

Development of an Environmental-Friendly Wax Inhibitor for Crude Oil Transport in Oil and Gas Pipelines

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Abstract

Wax deposition has caused significant operational problems and economic losses for many oil and gas companies. Typically, crude oil is transported at conditions below Wax Appearance Temperature (WAT) causing wax to precipitate and build up leading to flow restriction and sometimes eventual pipe blockage. Wax inhibitors (WI) have been known to be a formidable solution to this problem. However, conventional wax inhibitors are known to have caused environmental and health concerns due to their toxicity. They could also be quite expensive therefore, the need to develop economically attractive and environmentally friendly WI. In this study, a wax inhibitor Methanol-Cellulose (WI-MC) was formulated from coconut husk bio raw material. The coconut husk was treated and prepared in the laboratory and applied at different dosages of 0.2ml, 0.6ml and 1.0ml to two different crude oil samples. The effect on WAT examined from viscosity readings obtained at varying temperature was compared with Toluene. Without inhibitor the WAT for samples A and B was 55°C. Comparative analysis however reveals WI-MC is comparable with Toluene. At 0.2ml dosage, WI-MC had no effect on WAT whereas at 0.6 and 1.0ml, the temperature-viscosity plot showed WAT lowered to 50°C. Similar result was also obtained for Toluene. This indicates WI-MC inhibitor can be considered a feasible and potentially cost-effective alternative for synthetic and imported WIs. The reduced dependence on such chemicals is an environmental advantage brought about by renewable and locally available materials.

Keywords: Wax Deposition; Wax Inhibitors; Coconut Husk, Wax Appearance Temperature

1.0 INTRODUCTION

The goal of flow assurance is to have smooth and efficient transport of hydrocarbons from the reservoir to the end users. However, one of the major flow assurance challenges is Wax deposition. This deposit usually clogs pipes, reducing flow rate and diameter. From various studies it's been reported that wax deposition is a huge problem that could cause production sites to shut down causing downtime which amounts to loss of money. In Lasmus oilfield in UK for example, wax deposit problem led to abandonment which cost over \$100million. This shows that wax deposition is a costly challenge (Nguyen *et al.*, 2001; Singh *et al.*, 2011). Wax from the crude oil typically consists of a variety of light and transitional hydrocarbons (paraffin's, aromatics, naphthenic, etc.) and diversity of other heavy organic (non-hydrocarbon) compounds, even though at very low concentrations including resins, asphaltenes, diamondoids, mercaptans, organo- metallic's, and so on.

Temperature difference is a crucial factor in wax formation and deposition due to its direct link to the solubility of paraffins. The temperature gradient between the pipe wall and the flowing oil plays a significant role in this process. Singh *et al.* (2011) explored the relationship between wax solubility and temperature, showing that as crude oil flows through pipelines, it loses heat to the surrounding environment, making the pipe wall cooler than the bulk oil. This temperature triggers wax precipitation. Erickson *et al.* (1993) reported that wax will not deposit unless the operating temperature drops to or below the Wax Appearance Temperature (WAT). Below WAT, waxy components in the oil begin to crystallize into tiny solids. A larger temperature difference thus accelerates and intensifies wax precipitation. The composition of the crude oil is another significant factor that influences wax deposition. Wax

components become less soluble and crystalline as temperature drops below the WAT. Solid crystals continue to grow and distribute themselves in the crude oil system resulting in viscosity build up thus, forming wax deposition on the pipeline walls. (Mojtaba *et al.* 2019, Mohammad, 2014, Priyank *et al.* 2013). Saturates, Aromatics, Resins, and Asphaltenes often referred to as the SARA fractions, play a crucial role. The proneness of crude oil to deposit wax solids on the wall of pipeline is often determined by SARA fractions and stability of the crude oil. Higher concentrations of paraffins, the main component of waxes, naturally lead to higher deposition potential because of their high flexibility. Iso-paraffins also have high chance of wax deposition but form more unstable waxes. The rate at which oil cools significantly impacts wax deposition. Pedersen *et al.* (1991) reported that rapid cooling leads to the formation of numerous small, poorly dispersed crystals. These smaller crystals have a larger surface area, making them more likely to adhere to the pipe wall, resulting in faster but less organized deposition. The study found that higher cooling rates corresponded to an increase in the rate of wax deposition. In contrast, slower cooling allows for the formation of fewer, larger crystals, which are more stable and tend to settle at the bottom of the pipe, potentially reducing deposition on the pipe walls. Flow regimes vary between pipelines and are a key factor in wax deposition. In laminar flow, the smooth, layered movement creates low shear forces, offering little resistance to wax crystals as they approach the pipeline wall. This allows the crystals to easily stick and accumulate, leading to wax deposition. In contrast, turbulent flow, with its chaotic eddies and higher shear forces, creates a dynamic environment that disrupts and displaces deposited wax crystals, reducing their buildup on the walls and thus delaying or minimizing overall deposition. Pipeline wall roughness can significantly affect the rate of wax deposition. Rougher walls offer more surface area and create additional crystallization sites where wax crystals can attach and grow, leading to faster and larger deposits. Conversely, smoother walls have less surface area and fewer crystallization sites, which can hinder deposition and make it more difficult for wax crystals to adhere. Lastly, the presence of water content in crude oil could also influence wax deposition. High water content, especially those with dissolved salts and minerals, can act as crystallization sites which dissolves solids, promote crystal growth and aggregation, ultimately accelerating deposition. Other researchers including Esmailzadeh and Mehranbod (2011); Adeyanju (2012) and Adeyanju and Oyekunle (2014) have investigated various aspects of wax formation and mitigation.

Over the years, companies have applied various techniques to mitigate and predict wax deposition from use of chemicals as inhibitors, mechanical techniques such as pigging and thermal technique. Most companies prefer and have adopted the use of chemicals inhibitors. Inhibitors added to the flow lines and down-hole have proven to be efficient in removing wax deposition, which solves the problem of precipitated wax, making it easier for the transport of the crude oil to the surface. In addition, there are different abilities of different wax inhibitors in dissolving wax deposits according to the properties of the wax itself, the temperature, and location in the system. In the oil industry today, there are several wax management methods that have been implemented that require ethical and technical practices to achieve the desired result. There are three major categories of wax prevention and mitigation methods which are thermal, mechanical and chemical. The thermal supplies heat into wells to melt the deposited wax for efficient crude transportation to the surface. This can be achieved by direct heating, indirect heating and using heated pigs. Pigging is a method commonly used to remove wax in the field. This is achieved mechanically by

launching a pipeline pig through the line, which scrapes off wax from its walls as it is forced along by the pressure of oil. However, this risks the potential for creation of a wax plug downstream from the pig as wax scraped off accumulates and is compressed ahead of the pig. If this occurs, the pipeline could be damaged and lost. Most oil companies prefer using chemical inhibitors to manage the economics of waxy crude oil production in cold environments, as they are considered crucial for reducing wax deposition rates in pipelines. The use of chemical wax inhibitors is advantageous because it does not require halting production to clean the pipes, serving as an effective mitigation strategy. However, the current application of inhibitors is not flawless. According to Ridzuan *et al.* (2014), there is no universal inhibitor suitable for all types of crude oil due to their varying properties, preventing a standardized approach.

Many studies have primarily concentrated on temperature-related issues and the comparison of conventional wax inhibitors, which often contain synthetic chemicals that can pose risks to workers and the environment. In addition to these health and environmental concerns, conventional wax inhibitors can be costly, making them less accessible for smaller companies or operations with limited budgets. However, Wax inhibitors made from locally sourced materials provide a sustainable solution for reducing wax deposition in oil and gas pipelines. Osokogwu *et al.* (2022) developed a local wax inhibitor from soya bean husk and orange peel to explore the effects of the inhibitor on wax appearance temperature. These inhibitors are cost-effective and can lower the carbon footprint associated with transportation and processing, enhancing environmental sustainability while being adaptable to specific conditions. Although some studies have explored the effectiveness of these natural inhibitors, there are still gaps in understanding their performance, economic feasibility, and scalability. This research focuses on developing wax inhibitors using coconut husks as raw material and will examine their effectiveness in inhibiting wax formation in waxy crude oil.

2.0 MATERIALS AND METHODOLOGY

The experiment was carried out with the use of two crude oil samples labeled sample A and sample B to test the effect of the prepared inhibitor. The method used in this work is based on the principle that as temperature decreases, crude oil viscosity generally increases and when wax starts to precipitate at WAT, the viscosity increases more sharply due to the formation of solid wax crystals. The method was adopted from the work of Dantas *et al.* (2009), Mohammad S.I (2014), Reidar *et al.* (2009), Osokogwu *et al.* (2022) in determining WAT. The determination of WAT was adapted from Osokogwu *et al.* (2022), Mohammad (2014), Dantas *et al.* (2009), Reidar *et al.* (2009).

2.1 Preparation and Processing of Coconut Husk

Fresh mature coconuts were harvested from a farm in Ogun State thoroughly washed with distilled water to remove dirt and debris, and the husks were separated. The preparation of the coconut husk involved three stages: washing, alkaline treatment, and acidic treatment. First, the cleaned husks were air-dried for 48 hours before being granulated. In the alkaline treatment, a solution of Sodium Hydroxide (NaOH) was prepared by dissolving NaOH pellets in 5000 ml of distilled water and stirring until homogeneous. The granulated coconut husk was then added to the NaOH solution, stirred for 20 minutes, and left to settle for 24 hours. The mixture was thoroughly washed and filtered using a muslin cloth and distilled water to remove the NaOH until a neutral pH of 7 was achieved. For the acidic treatment, 2500 ml of

sulfuric acid solution was prepared with distilled water and stirred for homogeneity. The alkaline-treated coconut husk was then added to the acid solution, stirred, and allowed to settle. It was subsequently washed and filtered using a muslin cloth with distilled water until a neutral pH of 7 was reached. The treated coconut husk was then dissolved in methanol to extract the cellulose for 72 hours, after which the liquid was extracted to form the methanol cellulose wax inhibitor (WI-MC).



Figure 1: Coconut Husk undergoing treatment



Figure 2: Prepared Inhibitor



Figure 3: Crude samples measured in bottles

2.2 Determination of Wax Content

Determination of the wax content of the crude oil samples was done by the standard acetone process, which is the UOP 46-64 procedure. Each crude oil sample was measured using a 100 ml beaker, 25 ml of toluene was then added to each sample and the mixture was stirred for ten minutes. 5g of aluminum silicate was measured and added to both mixtures. After that, the toluene was extracted by evaporation at 45 °C in the oven. After dissolving the deposit in a 3:1 ether-acetone combination, the mixtures were placed in the freezer for two hours. The mixtures were run through pre-weighed filter papers. The filter paper's wax precipitate was oven-dried. The amount of wax was determined using equation 1.

Wax content

$$= \frac{(\text{weight of filter paper} + \text{precipitated wax}) - (\text{weight of empty filter paper})}{\text{weight of oil measured}} \quad (1)$$

100ml of crude oil samples A and B were divided into various sample bottles and then dosed with coconut husk inhibitor (WI-MC) and Toluene at 0.2, 0.6 and 1.0 ml. The effect of the inhibitors was then evaluated.

2.3 Crude Oil Sample Rheology

The viscosity measurements of the crude samples were taken using a digital viscometer. The viscosity readings of the crude oil containing an inhibitor and that not containing an inhibitor were taken at a temperature range between 30 and 60 °C adopted from the work of Osokogwu *et al.* (2022). The density of each sample was also measured using a density bottle and a weighing balance at the same varying temperature carried out according to the ASTM1217 method.

3.0 RESULTS AND DISCUSSION

The characterization of the crude oil samples used in this study, as presented in Table 1, shows that Sample B is a lighter crude with 26.8 °API gravity, typically associated with lower wax content. In contrast, Sample A is a heavier crude oil 18.7 °API gravity with a higher wax content. Table 2 presents the properties of the crude samples without inhibitors.

Table 1: Characteristic Properties of Waxy Crude Oil Samples

	Sample A	Sample B
API	18.71	26.77
BTEX Analysis	7.835	130.987
Polyaromatic Hydrocarbon	9.722	371.67
Aliphatic Hydrocarbon	399.35	876.21
Wax Content	16%	14%

Table 2: Experimental result of density and viscosity of crude oil sample A & B without wax inhibitor

Temperature (°C)	Sample A without WI-MC Wax Inhibitor			Sample B without WI-MC Wax Inhibitor		
	Density (kg/m ³)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Density (kg/m ³)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)
30	941.8	0.182	193.247	894	0.132	147.651
35	925.2	0.162	175.0973	893.4	0.114	127.6024
40	921.8	0.145	157.3009	891.6	0.104	116.6442
45	917.8	0.097	105.6875	891	0.084	94.27609
50	916.6	0.089	97.09797	889.8	0.07	78.66936
55	912	0.072	78.94737	887	0.064	72.15333
60	910.4	0.047	51.62566	886.04	0.052	58.6881

As crude oil flows inside a pipe, the general trend is that there is a temperature gradient along the wall of the pipe, and it is due to heat being conducted away from the oil to the cooler pipeline surface. The temperature drop has an impact on the wax appearance temperature of the oil. With high rates of cooling at the wall of the pipeline, wax crystals may begin to nucleate at a higher temperature compared to when the rate of cooling is slower. This is because the rapid cooling favors the nucleation of wax crystals before they have any time to grow larger and more ordered structures. The WAT can be determined at the point in which temperature is gradually decreasing and viscosity is increasing. This is the temperature at which wax crystals start to form and affect the flow properties of the oil.

Figures 4 and 5 shows the profile of the experimental data of dynamic viscosity and density of crude oil samples A and B in the absence of wax inhibitor at varying temperatures. The wax appearance temperature (WAT) was determined as the first point on the viscosity vs temperature plot where the viscosity curve converts from a straight to an inclined line at which viscosity is increasing gradually and as temperature decreases (cooling) and this point is 50 °C and 55°C for samples A and B respectively. These observations suggest that higher wax content in heavier crudes leads to easier wax precipitation, aligning with findings reported by Solomon and Osokogwu (2022) and Bai and Zhang (2013).

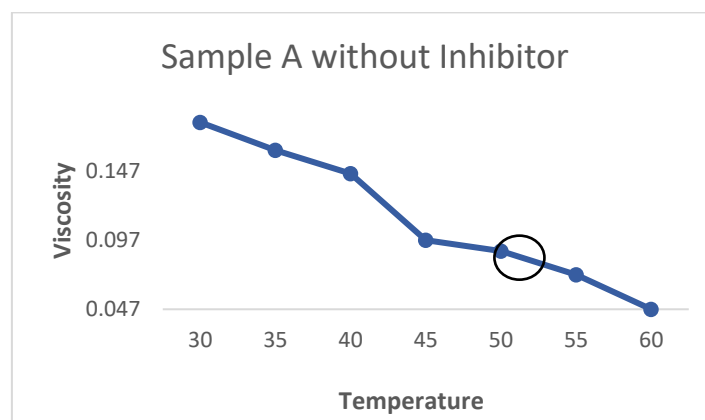


Figure 4: Dynamic viscosity against Temperature of crude oil sample A without WI

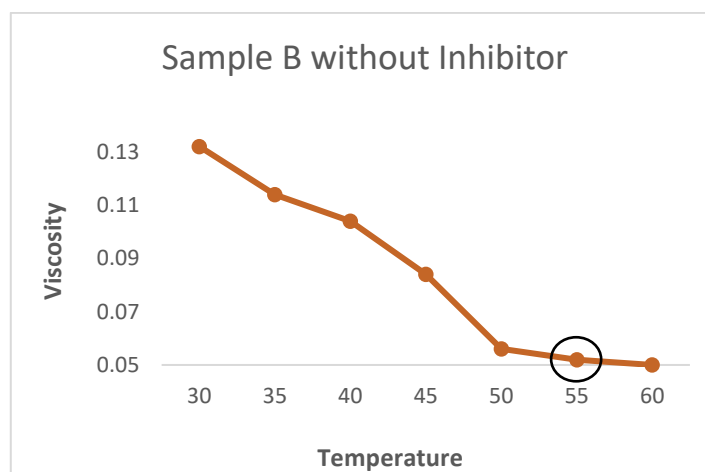


Figure 5: Dynamic viscosity against Temperature of crude oil sample B without WI

Figure 4 shows the Wax Appearance Temperature (WAT) of Sample A can be easily identified, whereas determining the WAT for Sample B requires closer examination as shown in Figure 5. This is because WAT is easier to determine in heavier crudes with higher wax content compared to lighter crudes. These findings are consistent with the work of Bai and Zhang (2013) and Solomon and Osokogwu *et al.* (2022).

Tables 3 and 4 show the properties of crude A with coconut husk and Toluene wax inhibitors respectively at various dosages and temperature conditions. Figure 6 shows a plot of dynamic viscosity and temperature of crude oil sample A with WI-MC inhibitor and different dosage (0.2ml, 0.6ml and 1.0ml) at varying temperature shows the wax appearance temperature (WAT) to be 55°C, 45°C, 50°C respectively. This shows that there is a reverse trend of the effect of the inhibitor after 0.6ml dosage.

Table 3: Properties of crude oil sample A with coconut husk wax inhibitor at various dosages

Temp (C)	WI-MC @ 0.2ml			WI-MC @ 0.6ml		WI-MC @ 1.0ml	
	Density (kg/m ³)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)
30	941.8	0.17	177.3	0.17	183.7	0.164	174.1
35	925.2	0.14	148.08	0.13	141.6	0.144	155.6
40	921.8	0.11	121.50	0.11	123.8	0.112	121.5
45	917.8	0.10	115.49	0.10	115.5	0.098	106.8
50	916.6	0.09	103.65	0.09	100.4	0.089	97.1
55	912.0	0.08	82.24	0.08	84.4	0.075	82.2
60	910.4	0.06	68.10	0.06	68.1	0.060	65.9

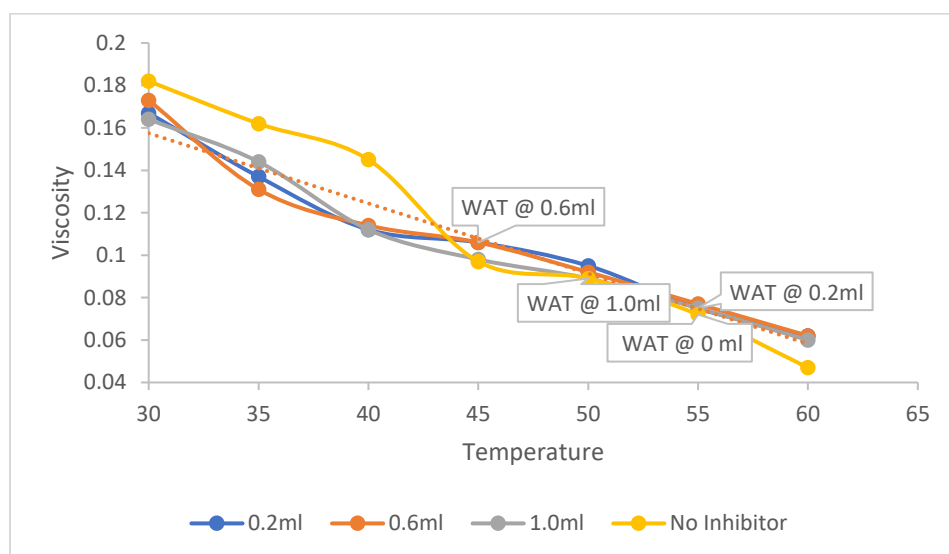


Figure 6: Plot of Dynamic viscosity against Temperature with and without WI-MC Inhibitor for Sample A

Table 4: Properties of crude oil sample A with Toluene wax inhibitor at various dosages

Temp (C)	Density (kg/m ³)	Toluene @ 0.2ml		Toluene @ 0.6ml		Toluene @ 1.0ml	
		Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)
30	941.8	0.27	287.8	0.22	236.8	0.24	255.9
35	925.2	0.20	210.8	0.18	196.7	0.19	208.6
40	921.8	0.15	162.7	0.13	136.7	0.14	155.1
45	917.8	0.14	152.5	0.11	119.9	0.11	123.1
50	916.6	0.12	130.9	0.10	115.7	0.10	109.1
55	912.0	0.09	101.9	0.07	78.9	0.07	80.0
60	910.4	0.08	82.4	0.05	51.6	0.04	47.2

Figure 7 shows a plot of dynamic viscosity and temperature of crude oil sample A with Toluene inhibitor and different dosage (0.2ml, 0.6ml and 1.0ml) at varying temperature shows the wax appearance temperature (WAT) to be 55°C, 50°C, 50°C respectively. This shows that 0.6ml is an optimum dosage beyond which effect of the inhibitor is no longer felt.

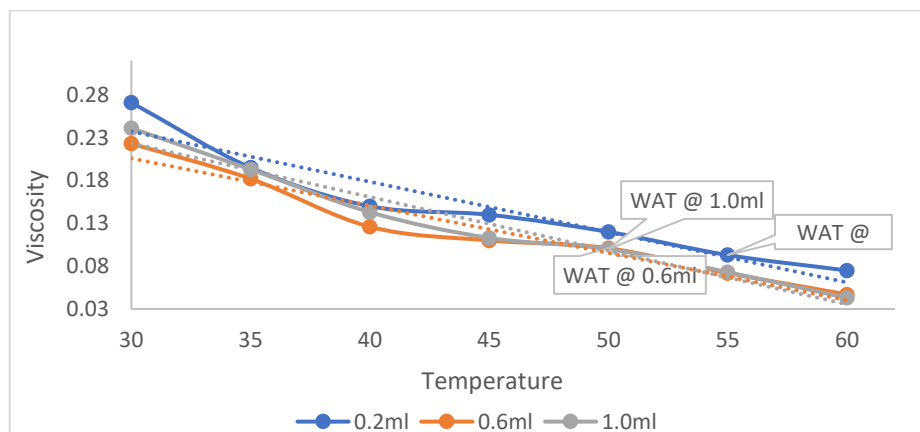


Figure 7: Plot of Dynamic viscosity against Temperature with Toluene Inhibitor for Sample A

Tables 5 and 6 show the properties of crude B with coconut husk and Toluene wax inhibitors respectively at various dosages and temperature conditions.

Table 5: Properties of crude oil sample B with coconut husk wax inhibitor at various dosages

Temp (C)	Density (kg/m ³)	WI-MC @ 0.2ml		WI-MC @ 0.6ml		WI-MC @ 1.0ml	
		Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)
30	894.0	0.13	144.3	0.14	156.6	0.13	148.8
35	893.4	0.12	128.7	0.12	137.7	0.12	133.2
40	891.6	0.11	123.4	0.11	121.1	0.11	124.5
45	891.0	0.10	113.4	0.09	106.6	0.09	108.9
50	889.8	0.09	101.2	0.07	80.9	0.07	84.3
55	887.0	0.07	81.2	0.06	73.3	0.06	73.2
60	886.0	0.06	68.9	0.05	65.5	0.05	62.1

Table 6: Properties of crude oil sample B with Toluene wax inhibitor at various dosages

Temp (C)	Density (kg/m ³)	Toluene @ 0.2ml		Toluene @ 0.6ml		Toluene @ 1.0ml	
		Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)	Dynamic Viscosity (Pa.s)	Kinematic Viscosity (mm ² /s)
30	894.0	0.14	156.6	0.14	151.0	0.15	165.5
35	893.4	0.12	137.8	0.12	137.7	0.12	137.7
40	891.6	0.11	121.1	0.11	128.9	0.11	131.2
45	891.0	0.10	118.9	0.09	108.8	0.10	121.2
50	889.8	0.09	106.8	0.08	100.0	0.09	109.0
55	887.0	0.08	87.9	0.07	81.2	0.07	83.4
60	886.0	0.07	79.0	0.05	79.0	0.05	62.1

Figure 8 shows a plot of dynamic viscosity and temperature of crude oil sample B with WI-MC inhibitor and different dosage (0.2ml, 0.6ml and 1.0ml) at varying temperature shows the wax appearance temperature (WAT) to be 55°C, 50°C, 50°C respectively. This shows 0.2 ml dosage is no effect on WAT and that beyond 0.6ml there is no significant effect on WAT.

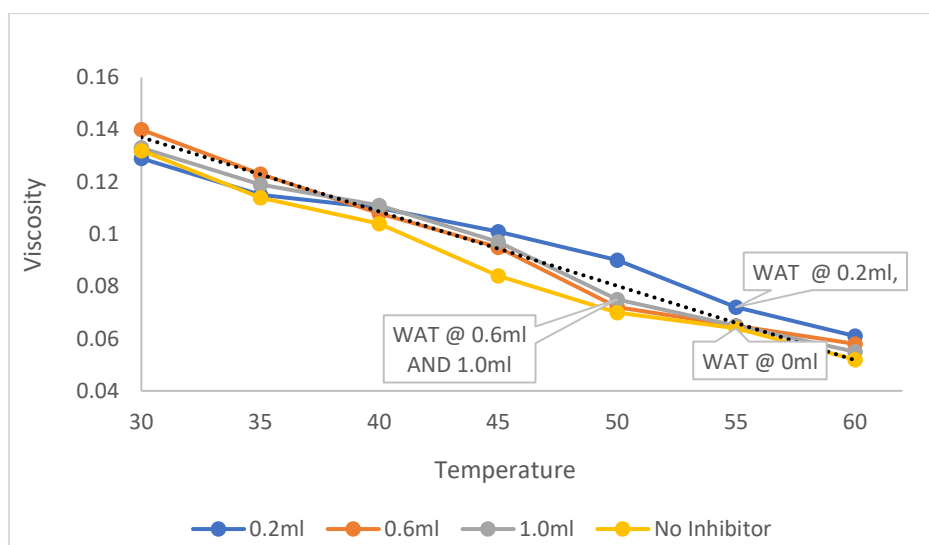


Figure 8: Plot of Dynamic viscosity against Temperature with and without WI-MC Inhibitor for Sample B

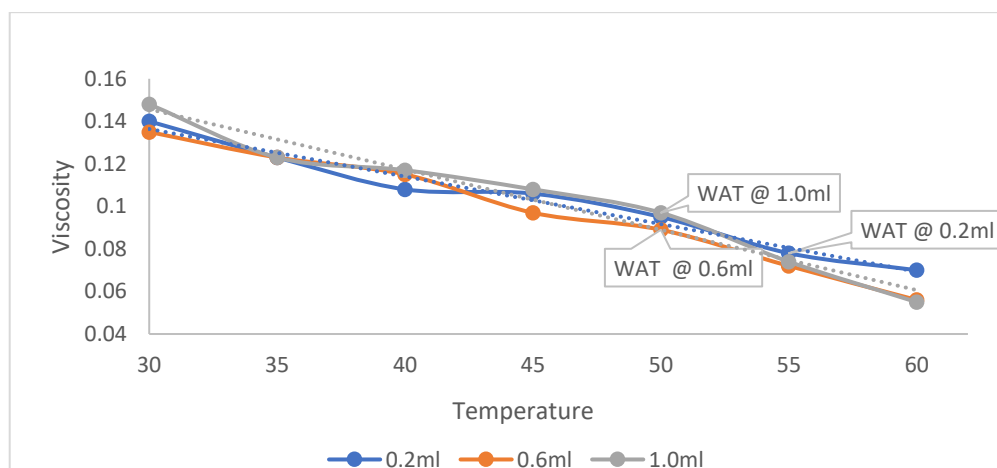


Figure 9: Plot of Dynamic viscosity against Temperature with Toluene Inhibitor for Sample B

The effect of Toluene inhibitor was shown in Figure 9. At different dosages of 0.2ml, 0.6ml and 1.0ml, the wax appearance temperature (WAT) were observed to be 55°C, 50°C, 50°C respectively. This shows a similar trend with the WI-MC inhibitor. The optimum dosage was observed to be 0.6 ml beyond which no effect on WAT was recorded.

The wax appearance temperature (WAT) was determined as the first point on the viscosity vs temperature plot where the viscosity curve deviates from a straight line. At this point, viscosity increases gradually and as temperature decreases (cooling). The dotted lines in each plot was used as a reference point for when viscosity deviates as temperature reduces. The first point of deviation is taken as the WAT. Osokogwu *et al.*, (2022).

This results showed that WAT was least affected by both the WI-MC and toluene at very low concentrations. This could be explained based on the general understanding that the efficiency of wax inhibitors increases with increase in concentration up to a certain threshold. This is as shown in Figures 6, 7, 8 and 9. Also, reduction in WAT at higher inhibitor concentrations is consistent with the mechanism of wax inhibitors. They interfere with the formation and growth of wax crystals, therefore lowering the temperature at which wax becomes visible in oil. At 0.2ml there is no visible change in WAT in both samples. The WAT remains 55°C which suggests that at low dose, there is not enough chemicals to inhibit wax formation effectively This is in accordance to the result reported by Kreangkrai Maneeintr *et al.* (2018). At 0.6ml WI-MC inhibitor dosage in sample A, the WAT reduced to 45°C. In comparison with Toluene, at 0.6ml and 1.0ml dose, the WAT reduces to 50°C and 50°C respectively. This observation is supported by the report of Bai and Zheng (2013) where wax inhibitors significantly reduced WAT with increasing concentrations, reflecting improved performance in preventing wax crystallization. Similarly, Maneeintr *et al.* (2021) found that the efficiency of wax inhibitors depends on their concentration, reinforcing the need for optimal dosage to achieve the best results.

4. CONCLUSION

Coconut husk was used as raw material to develop a wax inhibitor for crude oil. The inhibitor was applied on crude oil samples to determine the wax appearance temperature (WAT) of the crude oil. The results obtained showed that the developed wax inhibitor from coconut husk reduced the WAT of crude oil samples A and B similarly to the toluene inhibitor. To confirm the efficiency, various concentrations of the wax inhibitors were dosed in the amount of 0.2 ml, 0.6 ml, and 1.0 ml on the two different crude oil samples, A and B. The performance of toluene validated that of the WI-MC, indicating that the coconut husk-based inhibitor can be used as an alternative to conventional wax inhibitors. This result is very important in proving that local materials can be very useful in the production of effective wax inhibitors, hence reducing the cost associated with this in the industry and making it more sustainable. Although the results obtained in this study compared favorably with commercial wax inhibitor, the study was conducted in a controlled laboratory environment. Therefore, a field trials may be carried out to assess the WI-MC long-term performance and compatibility with other oilfield chemicals. A thorough economic analysis may also be attempted to evaluate the cost-effectiveness of the WI-MC compared to commercial wax inhibitors.

ACKNOWLEDGEMENT

The authors acknowledge the Technological staff of the Department for support during the study.

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