

**Application of Local Materials for Treatment of Waxy Crude in Some Niger Delta Fields**

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| **Article information** |  | **Abstract** |
| *Article History**Received 25 April 2024**Revised 19 May 2024**Accepted 30 May 2024**Available online 2 June 2024* |  | *It has been reported that 70% of Nigerian Oilfields are affected by wax formation issues which cause problems in flow of crude oil. Unfortunately, there are key knowledge gaps that make it difficult to predict and manage these issues, resulting in negative impact on production, revenue and energy security. To proffer solution to this problem, a study was conducted to investigate the crystallization behavior of waxy crude oil and the effectiveness of various wax inhibitors in mitigating wax related issues. The study analyzed crude oil samples from identified waxy wells in Niger Delta Oilfields using standard methods. This involved characterization of the crude oil through API Gravity measurement, SARA Analysis, Cloud and Pour Point determination, wax content measurement, viscosity/temperature profiling and compositional analysis. The work explored methods for treating wax crude oil, including extraction of orange peel oil and soya bean husk. In order to ensure accuracy and reliability of the results, the study took rigorous experimental precautions. The efficacy of various wax inhibitors including orange peel oil and soya bean husk oil was evaluated through performance testing on crude oil samples at different concentrations. The findings obtained from this study contribute to the optimization of wax inhibition strategies and offers practical solutions for mitigating wax depositional issues in petroleum production and transportation systems* |
| *Keywords:* *Wax, Temperature, Crude, dynamic viscosity, orange peel oil, soya bean husk oil.*[https://doi.org/10.5281/zenodo.](https://doi.org/10.5281/zenodo.10431883)11421020 **https://nipes.org****© 2024 NIPES Pub. All rights reserved** |  |

1. **Introduction**

Crude oil is a complex, multi-component organic mixture primarily composed of hydrocarbons with a few non-hydrocarbon constituents. Its overall composition can be classified into distinct groups, including saturates, aromatics, resins, and asphaltenes [1]. Within the saturate fraction, certain compounds possess elevated melting points, making them susceptible to solidifying at lower temperatures. As the temperature decreases, these compounds undergo precipitation from the fluid, resulting in the formation of colloidal structures known as wax. The primary contributors to wax formation are predominantly normal and branched alkanes, although some naphthene attached to long carbon chains can also participate in the wax formation process [1].

Temperature plays a crucial role in wax solubility in crude oil, and it is used to quantify solubility under specific conditions. Changes in temperature, moving upward and downward, influence wax precipitation by affecting the solubility limit. Additionally, other factors, such as the composition of the oil and the gas available for solution, the pressure of the oil impacting the gas in solution, the flow rate, completion, and the roughness of the deposition surface, all contribute to the deposition process by creating an environment conducive to deposition [2].

The onset temperature denoting initial wax precipitation is termed the wax appearance temperature (WAT). Tracking variations in indigenous Wax Appearance Temperature WAT values across regional crudes provides insight enabling better mitigation strategies. However, with over 70% of Nigerian oilfields affected by wax issues, persisting knowledge gaps around WAT prediction constrain proactive management of these flow issues which take a heavy toll on productivity and revenue [3].

The chemical composition of waxy crude oil primarily consists of paraffins, which are saturated hydrocarbons, also known as alkanes. Paraffins can exist as straight-chain alkanes (normal alkanes) or branched-chain alkanes (iso-paraffins), but they do not contain any ring structures. The general formula for paraffins is CnH2n+2, where "C" represents a carbon atom, "H" represents a hydrogen atom, and "n" is an integer. Paraffins typically consist of long-chain hydrocarbons with carbon chain lengths ranging from 18 to 30 or even more.

When the inner wall temperature of oil drops below the Wax Appearance Temperature (WAT), paraffinic components in crude oil, including high molecular weight alkanes with carbon numbers above 20, precipitate and deposit wax on it. Although wax buildup during oil flow reduces flow, difficulties might arise when production is halted and wax precipitation obstructs flow [4]. Wax molecules precipitate out of the liquid phase and remain static when pipeline transport is halted due to planned maintenance or emergency conditions, such as severe weather conditions on an offshore platform [5].

When waxy oil flowing in cold lines is cooled, a network of wax crystals are formed, causing the oil to gel. The wax crystals have a strong contact and affinity, which leads to the creation of the network, in contrast to inorganic solutions where there is little interaction between the salt crystals [6]. Despite having comparable chemical properties, wax and oil have very different molecular weights. Waxes tend to generate stable wax crystals that interlock to create a strong network and have a greater molecular weight. A significant amount of oil is captured by the network of wax [7]. Consequently, the creation of a gel layer with a significant amount of trapped oil is the initial stage of the deposition of waxy oil blend on a cold surface and the severity of wax issues can range from slight to uncontrollable.

Utilizing a cold finger device, Patton and Casad [8] conducted a study on wax deposition at lower temperatures, keeping the coolant temperature below the pour point temperature. Their findings revealed that the thickness and volume of the wax deposit increased as the temperature differential dropped. [8]

Similarly, Jennings and Newberry [9] investigated wax deposition at various coolant temperatures with a constant bulk oil temperature, using a crude oil sample from the Gulf of Mexico in a cold finger apparatus. Their results indicated that the wax coating decreased with each rise in coolant temperature. The study also demonstrated a correlation between a drop in coolant temperature and an increase in wax deposit and the proportion of trapped oil in the deposit [9]. Accumulation of wax in tubing and flow lines poses a prevalent flow assurance challenge, rendering the economic viability of transporting waxy crudes questionable. Addressing this challenge requires enhancing oil mobility and reducing viscosity, achievable through various methods such as heating, blending with lighter oils, or incorporating solvents to depress the pour point and crucial to ensuring oil mobility is the use of solvents that keep wax in solution [9]. Theyab [10] cited Mechanical and Chemical removal methods, among the oldest in the industry, are employed for wax removal and utilize various techniques such as rod scrapers, wireline scrapers, flow line scrapers, free-floating piston scrapers (commonly used in gas lift wells), pigging flow lines, etc. The hot fluid method, widely employed in oil fields, involves injecting hot oil or hot water at temperatures ranging from approximately 65 to 150°C down the well tubing or casing to melt waxes causing restrictions in downhole equipment. However, challenges may still arise due to added chemicals that disperse the wax without diluting or dissolving paraffin. Chemical techniques are considered the most cost-effective and efficient approach to prevent wax deposition [11]. These methods utilize specially formulated chemicals that either dissolve the deposited paraffin and asphaltene or hinder the deposition process by preventing agglomeration and cluster formation. Although these chemicals may not completely eliminate deposition, they significantly improve cleaning efficiency, whether using mechanical or thermal methods. As a result, more research is required to identify the most appropriate chemical solvents, taking into account both their effectiveness and the associated economic feasibility.

The purpose of this research is to use local materials to treat oil wells producing waxy crude thereby developing local content, boosting oil production and increasing foreign exchange for the nation.

1. **Materials and Method**

Two (2) crude oil samples of about 500milliliter (500ml) were selected to study the effects of the chemical and physical properties of crude oils on crystallization behavior of wax in the presence of inhibitor. The crude oil samples were received from two different identified waxy wells located in an Oilfield in Western Delta, Niger Delta. Basic properties of these crude oils were determined using standard methods, including Wax appearance temperature (WAT) of crude oil samples wax which was measured by using ASTM D2500, the wax content was measured using UOP 46-64 method and pour point was measured using ASTM D5853.



Figure 1: Crude oil samples**.**

**2.1 Operational Sequence of Treating Waxing Crude**

**2.1.1 Extraction of Orange Peel and Soya Bean Husk Oil**

800 kg of orange peels and the soya bean husks were sourced from Port Harcourt in Rivers State, Nigeria and was dried in the sun. It took 7 days to properly dry the samples. After which, the samples were ground using a grinding machine. 80 g of the ground orange peel and soya bean husk were enfolded in a filter paper and put into the extraction chamber of the sox let respectively, for effective and efficient extraction of the oil from the ground orange peel and soya bean husk oil, n-hexane (400 ml) was added through the top of the condenser with the aid of a funnel, the solvent passed through the condenser to the extraction chamber and lastly resolves at the round bottom flask with the anti-bumping. Next the set-up was inspected and then the heating mantle (water bath) was put on and set at 70℃, and the mantle was set to n-hexanes boiling point.

As the n-hexane begins to boil, there is evaporation of the n-hexane from the round bottom flask through the extraction chamber which contains the sample and lastly the condenser containing the water that cools the system traps the n-hexane and condenses it and it drops as liquid back into the extraction chamber and as the liquid n-hexane increases in volume in the extraction chambers it reaches the siphon point and then siphon back into the round bottom flask, this process continues until the n-hexane in the extraction chamber becomes colorless, which is an indication that the oil in the sample has been extracted and the separated oil is put into the rotary evaporator to gently and efficiently remove the solvent from the sample by evaporation at reduced pressure. This process was repeated until the required volume of the essential oil was obtained from the extraction process.



Figure 2: Soya bean husk oil and orange peel orange oil, and schematic of soxhlet extractor

### **2.1.2 Wax Content**

### The standard acetone method (UOP 46-64 method) was used to take out the wax from the crude oil sample. A sample of the crude oil was measured and weighed into a 100 ml beaker. Then 25 ml of Toluene was added to the sample and stirred for 5 minutes. Again, 5g of fuller’s earth (aluminum silicate) was weighed and added to the mixture to make the sample clear of all polar materials in the oil. Subsequently, the toluene was removed from the sample by evaporation in the oven at 45°C. Then, the deposit was dissolved in ether-acetone mixture in a ratio of 3:1 and was put into a freezer at -17°C for two hours. Then, the solution was filtered through a filter paper which was already weighed. The filtrate (precipitated wax) on the filter paper was dried in an oven at 45°C. The wax content was determined using the formula below:

$$Wax Content= \frac{\left(weight of filter paper+precipitated wax\right)-weight of empty filter paper}{weight of oil measured}$$

**2.1.3 Viscosity/Temperature Profile**

A Cannon Fenske Viscometer was used to measure the kinematic viscosity of the crude oil with and without inhibitors at a cooling range from 60°C down to 30°C. For each occasion, the value of the kinematic viscosity was determined after the crude oil with or without inhibitors flowed from the top of the graduated red line to the bottom of the graduated red line. The density of the crude oil with and without inhibitors was determined by the aid of a density bottle and an electronic weighing balance at varying temperature of the crude oil with and without inhibitors and the dynamic viscosity was computed by multiplying the kinematic viscosity with a cannon Fenske viscometer constant with the density of the crude oil with and without inhibitors at various temperature ranges considered.

The data for the crude oil samples with inhibitors should then be compared to the crude oil without inhibitors (blank) results, and the inhibitor that maintains the flattest, smoothest trace for the longest time is considered the most effective wax inhibitor.

**2.1.4 Wax Appearance Temperature (WAT)**

The cannon Fenske viscometer was used to measure the viscosity of the crude oil with and without inhibitor at different temperatures. The cloud point of the crude oil was estimated by plotting viscosity values versus reciprocal temperature. The WAT was determined from the converted point in the viscosity curve from the straight line to the incline line, at which the viscosity start to increase gradually when the temperature is decreased.

**2.2 Performance Evaluation of Soya Bean Husk Oil and Orange Peel Oil as Wax Inhibitors on Waxy Crude Oil**

10 ml of the crude oil sample was poured into 27 different sample bottles, which was then dosed with individual and combined pour-point depressants using micro-syringe at dosage concentration 2100 ppm. The effect of the inhibitor on the sample of crude-oil pour point and viscosity was evaluated.

1. **Results and Discussions**
	1. **Wax Analysis**

Table1: Wax Analysis of Crude Oil

|  |  |  |  |
| --- | --- | --- | --- |
| **Sample** | **WAT (oC)** | **Wax Content (%)** | **Pour Point (oC)** |
| Field X | -18.19 | 0.0650 | 9 |
| Field Y | 24.59 | 0.6549 | 18 |

From the Table 1, Wax Appearance Temperature (WAT) of crude oil samples was measured by using ASTM D2500, wax content was measured using UOP 46-64 method and pour point was measured using ASTM D5853. From the results obtained, there appears to be a direct relationship between the wax content, pour point and wax appearance temperature. This indicates that crude oil from Field X is less prone to wax precipitation at higher temperatures than Field Y. Lower WAT values are generally favorable for crude oil as they suggest that wax formation occurs at lower temperatures, reducing the risk of wax-related issues during production and transportation. Field X has a lower WAT (-18.19 °C) compared to Field Y (24.59 °C). Field X has a lower pour point (9 °C) compared to Field Y (18 °C). Field Y has a significantly higher wax content (0.6549%) compared to Field X (0.065%). Higher wax content can lead to increased wax deposition in pipelines and equipment, potentially causing operational challenges. The pour point is the lowest temperature at which the oil can still flow. Lower pour points are advantageous as they indicate that the crude oil from Field X can flow at lower temperatures, making it less likely to experience flow restrictions due to cold weather.

Table 2: Characteristic property of waxy crude oil sample

|  |  |
| --- | --- |
| **Waxy crude oil sample** | **Wax content (%)** |
| X | 7 |
| Y | 65 |

From the data in Table 2, Field Y has a significantly higher wax content (0.6549%) compared to Field X (0.065%). The data obtained from literature has more suitable wax content for crude oil than the current values used in this study. This indicates that the wax content of oil sample Y has more tendencies for wax precipitation than sample X.

Table 3: Viscosity (cp)/Temperature (˚F) for crude oil sample X without wax inhibitor

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temp (°F)** | **Kinematic Viscosity (mm²/s) of crude oil sample** | **Cannon fenske viscosity constants** | **Density (kg /m³)** | **Dynamic viscosity (cp)** |
| 140 | 7.11 | 0.03633703 | 880 | 0.256 |
| 131 | 7.58 | 0.03634959 | 880 | 0.273 |
| 122 | 8.25 | 0.03636216 | 880 | 0.297000 |
| 113 | 9.3 | 0.03637472 | 880 | 0.335 |
| 104 | 9.58 | 0.03638728 | 880 | 0.346000 |
| 95 | 13.59 | 0.03639985 | 880 | 0.489 |

Results from Table 3 above shows the data of dynamic viscosity and temperature of the waxy crude oil sample X without wax inhibitor. The WAT was determined from the converted point in which the viscosity started to increase gradually when the temperature was decreased and this point is at 132˚ F.

Table 4: Viscosity (cp)/Temperature (˚F) for crude oil sample Y without wax inhibitor

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temp (˚F)** | **Kinematic Viscosity (mm2/s) of crude oil sample** | **Cannon fenske viscosity constants** | **Density (kg/m3)** | **Dynamic viscosity (cp)** |
| 140 | 7.08 | 0.03633701 | 885 | 0.255 |
| 131 | 7.38 | 0.03639594 | 885 | 0.266 |
| 122 | 8.05 | 0.03636157 | 885 | 0.29 |
| 113 | 9.1 | 0.03637472 | 885 | 0.328 |
| 104 | 9.38 | 0.036387283 | 885 | 0.339 |
| 95 | 13.39 | 0.036399846 | 885 | 0.483 |

From Table 4 above, the result of dynamic viscosity and temperature of the waxy crude oil sample Y without wax inhibitor could be seen. The WAT was determined from the converted point in which the viscosity started to increase gradually when the temperature was decreased and this point was at 135˚F.

**3.2 Treatment Results**

Table 5: Viscosity (cp)/Temperature (˚F) for soya bean husk oil (2100ppm) oil plus crude oil sample X.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temp (°F)** | **Kinematic Viscosity (mm²/s) of crude oil sample** | **Cannon fenske viscosity constants** | **Density (kg /m³)** | **Dynamic viscosity (cp)** |
| 140 | 6.55 | 0.036337 | 889 | 0.237 |
| 131 | 7.4 | 0.0363496 | 889 | 0.267 |
| 122 | 8.2 | 0.0363622 | 889 | 0.296 |
| 113 | 9.1 | 0.0363747 | 889 | 0.329 |
| 104 | 9.45 | 0.0363873 | 889 | 0.342 |
| 95 | 13.44 | 0.0363999 | 889 | 0.486 |

Table 5 above shows the result of dynamic viscosity and temperature of the waxy crude oil sample X plus SBH. The WAT was determined from the converted point in which the viscosity started to increase gradually when the temperature was decreased and this point is at 131˚F.

Table 6: Viscosity (cp)/Temperature (˚F) for soya bean husk (2100ppm) oil plus crude oil sample Y

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temp (°F)** | **Kinematic Viscosity (mm²/s) of crude oil sample** | **Cannon fenske viscosity constants** | **Density (kg /m³)** | **Dynamic viscosity (cp)** |
| 140 | 7.05 | 0.036337 | 890 | 0.254 |
| 131 | 7.14 | 0.03635 | 890 | 0.257 |
| 122 | 8.12 | 0.036362 | 891 | 0.293 |
| 113 | 9.21 | 0.036375 | 891 | 0.3333 |
| 104 | 9.55 | 0.036387 | 891 | 0.345 |
| 95 | 13.54 | 0.0364 | 891 | 0.49 |

Results observed in Table 6 above showed values of dynamic viscosity and temperature of the waxy crude oil sample Y plus SBH. The WAT was determined from the converted point in which the viscosity started to increase gradually when the temperature was decreased and this point is at 130˚F.

Table 7: Viscosity (cp)/Temperature (˚F) for orange peel oil (2100ppm) plus crude oil sample X

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temp (˚F)** | **Kinematic Viscosity (mm2/s) of crude oil sample** | **Cannon fenske viscosity constants** | **Density (kg/m3)** | **Dynamic viscosity (cp)** |
| 140 | 6.45 | 0.03633703 | 889 | 0.232 |
| 131 | 7.25 | 0.03634959 | 889 | 0.261 |
| 122 | 7.5 | 0.03636216 | 889 | 0.271 |
| 113 | 8.14 | 0.03637472 | 889 | 0.294 |
| 104 | 8.47 | 0.03638728 | 889 | 0.306 |
| 95 | 11.02 | 0.03639985 | 889 | 0.398 |

Table 7 above shows the values of dynamic viscosity and temperature of the waxy crude oil sample X doped with OP. The WAT was determined from the converted point in which the viscosity started to increase gradually when the temperature was decreased and this point is at 122˚F.

Table 8: Viscosity (cp)/Temperature (˚F) for orange peel oil (2100ppm) plus crude oil sample Y

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temp (°F)** | **Kinematic Viscosity (mm²/s) of crude oil sample** | **Cannon fenske viscosity constants** | **Density (kg /m³)** | **Dynamic viscosity (cp)** |
| 140 | 6.25 | 0.03633703 | 889 | 0.225 |
| 131 | 7.05 | 0.03634959 | 889 | 0.254 |
| 122 | 7.3 | 0.03636216 | 889 | 0.263 |
| 113 | 7.58 | 0.03637472 | 889 | 0.273 |
| 104 | 8.27 | 0.03638728 | 889 | 0.299 |
| 95 | 10.45 | 0.03639985 | 889 | 0.377 |

The results in Table 8 show the values of dynamic viscosity and temperature of the waxy crude oil sample Y doped with OP. The WAT was determined from the converted point in which the viscosity started to increase gradually when the temperature was decreased and this point is at 122˚F.

Table 9: Viscosity (cp)/Temperature (˚F) for Toluene (2100 ppm) plus crude oil sample X

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temp (°F)** | **Kinematic Viscosity (mm²/s) of crude oil sample** | **Cannon fenske viscosity constants** | **Density (kg /m³)** | **Dynamic viscosity (cp)** |
| 140 | 7.08 | 0.03633703 | 893 | 0.253 |
| 131 | 7.4 | 0.03634959 | 893 | 0.26 |
| 122 | 8.02 | 0.03636216 | 893 | 0.29 |
| 113 | 9.05 | 0.03637472 | 893 | 0.327 |
| 104 | 9.3 | 0.03638728 | 893 | 0.337 |
| 95 | 13.5 | 0.03639985 | 893 | 0.471 |

Table 9 above shows the results of dynamic viscosity and temperature of the waxy crude oil sample X doped with T inhibitor. The WAT was determined from the converted point in which the viscosity started to increase gradually when the temperature was decreased and this point is at 130˚F.

Table 10: Viscosity (cp)/Temperature (˚F) for Toluene (2100 ppm) plus crude oil sample Y

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Temp (˚F)** | **Kinematic Viscosity (mm2/s) of crude oil sample** | **Cannon fenske viscosity constants** | **Density (kg/m3)** | **Dynamic viscosity (cp)** |
| 140 | 7 | 0.03633703 | 895 | 0.253 |
| 131 | 7.2 | 0.03634959 | 895 | 0.26 |
| 122 | 8.02 | 0.03636216 | 895 | 0.29 |
| 113 | 9.05 | 0.03637472 | 895 | 0.327 |
| 104 | 9.3 | 0.03638728 | 895 | 0.337 |
| 95 | 13 | 0.03639985 | 895 | 0.471 |

Table 10 above shows the values of dynamic viscosity and temperature of the waxy crude oil sample Y doped with Toluene inhibitor. The WAT was determined from the converted point in which the viscosity started to increase gradually when the temperature is decreased and this point is at 122˚F.

Also, from the results obtained, it is observed that crude oil from Field X and Field Y in the Niger Delta region reveals unique differences in composition of wax. The wax content of Field Y's crude oil exhibited higher than that of Field X, thus signifying the distinction of content of crude wax in different regions. The investigation into wax appearance temperature (WAT) and pour point values provided a detailed understanding of the waxy nature of crude oils from both fields. Field Y demonstrated higher pour point and WAT values, indicating a higher susceptibility to wax-related issues.

1. **Conclusion**

The comprehensive analysis of crude oils from Field X and Field Y in the Niger Delta region revealed distinct differences in wax composition. Field Y's crude oil exhibited significantly higher wax content, emphasizing the regional variability in crude wax composition. Innovative waxy crude oil inhibitors were formulated using locally sourced non-edible orange peel soya bean husk. These indigenous inhibitors showed promising results in inhibiting wax deposition. From result shown in Table 5-10, it could be seen that the wax-related properties of the samples X and Y treated with local inhibitors improved significantly when compared to the properties of the original samples X and Y without inhibitors in Table 3-4. This is favorable when compared to similar results of commercially viable inhibitors as cited by Kiyingi et al. [1].

The inhibitors, derived from orange peel and soya bean husk, were applied to waxy crude oil samples. From the results obtained, Field X appears to have crude oil with more favorable properties regarding wax management, including a lower WAT, lower wax content, and a lower pour point compared to Field Y. These characteristics can contribute to smoother oil production and transportation operations. The results indicated effective inhibition, highlighting the potential use of these locally sourced inhibitors as alternatives to traditional methods.

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