.35 cm and the pipe wall thickness is 0.09 cm. The copper pipe material properties are 385 J/kg.°C heat capacity, thermal conductivity is 401 W/m.k, density is 8960 kg/m^3 and pipe wall roughness is 0.00009 m. The high thermal conductivity of copper pipe facilitates heat transfer between the crude oil and the environment leading to form wax crystals and precipitates on the wall of pipe. This type of pipe material is suitable for the lab studies and not suitable for the oil fields in the cold environment.

Copper pipe jacket

The test section is jacketed with a copper pipe jacket in which cold glycol-water mixture is pumped from the chillier to maintain a pipe wall temperature lower than the WAT. The inside diameter of the jacket pipe is 2.54 cm and the pipe wall thickness is 0.12 cm. The copper pipe material properties are 385 J/kg.°C heat capacity, 401 W/m.k thermal conductivity, 8960 kg/m^3 density and 0.00009 m pipe wall roughness.

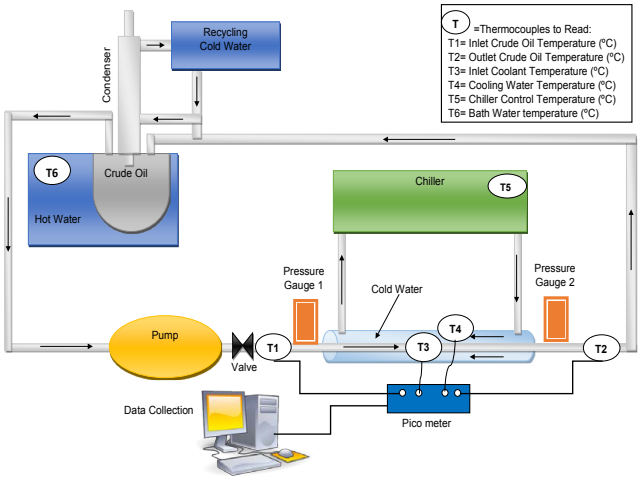

Figure 4: Diagram of wax deposition flow loop [8-10].

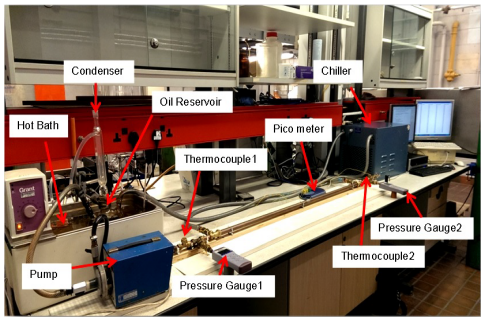

Figure 5: Apparatus of wax deposition in the lab for this study [12].

Pump

A Charles Austin pump C25C used to recycling crude oil through the test section and go back to the oil reservoir. This pump is connected with a valve to control the desired flow rate. The pump flow rate had ± 0.05 L accuracy in the flow range of 0-4.8 L/min. The pump was calibrated to give a specific flow rate; this is achieved by measuring the amount of crude oil at a given time at various flow rates 2.7 and 4.8 L/min, until the specified rate is reached.

Crude oil reservoir

A three-neck glass boiling flask, round bottom glass, 2000 ml, made from borosilicate glass 3.3, weight 0.8kg, and range of operating temperature between -80°C to 200°C, was used as a reservoir for crude oil. Crude oil is entering into the flask from the first neck after recycling in the test section; crude oil exits from the second neck to the pump; and the third neck is connected with the condenser. This flask is fixed in a controlled heating bath. The crude oil reservoir volume was calibrated by measuring its volume with 2000 ml of crude oil.

Hot bath water

A water bath is made from a container stainless steel, length 0.5 m, width 0.4 m, and height 0.4 m, filled with heated water was used to incubate the crude oil reservoir in water to control the oil at a constant temperature at a constant temperature. The water bath has a manual control to set the desired temperature for the experiment. The preferred heat source for heating the flammable chemicals is the water bath because it considered as a safe heating source to prevent ignition. The bath water temperature range is reached up to 100 °C. A thermometer was used for verification of the temperature of the hot bath.

Churchill conair chiller

The type of the chiller used during this study was Churchill Conair; the cooling liquid used inside the chiller is glycol-water mixture and the temperature range of the chiller is between -60 to +60°C. The chillier model number is 02 CTCV, serial number 8341695, length 0.6 m, width 0.4 m, and height 0.5 m.

The chiller has dual rules in the experiments, where it was used to maintain the pipe wall temperature of the test section at the desired temperature, also it was used to melt the wax deposit on the pipe wall after each experiment, by heating the recycling glycol-water mixture to increase the pipe wall temperature and melt the deposit wax.

Manometer pressure gauge

Two manometers Pressure/Vacuum Gauges type Fisher Scientific™ Traceable™ were used to measure the inlet pressure and outlet pressure of the test section pipe.

The pressure gauge is portable unit measure and display differential pressure/vacuum in 11 different units (psi, mbar etc.). The pressure range is between 0 - ±100 Psia, the accuracy ±0.3% according to the company record, dimensions (L × W × H) (18.1 × 7 × 2.9 cm), and display type LCD.

Thermocouples

Self-adhesive patch thermocouples were used in four different positions to measure the temperature. These types of thermocouples are appropriate for attaching to flat or curved surfaces, these self-adhesive patch thermocouples are suitable for measuring operation temperature between -50°C and +250°C. The self-adhesive patch thermocouple was

pre-calibrated for temperature from Thermosense Direct Company. They are made from PFA insulated cable which is attached to a high temperature rated patch of PTFE impregnated adhesive fibreglass tape. The patch size is 25 mm × 20 mm, 0.2 mm diameter solid PFA insulated twisted pair leads, and the lead length is 2 metres.

Two of the thermocouples were used to measure the inlet and outlet temperature of the crude oil along the test section. The rest two thermocouples, one of them used to measure the temperature of the recycling cooling water and the second used to measure the inlet coolant temperature (pipe wall temperature). On another hand, a manual thermometer was used for verification.

Condenser

A condenser is a device or unit used to condense a substance from its gaseous to its liquid state, by cooling it. In this study, it was used to condense the remaining light components that evaporated when heating the crude oil.

The condenser is made from Pyrex borosilicate glass with quick fit ground glass joints for easy assembly. The total length of the condenser is 32.5 cm, body length 20 cm bottom diameter 15 mm, upper diameter 25 mm, and joints both are 24/29.

Pico-Meter

Pico is a full-featured multimeter that accurately measures AC/DC voltage, AC/DC current, resistance and ambient temperature. The four types of self-adhesive patch thermocouples was connected to the Pico Meter for data accumulation to a PC via Pico Logger software using intervals of one minute to achieve the best resolution of the results.

Mitigation Methods

In this experimental study, the chemical inhibitor and spiral flow were used as mitigation methods to reduce or prevent wax deposition on the hydrocarbon pipeline. The chemical inhibitor is Polyacrylate polymer to study its effect on crude oil rheology and to reduce wax deposition.

On the other hand, the technique of Theyab and Diaz by using spiral flow inside the pipe in order to increase the shear rate and shear dispersion and mitigate wax deposition was used during this study [5].

As mentioned before, the spiral flow was generated by inserting a twisted plate inside the pipe and examined in the test section of the pipe as shown in Figure 6. The purpose of examined the spiral flow was to study its effect on wax deposition at different flow rates and it was shown an efficient result comparing with the effect of the chemical inhibitors.

Process of Experiments

The most important point before starting any wax deposition experiment is to make sure that the pipe section is clean of any wax deposits remaining from previous experiments. For this, oil is flowing through the test section, which will be used for the following experiment at a temperature well above the wax appearance temperature for about half an hour to dissolve all the remaining wax from the previous experiment.

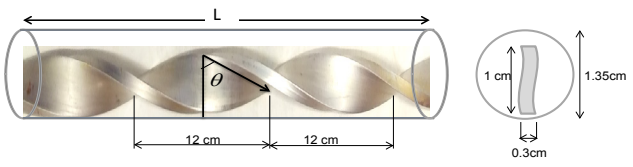

Figure 6: The twisted plate inside the test section of the pipe to create spiral flow.

After this step, the oil temperature is set at the desired operating conditions (44.5°C). on another hand, the previous oil temperature helps to prevent the wax deposition on the pipe wall surface. The glycol-water mixture that recycle through the chiller is kept flowing in the jacket pipe at a temperature close to WAT of the oil (39°C) to avoid any possible thermal gradient in the radial direction. The purpose is to ensure that no wax will deposit on the test section wall before the experiment starts.

After the oil temperature reaches the desired value, the glycol-water mixture temperature should be cooled down to the set value to start the experiment. Each experiment of wax deposition was implemented by pumping the crude oil through the test section at a relatively higher temperature (44.5°C) than the temperature of the pipe wall 14, 24, and 33°C, to create the appropriate wax deposition environment inside the test section. Two manometers pressure gauges were used to monitor the pressure drop of the fluid along the test section.

A change in the temperatures in different positions of the test section of the pipe was monitored using four types of self-adhesive patch thermocouples. Two thermocouples used to measure the crude oil temperature at the inlet and outlet of the pipe. Moreover, two thermocouples used to measure the recycling cooling temperature and the inner pipe wall temperature respectively. The collected data of the different types of temperatures was accumulation to the PC via the Pico Meter through Pico Logger software.

Eight different sets of experiments were implemented to study the effects of different parameters on wax deposition; the different experiments were carried out using just crude oil and run the experiments using crude oil with the inhibitor at different concentration 500 ppm, 1000 ppm and 2000 ppm. The experiments were carried out at flow rates 2.7 and 4.8 L/min, at different pipe wall temperatures 14°C, 24°C, and 33°C, and at various time 2, 3, 4, 5 and 6 hrs, as shown in Tables 2 and 3.

The previous experiments were carried out at different parameters including flow rate, inlet coolant temperature, pressure drop, oil

Experiment at Flow Rate 2.7 L/min.	Inlet Coolant Temperature 14°C					Inlet Coolant Temperature 24°C					Inlet Coolant Temperature 33°C				
	Experimental Time (hour)					Experimental Time (hour)					Experimental Time (hour)				
Crude Oil	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
500 ppm W802	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
1000 ppm W802	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
2000 ppm W802	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
Oil + Spiral Flow	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
500 ppm + Spiral Flow	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
1000 ppm + Spiral Flow	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
2000 ppm + Spiral Flow	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6

Table 2: Experiments carried out at a flow rate of 2.7 L/min., different inlet coolant temperatures, different recirculation times and different methods.

Experiment at Flow Rate 2.7 L/min.	Inlet Coolant Temperature 14°C					Inlet Coolant Temperature 24°C					Inlet Coolant Temperature 33°C				
	Experimental Time (hour)					Experimental Time (hour)					Experimental Time (hour)				
Crude Oil	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
500 ppm W802	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
1000 ppm W802	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
2000 ppm W802	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
Oil + Spiral Flow	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
500 ppm + Spiral Flow	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
1000 ppm + Spiral Flow	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6
2000 ppm + Spiral Flow	2	3	4	5	6	2	3	4	5	6	2	3	4	5	6

Table 3: Experiments carried out at a flow rate of 4.8 L/min., different inlet coolant temperatures, different experimental times and different methods.

viscosity, shear stress, time, spiral flow and the chemical inhibitors, to study its effect on wax deposition process. Once an experiment is completed, the shutdown procedure for the experiment should be initiated. Before stopping the flow loop system, should make sure that all the data are monitored. The next step is to shut down the hot bath water that is responsible for heating the crude oil and then shut down the oil pump and after that shut down the chiller.

After completing the shutdown procedure, the sampling ports are opened to drain the remaining fluids in the test section. The main purpose in this step is to have a visual observation of the wax deposit and collect the wax samples to measure its volume. After this, the wax is scratched out from the pipe wall using a plastic conical and stored in a small cup to measure both of its volume and weight. After the scratched process, there is a small amounts of wax are still inside the wall of the test section, therefore, the chiller is used to melt all the remaining wax by increase the temperature of the recycled glycol-water mixture to 60°C. Then clean the test section from the remaining deposits by pigging it many times and prepare it for the next experiment.

During this study, four different techniques were used to estimate the wax deposit thickness including pigging, pressure drop, heat transfer and liquid displacement-level detection technique. The details and the procedure of those four techniques are illustrated in the next section.

The following steps were implemented to investigate its influence on wax deposition process, including:

• Investigation the effect of the different inlet coolant temperatures 14, 24, and 33°C on wax deposition at flow rate 2.7 L/min and oil temperature 44.5°C.

• Investigation the effect of the different inlet coolant temperatures 14, 24, and 33°C on wax deposition at flow rate 4.8 L/min and oil temperature 44.5°C.

• Evaluation the effect of the different flow rates 2.7 and 4.8 L/min on wax deposition at inlet coolant temperature 14 °C and oil temperature 44.5°C.

• Examine the effect of the flow rates 2.7 and 4.8 L/min on wax deposition at pipe wall temperature 24 °C and oil temperature 44.5°C.

• Estimate the effect of the various flow rates 2.7 and 4.8 L/min on wax deposition at inlet coolant temperature 33 °C and oil temperature 44.5°C.

• Studying the influence of the chemical inhibitor polyacrylate polymer W802 at concentrations 500, 1000, and 2000ppm and the influence of spiral flow on wax deposition process.

• Studying the effect of merging spiral flow with the inhibitor at different flow rates 2.7 and 4.8 L/min and different pipe wall temperatures 14, 24, and 33°C on wax deposition.

• Carry out all the steps above at different experimental time 2, 3, 4, 5, and 6 hours.

Techniques used to Estimate Wax Thickness

During this work, four different techniques are used to estimate wax deposit thickness. These techniques are including pressure drop, pigging, heat transfer, and liquid displacement-level detection technique (LD-LD).

Pigging technique

The pigging technique is a procedure followed to measure wax deposit thickness inside the test section. The concept of this technique is pass a sphere through the pipe and measuring the wax volume removed [1,2,6]. A plastic conical was used during this study instead of the sphere to pig the deposit wax. This technique is providing a clear visual examination and it is still widely used in the wax deposit in low pressure and single phase flow.

Pressure drop technique

This technique is considered an online technique due to it does not require depressurisation and restart to obtain wax thickness measurement [6]. This procedure was used in this study to estimate wax thickness inside the test section depending on the concept of increasing the pressure drop along the test section due to reducing the hydraulic diameter because of wax deposition [1,2,6,7]. The wax thickness can be calculated accurately from wax equation (3) presented by Chen, Theyab and Diaz [1,2,6].

$$\left(d_i-2\delta_w\right)^{5-n}=\frac{2c\rho L}{\Delta p_f}\left(\frac{\mu}{\rho}\right)^n\left(\frac{4Q}{\pi}\right)^{2-n} \quad (3)$$

Where ΔP_f is the pressure drop, L is the pipe length, d_i is the pipe diameter, δ_w is the wax thickness, Q is the volumetric flow rate, ρ is the crude oil density, μ is the viscosity of the crude oil. c = 16, n = 1 for laminar flow and c = 0.046, n = 0.2 for turbulent flow. Laminar flow exists when NRe <2000.

Heat transfer technique

Once the deposition occurred on the inner pipe wall surface, a convective heat transfer will apply between the crude oil and the layer of wax. A thermal resistance term due to heat conduction through the wax layer is added to the total resistance to heat transfer from the flowing fluid to the environment. The following heat transfer equation can be used to estimate wax deposit thickness [1,2,6].

$$\frac{T_f-T_o}{q_o}=\frac{1}{h_w}\frac{r_o}{r_i-\delta_w}+\frac{r_o}{k_w}ln\frac{r_i}{r_i-\delta_w}+\frac{r_o}{k_p}ln\frac{r_o}{r_i} \quad (4)$$

Where T_f is the fluid temperature in the pipe, T_o is the outside pipe wall temperature, q_0 is the heat flux through the outside pipe wall, r_o and r_i and are the outside and inside diameters of the pipe respectively, h_w is

the film heat transfer coefficient from the flowing fluid to the wax layer, k_p and k_w are the thermal conductivities of the pipe wall and deposited wax respectively, and δ_w is the thickness of the wax layer. r_i, r_o and k_p are usually known for a given pipeline. k_w can be assumed to be equal to the thermal conductivity of waxy crude oil since wax thermal conductivity is very close to that of waxy crude oil and a significant amount of oil is usually trapped in the wax deposits. h_w can be estimated using an appropriate correlation or model. Thus, the wax thickness can be calculated from equation (4) when the bulk oil is temperature T_f, the outside pipe wall is temperature T_o, and the heat flux through the outside pipe wall q_o are measured. q_o can also be obtained from a heat balance. The heat lost from the fluids over a length of pipe must equal the heat transfer to the surroundings. Thus,

$$C_p \rho Q \Delta T_f = 2\pi r_o L q_o \quad (5)$$

Where ΔT_f is the oil temperature drop over the wax measurement pipe section and C_p is the specific heat of the waxy oil.

Liquid Displacement-level Detection Technique (LD-LD)

This technique allows estimating the wax deposit volume by measuring the volume difference of the test section before and after wax deposit inside the pipe [1,2,6,7]. During this work, the test section filled with water before carrying out the experiment to measure the pipe volume when its clean then measure it after wax deposit in the pipe. Therefore, the deposit wax volume can be measured from the difference between the two water volumes.

This technique cannot be suitable to measure the soft or thin wax deposits where, the residual oil layer on top of the deposit can further increase measurement error.

Uncertainty, Standard Deviation (SD), and Reproducibility of Experimental Data

Uncertainty

The experimental uncertainty analysis can be defined as a technique that analyses a derived quantity, based on the uncertainties in the experimentally measured quantities that are used in some form of mathematical relationship (model) to calculate that derived quantity [8].

The uncertainty can be calculated from the following equation:

Uncertainity = (Maximum Value – Minimum value / 2) (6)

Standard deviation (SD)

Standard deviation is a measure that is used to quantify the amount of variation or dispersion of a set of data values. A low standard deviation indicates that the data points tend to be close to the mean of the set, while a high standard deviation indicates that the data points are spread out over a wider range of values [9].

The standard deviation can be calculated from the following equation:

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}} \quad (7)$$

Where, s is the standard deviation, x_i is the test result value, $\bar{x}$ is the average value of the results, and n is the number of the test results. The average value of the test results can be calculated from the equation bellow [10].

$$\bar{x} = \frac{\sum x_i}{n} \quad (8)$$

For example: To calculate the uncertainty using equation (6) and standard deviation using equation (7) and (8) of the experimental results of wax thicknesses (1.5, 1.61, 1.3, 1.5) using different measurement methods, can be calculated as follow:

Uncertainty = (1.61-1.3)/2 = ± 0.16

To calculate the standard deviation, should at first estimate the average value of the results.

The average value= $\bar{x} = \frac{\sum x_i}{n}$ (8) = (1.5+1.61+1.3+1.5)/4 = 1.48 mm

Calculate $\sum (x_i - \bar{x})^2$ for each value

$(1.5 - 1.48)^2$ = 0.0004 mm

$(1.61-1.48)^2$ = 0.0169 mm

$(1.3-1.48)^2$ = 0.0324 mm

$(1.5-1.48)^2$ = 0.0004 mm

$\sum (x_i - \bar{x})^2$ = 0.0004+0.0169+0.0324+0.0004= 0.0501 mm

The standard deviation= $s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$

$$s = \sqrt{\frac{0.0501}{4-1}} = 0.129 mm$$

Reproducibility

Reproducibility is the ability of an entire analysis of an experiment or sudy to be duplicated, either by the same researcher or by someone else working independently, whereas reproducing an experiment is called replicating it. Reproducibility and replicability together are among the main principles of the scientific method [11,12]. The results of wax thicknesses demonstrates a high degree of reproducibility of the test data for the different measurement methods conducted under the same operating conditions (Table 4).

Results and Discussion

Wax deposit volume

In order to study the wax deposition process many experiments were carried out during this work. The impact of various factors on the wax deposition process were studied, for instance the pipe wall temperature 14, 24 and 33°C, circulating time of oil at 2, 3, 4, 5 and 6 hours and flow rate 2.7 and 4.8 L/min.

Eight different experiment sets were applied to the above conditions, as shown in Tables 5-7. Seven of these were mitigation methods,

Variables		Wax Volume (ml) at Q=2.7 L/min					Wax Volume (ml) at Q=4.8 L/min			
Experiment at 14°C	At 2 hrs	At 3 hrs	At 4 hrs	At 5 hrs	At 6 hrs	At 2 hrs	At 3 hrs	At 4 hrs	At 5 hrs	At 6 hrs
Crude Oil	120	122	125	124	130	85	87	90	94	100
500 ppm W802	102	102	103	102	103	76	76	77	75	76
1000 ppm W802	70	72	74	77	80	62	63	65	64	67
2000 ppm W802	70	71	73	73	75	60	60	63	63	65
Oil + Spiral Flow	40	40	42	43	43	23	25	25	27	27
500 ppm + Spiral Flow	36	37	37	36	37	21	20	22	19	20
1000 ppm + Spiral Flow	30	31	33	34	35	16	18	18	17	18
2000 ppm + Spiral Flow	30	30	31	32	33	14	16	16	17	17

Table 4: The wax deposit volume at pipe wall temperature of 14°C.

including the commercial inhibitor W802 method at a concentration of 500 ppm, the commercial inhibitor W802 method at a concentration of 1000 ppm, the commercial inhibitor W802 method at a concentration of 2000 ppm, the new spiral flow method, the new method of bending the spiral flow with the inhibitor W802 at a concentration of 500 ppm and 1000 ppm respectively, and finally, the method of bending the spiral flow with the inhibitor W802 at a concentration of 2000ppm. These included a set of experiments ran at different flow rates, different pipe wall temperature and different aging time.

Table 5 shows the wax deposit volumes at pipe wall temperature 14°C for each experiment set. At the flow rate of 2.7 L/min, the maximum volumes of wax were found by carrying out the experiment using just crude oil at a different experimental time, while the minimum volumes of wax were found by carrying out the experiment using the effect of bending spiral flow with 2000ppm of polyacrylate polymer (C16-C22).

The wax volume reduced more than 67% while using spiral flow and it reduced by about 75% afterward merging spiral fow with the chemical inhibitor at a concentration of 1000ppm, and using the effect of the spiral flow with the inhibitor at a concentration of 2000ppm. Changing the flow rate to 4.8 L/min leds to reducing wax deposit in the pipe because of increasing the effect of shear stress.

Variables	Wax Volume (ml) at Q=2.7 L/min					Wax Volume (ml) at Q=4.8 L/min				
Method at 24°C	At 2 hrs	At 3 hrs	At 4 hrs	At 5 hrs	At 6 hrs	At 2 hrs	At 3 hrs	At 4 hrs	At 5 hrs	At 6 hrs
Crude Oil	83	83	87	89	89	70	74	75	75	77
500 ppmm W802	63	63	65	64	64	50	49	52	52	51
1000 ppm W802	45	47	47	46	50	30	31	32	33	33
2000 ppm W802	42	43	43	42	44	28	30	30	29	31
Oil + Spiral Flow	27	27	29	28	26	14	13	15	14	15
500 ppm + Spiral Flow	22	22	23	22	22	9	8	10	9	9
1000 ppm + Spiral Flow	7	8	8	9	9	4	7	6	8	7
2000 ppm + Spiral Flow	6.5	7	7	8	8	4	4	5	4	6

Table 5: Wax deposit volume in different experiment sets, at inlet coolant temperature 24°C.

Variables	Wax Volume (ml) at Q=2.7 L/min					Wax Volume (ml) at Q=4.8 L/min				
Method at 33°C	At 2 hrs	At 3 hrs	At 4 hrs	At 5 hrs	At 6 hrs	At 2 hrs	At 3 hrs	At 4 hrs	At 5 hrs	At 6 hrs
Crude Oil	25	27	28	28	30	18	17	19	18	20
500 ppm W802	20	23	22	22	23	15	14	16	15	15
1000 ppm W802	0	0	0	0	0	0	0	0	0	0
2000 ppm W802	0	0	0	0	0	0	0	0	0	0
Oil + Spiral Flow	5	7	7	6	8	6	5	7	7	8
500 ppm + Spiral Flow	1	2	2	1	2	0	0	0	0	0
1000 ppm + Spiral Flow	0	0	0	0	0	0	0	0	0	0
2000 ppm + Spiral Flow	0	0	0	0	0	0	0	0	0	0

Table 6: The wax deposit volume at pipe wall temperature 33°C.

The results in Table 4 show that the wax deposit volumes are very close to each other after using the the chemical inhibitor at a concentration of 1000ppm and 2000ppm.

Table 5 illustrates the effect of the eight experiment sets on wax deposit volume at an inlet coolant temperature of 24°C, at different time and flow rates.

The wax deposit volume decreased by increasing the pipe wall temperature due to an increase in dissolving the wax molecules and reducing the oil viscosity. It could be seen that in general the wax volume reduced and this was highest with the method of using just crude oil and lowest when using the effect of bending the spiral flow with the inhibitor at 2000ppm.

Table 6 presents the wax volume deposit on the pipe wall surface at an inlet coolant temperature of 33°C, at flow rate 2.7 and 4.8 L/min, and at different aging times. The wax volume was 25 ml from running the experiment using just crude oil at 2.7 L/min for 2 hours; this reduced to 18 ml at a flow rate of 4.8 L/min for the same experimental time. The wax deposit inhibition was 100% while using the inhibitor at a concentration of 1000 and 2000ppm and a flow rate 2.7 and 4.8 L/min respectively for the different experimental times. However, the inhibition of wax deposition was 20% at concentration 500ppm of the inhibitor at flow rate 2.7 L/min, and reduced to about 23% at flow rate 4.8 L/min.

The wax deposit inhibition was 100% after bending spiral flow with the inhibitor W802 at a concentration of 1000 ppm and 2000 ppm at different time and flow rates. The reduction in wax deposition was 100% after using the effect of bending the spiral flow with the inhibitor W802 at a concentration of 500ppm at flow rate 4.8 L/min, and the reduction in wax deposition was 94% at the same concentration and flow rate 2.7 L/min.

From the above three tables, it can be concluded that the spiral flow method and the spiral flow with the 1000 ppm W802 method are the best economical methods to reduce the wax deposit mitigation cost. Therefore, these two methods will be illustrated in detail, comparing them with the method of carrying out the experiments with just crude oil.

The reproducibility degree of each value in the three tables above was in the high level under the same operating conditions due to the uncertainty average of each value is about ±1 ml at different sets of experiments; and the average standard deviation of each value is about 1.41 ml.

Techniques for measuring wax thickness

Four different techniques were used during this work to estimate wax deposit thickness inside the test section of the pipe including pressure drop, pigging, heat transfer and liquid displacement-level detection technique.

Tables 7 and 8, shows the results of wax deposit volumes estimated using the previous four techniques at different flow rates. There was a large correspondence in the results of wax volumes by using the pigging technique and the LD-LD; this illustrates the validity of the experimental method to estimate the wax thickness. At the high pressure drop, the results of wax volumes were nearly similar by using the pressure drop and heat transfer techniques and they are slightly different at the lower pressure drop at the same flow rate.

Table 7, shows that at the lower flow rate and temperature the wax thickness was the highest and as a result the pressure drop increased.

Inlet Coolant Temperature	14ºC	24ºC	33ºC	40ºC
Pressure Drop(Pa)	1200	1000	900	600
Exp. Wax Volume (ml)	125	83	19	0
Wax Volume (ml) LD-LD	126	83	20	0
δ_w mm (Pigging Method)	1.82	1.5	0.7	0
δ_w mm (Pressure Drop)	1.83	1.61	0.69	0.04
δ_w mm (Heat Transfer)	1.83	1.3	0.71	0.06
δ_w mm (LD-LD)	1.84	1.5	0.73	0
Uncertainty mm	± 0.01	± 0.16	± 0.02	± 0.03
Standard Deviation (SD) mm	0.01	0.129	0.017	0.03

Table 7: Estimating wax thickness (δ_w) using the different techniques at flow rate 2.7 L/min.

Inlet Coolant Temperature	14ºC	24ºC	33ºC	40ºC
Pressure Drop (Pa)	3000	2700	2100	1200
Exp. Wax Volume(ml)	85	70	15	0
Wax Volume (ml) LD-LD	87	70	17	0
δ_w mm (Pigging Method)	1.5	1.36	0.63	0
δ_w mm (Pressure Drop)	1.72	1.45	0.79	0.054
δ_w mm (Heat Transfer)	1.72	1.27	0.65	0
δ_w mm (LD-LD)	1.52	1.36	0.67	0
Uncertainty mm	± 0.11	± 0.09	± 0.08	± 0.03
Standard Deviation (SD) mm	0.122	0.073	0.072	0.03

Table 8: The wax thickness (δ_w) resulting from the different four techniques at flow rate 4.8 L/min.

Overall, the wax thickness was about 1 m, 83 mm at pipe wall temperature of which is higher than that of 24 and 33ºC, respectively; by running the experiments using just crude oil at flow rate 2.7 L/min. This indicates that the inlet coolant temperature has the main effect on wax deposition process, and the four different methods to estimate the wax thickness shows agreement between them.

The uncertainty range of the results arise between (0.01 - 0.03mm) at different pipe wall temperature (14, 24, 33, 1nd 40ºC). The standard deviation range is between (0.01 – 0.03mm). The results of wax thicknesses demonstrate a high degree of reproducibility of the test data for the four different measurement methods conducted under the same operating conditions.

Table 8 illustrates that increasing the flow rate to 4.8 L/min leads to increase the shear stress and that was obvious on wax deposit thickness. Overall, it was noticed that the highest wax deposit thickness was at the lower inlet coolant temperature; and it was reduced by increasing the inlet coolant temperature to 24ºC and 33ºC, by implementation the experiments with just crude oil. The results of wax thicknesses demonstrate a high degree of reproducibility of the test data for the four different measurement methods conducted under the same operating conditions.

The reproducibility degree of the four different measurement methods was in the high level under the same operating conditions due to the results uncertainty range is between (0.11 - 0.03 mm) at different pipe wall temperature (14ºC, 24ºC, 33ºC, and 40ºC); and the standard deviation range is between (0.122 – 0.03 mm).

Conclusion

In the cold environments, flowing the crude oil through the transfer pipelines leads to loss heat to the surrounding and as a result a solid phase will precipitate on the pipe wall when its temperature drops below the WAT. This solid phase leads to limit the crude oil flow in the pipeline, creating pressure abnormalities, and this blockage leading to reduce the crude oil production. The current work presented in details the methodology followed to estimate wax deposition volume and the mitigation methods to eliminate wax deposition. It presented the factors that influence on wax deposition process such as pipe wall temperature, chemical inhibitor and spiral flow. During this work, four different techniques were used to evaluate the wax thickness excluding pigging, pressure drop, heat transfer and LD-LD technique. Two mitigation methods had been used to reduce wax deposition including the chemical inhibitor polyacrylate polymer and spiral flow. The experimental results shows that at lower pipe wall temperatures the wax deposit was the highest and stopped above WAT.

The results illustrated that the reduction in wax deposition was 100% after using the influence of bending spiral flow with polyacrylate polymer W802 at a concentration of 1000 ppm and 2000 ppm at different time and flow rates. The reduction in wax deposition was 100% after using the effect of bending the spiral flow with the inhibitor W802 at a concentration of 500 ppm at flow rate 4.8 L/min, and the reduction in wax deposition was 94% at the same concentration and flow rate 2.7 L/min.

The reproducibility degree of the four different measurement methods was in the high level under the same operating conditions due to the results uncertainty range is between (0.11 - 0.03 mm) at different inlet coolant temperature (14ºC, 24ºC, 33ºC, and 40ºC); and the standard deviation range is between (0.122 – 0.03 mm).

In conclusion, this study presented in details the methodology used to study and to evaluate wax deposition process in the hydrocarbon pipeline and the mitigation methods implemented to reduce or eliminate wax deposition.

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