Effects of Crude Palm Oil and Crude Palm Kernel Oil Upon Wax Inhibition

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ABSTRACT: The ever-increasing demand for the finite source of oil has led oil production companies to produce and transport the produced crude oil as efficiently and economically as possible. One of the major concerns especially in waters like the South China Sea is the deposition of wax on the walls of the pipeline or wellbore, constricting and hindering the hydrocarbon flow. This is due to the low seabed temperatures, which can be below the wax appearance temperature (WAT), leading to the deposition of wax out of waxy crude oil through the molecular dispersion mechanism. Currently, many prevention and remedy methods are in place to overcome the problem, but most of the additives possess environmental threat, as most of the chemical solutions used are toxic, nonorganic, and costly. Hence, this paper aims to provide some insights into the effect of palm oil derivatives such as crude palm oil (CPO) and crude palm kernel oil (CPKO) on wax inhibition. The effect of aging time (i.e., immersion time) was also evaluated. A comparison was made between paraffin inhibition efficiency results (PIE %) obtained by CPO, CPKO, poly(ethylene-co-vinyl acetate) (EVA), and triethanolamine (TEA). It was observed that the average efficiency of 81.67% was obtained when 1% CPO was added to heavy crude oil. The wax inhibition performance reached a plateau after 1.5 h of aging time for all of the investigated samples.

1. INTRODUCTION

The demand for crude oil has been on the rise and is predicted to increase up to the year 2030, when the oil demand will peak at 104.7 mb/d. Therefore, for oil-producing countries and organizations like Petronas, Shell, and Exxon Mobil, the extraction, transportation, and storage of crude oil are significant in guaranteeing a safe rate of return in investment. Unfavorably, there are flow assurance issues such as paraffin wax deposition, hydrate formation, asphaltenes, scale, sand, erosion, and corrosion that require special attention in preventing blockage and other difficulties in producing the crude oil. In tropical waters like Malaysia, the problem of paraffin wax deposition is the major nuisance compared to the listed flow assurance issues. The average temperatures of the surface and the subsea are 34 and 25 °C, respectively, clearly leading to a temperature difference between the pipelines walls and the crude oil. Driven by molecular diffusion mechanism, paraffin wax deposition occurs during crude oil transportation via pipelines as well as during the production in the tubing string where the deposition of the unwanted substance causes an increase in the pressure drop (requiring higher pressure to transport the crude oil) in the pipeline. If the deposition is not treated, plugging of solid paraffin wax deposits will occur, leading to unwanted downtime to perform remedial operations.

Precipitation of wax happens when the temperature of the crude oil solution decreases below the wax appearance temperature (WAT). WAT is defined as the maximum temperature at which the first wax crystals start to form when the waxy crude oil is cooled. Although some researchers are still debating on the types of wax deposition mechanism, most of them agree that molecular diffusion is the most dominant wax deposition mechanism. This is because molecular diffusion involves the presence of a temperature gradient due to the colder pipe wall and warmer bulk fluid. The colder pipe wall causes crystallization and adhesion of wax out of the bulk fluid (i.e., oil) that is close to the pipeline. This leads to a low wax concentration near the pipe wall creating a concentration gradient in the pipeline, which then results in wax molecular diffusion from the bulk fluid to the pipe wall. A few researchers also portrayed that the bulk fluid does not have to be below WAT for wax deposition.
to occur as long as there is a temperature gradient present between the bulk crude oil and the pipe wall. The effect of molecular diffusion is very common in deep water pipelines, where the temperature of the seabed is near freezing, causing the submerged pipeline to mimic the near-freeze temperature.

There are various methods available to prevent wax deposition. One of the common methods is to inject chemical additives to prevent wax adhesion in the pipeline. The presence of chemical additives enhances crude oil flowability, reduces the pour point, and reduces the agglomeration of wax crystals. However, the current use of industrial synthetic chemical additives such as poly-(ethylene-co-vinyl acetate) (EVA) and triethanolamine (TEA) possess toxicity problems when exposed to the environment. This issue has necessitated the search for alternative, cheaper, and natural chemical inhibitors to tackle the wax deposition problem. Previously, oleic acid based chemical has been portrayed as a potential wax inhibitor by some researchers. Hafiz et al. revealed that a surfactant developed from the esterification of oleic acid with hexatriethanolamine is an excellent flow improver. They utilized a DeNouy Tensiometer (Kruss-K type) by applying a platinum ring approach to determine the surface tension measurements. They concluded that the presence of oleic acid in TEA further increased the performance of the wax inhibitor depending on the molecular weight of the crude oil tested. Hexa-triethanolamine mono-oleate performed best under a low-molecular-weight crude oil while hexa-triethanolamine tri-oleate performed best under a high-molecular-weight crude oil.

Meanwhile, Patel et al. investigated the rheological behavior of oleic acid based additive at various concentrations. They found that when the oleic acid is mixed with comb-shaped copolymers of maleic anhydride to form poly (hexyl oleate-co-hexadecyl maleimide-co-n-alkyl oleate), the resulted solution has excellent flow-improving properties, particularly at high concentration. Another rheological investigation was conducted by Soni et al. on comb-shaped polymeric diester additives mixed with oleic acid in reducing the pour point of Kathana crude oil, India, by utilizing an Advance Rheometer AR-500 from TA Instrument Company. The performance of the polymer additives was also evaluated by conducting a cold finger test. They concluded that among the polymer additives tested, polymer additives—oleic acid blend was the best pour point depressant as well as rheology modifier.

Recently, Akinyemi et al. had reported the effect of plant seed oils such as jatropha (JSO), rubber (RSO), and castor (CSO) on the wax deposition of Nigerian waxy crude oil. The content of oleic acid in JSO, RSO, and CSO is 43.11, 18.30, and 4.73%, respectively. In this research, they have utilized the cold finger apparatus and reported that these cheaper, natural chemicals of RSO, JSO, and CSO can reduce the amount of deposited wax by 69.9, 73.5, and 74.7%, respectively. Additionally, a blend of CSO with JSO improved the wax inhibition further by up to 79.1%. Although the effect of oleic acid on wax deposition has been extensively investigated, there is still a lack of literature on the potential effects of cheaper and natural plant-based oil on wax deposition such as palm oil, which also contains oleic acid. The aim of this paper is to shed some light on the effect of palm oil derivatives such as crude palm oil (CPO) and crude palm kernel oil (CPKO) on inhibiting the wax deposition. The effect of aging time (i.e., immersion time) was also evaluated.

2. RESULTS AND DISCUSSION

2.1. Static Wax Deposition Test. Two types of palm oil derivates were investigated for their effect on wax inhibition. In this work, the mass of the wax that deposits out of crude oil sample is weighed and plotted against the aging time as illustrated in Figure 1a. Figure 1b is a focalized view of Figure 1a, where the crude oil plots are omitted to observe the implication of the addition of 0.1, 1, and 10% of CPO and CPKO additives, respectively, on the accumulative mass of the wax deposited.

From Figure 1, results show that the cumulative mass of the deposited wax increases with aging time in the presence as well as in the absence of palm oil additives. It can be seen that the presence of 0.1, 1, and 10% of CPO and CPKO additives, respectively, reduces the amount of deposited wax progressively even up to 6 h of aging time. At 6 h aging, the highest amount of deposited wax was 70.67 g (cumulative) (Figure 1b) as depicted by the crude oil sample. At the same aging time, the lowest amount of deposited wax was found when the crude oil sample was added with 1% CPO, which is 13.91 g (cumulative). Consequently, the comparison of the highest amount of deposited wax in the presence of 1% EVA, TEA, CPO, and CPKO additives is 23.24, 16.34, 13.91, and 15.29 g, respectively. This reflects that natural additives can perform as effectively as synthetic additives.

For 1% CPO additive, the rate of wax deposition was found to be 3.42 g/h for the first hour of aging time and an average of 2.220 g/h for the second up to the sixth hour of aging time. It should also be pointed out that in the presence of the natural additives (CPO and CPKO) in the crude oil, all sample

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**Figure 1.** (a) Mass of wax deposited against the deposition time of chemical inhibitors in comparison with crude oil. (b) Mass of wax deposited against the deposition time of chemical inhibitors only.
mixtures portrayed a significant reduction on the wax deposition as compared to the crude oil only. This demonstrates that both the palm oil additives possess the ability to generate a barrier that prevents the wax crystals network from interlocking and hindering wax deposition.

To obtain a better understanding of the inhibition efficiency of the palm oil additives used, the graph of paraffin inhibition efficiency (PIE) against aging time and the average paraffin inhibition efficiency (PIE) against the concentration of additives were plotted, as illustrated in Figures 2 and 3, respectively.

![Figure 2](image1.png)

**Figure 2.** Paraffin inhibition efficiency (PIE) against aging time.

![Figure 3](image2.png)

**Figure 3.** Average paraffin inhibition efficiency (PIE) against the concentration of additives. Data shown are with a 5% error bar.

According to Figure 2, where the graph illustrates the paraffin inhibition efficiency (PIE) against deposition time, it can be clearly observed that 1% CPO has the highest pie inhibition efficiency at the end of 6 h, followed by 1% CPKO, 0.1% CPO, 0.1% CPKO, 10% CPKO, and lastly 10% CPO. The ranking of the additive performance based on wax inhibition is as follows: 1% CPO > 1% CPKO > 0.1% CPO > 0.1% CPKO > 10% CPO > 10% CPKO. Remarkably, the inhibition performance of 1% CPO > 1% CPKO > 0.1% CPO > 0.1% CPKO, 10% CPKO, and lastly 10% CPO. Meanwhile, the average PIE of CPKO in the presence of 0.1, 1, and 10% concentration are 62.84, 77.302, and 59.75%, respectively. In addition, for both CPO and CPKO, the PIE is seen to increase when the concentration increases from 0.1 to 1% and then decline when the concentration is 10%. However, with the limited concentration tested, this conclusion is too soon to be confirmed. Thus, future investigations should be conducted to obtain a better understanding of the optimum concentration of palm oil-based additives required to effectively inhibit wax deposition.

The main reason behind the excellent efficiency of the 1% CPO may be due to the high composition of oleic acid present in CPO as compared to that in CPKO, as mentioned in Table 1. However, it should also be noted that the increase in palm oil additives for both CPO and CPKO did not necessarily portray better inhibition. The increase in CPO and CPKO concentration above 1% concentration causes a decrease in PIE. This may be due to the high amount of CPO and CPKO molecules acting as nucleation sites for the wax molecules to flocculate and crystallize. Consequently, the existence of wax crystals in the presence of CPO and CPKO additives was investigated via cross-polarized microscopy (CPM).

### 2.2. Determination of Wax Appearance Temperature

In this work, cross-polarized microscopy (CPM) was employed to investigate the effect of palm oil derivatives on the wax appearance temperature (WAT). Besides the CPM test, there are several techniques for WAT measurement such as gas chromatography–mass spectrometry (GC/MS), nuclear magnetic resonance (NMR), viscometry, and differential scanning calorimeter (DSC). Here, Figures 4–9 illustrate the images obtained from the CPM test at the temperature where the earliest visible wax crystal can be seen as in (a) and the image of wax crystals in the sample mixture at the end of the experiment for each sample tested in (b). As can be seen from Figure 4 until Figure 6, the wax appearance temperature (WAT) decreases with the concentration of the CPO additive. The WAT recorded for 0.1, 1, and 10% CPO is 20.8, 8.7, and −6.8 °C, respectively. For 0.1 and 1% CPKO, the recorded

### Table 1. Composition of Fatty Acids in CPO and CPKO

| fatty acid | lipid number | CPO (%) | CPKO (%) |
|------------|--------------|---------|----------|
| oleic acid | C18:1        | 44.1    | 15.3     |
| palmitic acid | C16:0    | 45.8    | 8.4      |
| stearic acid | C18:0     | 5.4     | 2.5      |
| linoleic acid | C18:2   | 12.5    | 1.3      |
| myristic acid | C14:0     | 1.5     | 16.2     |
| arachidic acid | C20:0   | 0.5     |          |
| lauric acid | C12:0       | 0.5     | 48.2     |
| α-linolenic acid | C18:3 | 0.6      |          |
| palmitoleic acid | C16:1  | 0.4     |          |
| 9,10-dihydroxysearic | C18:2 |          |          |
WAT is 12.6 and 10.2 °C, respectively (see Figures 7 and 8). It should be noted that as the limitation of the equipment when 10% CPKO additive was added to the crude oil, the background of the image became yellowish (Figure 9). Hence, the recorded temperature measurement is vague and confusing. For the future investigation, the test should be repeated using a waxier crude oil to obtain more decisive results of the inhibition efficiency of the palm oil additives.

Figure 10 shows the average paraffin inhibition efficiency (PIE) and wax appearance temperature (WAT) reflected against the concentration of the additives used. It can be seen that the increase in CPO concentration decreases the WAT of the crude oil up to 1% concentration, but the WAT then slightly increases as the concentration of CPO further increases to 10%. Meanwhile, for CPKO, the increase in concentration does decrease the WAT of the crude oil and increases the PIE but to a certain concentration as well. At high concentration, like 10%, CPKO was not effective at reducing not only the WAT as well as PIE. This shows that CPO and CPKO may be active sites for the wax crystal to agglomerate and crystallize at high concentration, hence causing the ineffectiveness in reducing the PIE. Hence, when comparing both the additives for Mt Oversea Mckyle Arab heavy crude oil, CPO is a more suitable inhibitor to be used.
3. CONCLUSIONS AND RECOMMENDATION

Both the palm oil additives have the ability to prevent the interlocking of wax crystal network and thus hinder their deposition, but further research is required before the additives are applied in the field. Among the additives tested, CPO is a much more suitable candidate for inhibiting wax deposition for the Mt Oversea Mckyle Arab heavy crude oil due to the presence of oleic acid in high concentration. CPKO may still be effective as a sustainably produced inhibitor for crude oil with a higher pour point. Results showed that 1% CPO additive exhibits 81.67% inhibition efficiency. The wax inhibition performance reached a plateau after 1.5 h aging time for all of the investigated samples. The ranking of the additive performance based on wax inhibition is as follows: 1% CPO > 1% CPKO > 0.1% CPO > 0.1% CPKO > 10% CPO > 10% CPKO. Remarkably, the inhibition performance of 1% CPO natural additive is similar to that of 1% TEA synthetic additive. Hence, further research is recommended utilizing these palm oil additives under various types of crude oil, preferably wavier, studies under dynamic flow conditions, pour point, gelation temperature, rheological behavior, and comparing the development of WAT measurement with the cooling rate among different techniques using differential scanning calorimeter and viscometry.

Figure 7. (a) Image of the early wax crystal (in the red box) in the presence of 0.1% CPKO appears at 12.6 °C. (b) Image of wax crystal at the end of the experiment using 0.1% CPKO at −19.0 °C.

Figure 8. (a) Image of the early wax crystal (in the red box) in the presence of 1% CPKO appears at 10.2 °C. (b) Image of wax crystal at the end of the experiment using 1% CPKO at −19.2 °C.

Figure 9. (a) Image of the early crystal (in the red box) in the presence of 10% CPKO appears at 23.0 °C. (b) Image of wax crystal at the end of the experiment using 10% CPKO at −15 °C. Note that the nature of the CPKO sample at high concentrations resulted in the yellowish background of the CPM image.
4. EXPERIMENTAL SECTION

4.1. Materials. The crude palm oil (CPO) and crude palm kernel oil (CPKO) samples as shown in Figure 11 were obtained from United Plantation and Unitata Company. The composition of fatty acids in the CPO and CPKO additives used in the experiment is shown in Table 1. The crude oil used (Arab heavy) is from Mt Oversea Mckyle. Poly(ethylene-co-vinyl acetate), 80 wt % (EVA), and triethanolamine, MW 149 g/mol (TEA), were purchased from Sigma Aldrich.

4.2. Characterization of Crude Oil. The physicochemical properties of the Mt Oversea Mckyle crude oil sample including its density, American petroleum index (API) gravity and saturate, aromatics, resins, and asphaltenes (SARA) distribution are shown in Table 2. The WAT value of the crude oil sample was obtained using differential scanning calorimeter (DSC) measurement as shown in Figure 12. After a heating phase up to 80 °C, the crude oil sample was cooled down from 80 to 0 °C at 1 °C/min.

4.3. Static Wax Deposition Test. The cold finger test is the most appropriate test to represent the wax deposition tendencies of crude oil samples in an economic and time-friendly manner. The cold finger mimics the subsea condition where the warm oil is in contact with the colder pipe wall, creating a molecular diffusion mechanism of wax deposition.25 Hence, this research will utilize a cold finger system to measure the wax inhibition efficiency of palm oil additives under static conditions. The system setup of the cold finger is depicted in Figure 13.

First, the oil tank was filled with 300 mL of the crude oil sample. The bulk point temperature was maintained at 25 °C. The cold finger probe was set at 5 °C to produce a temperature gradient of 20 °C. At an interval of 1 h, the cold finger was removed from the apparatus and the wax deposited was weighed. The experiment was run for 6 h to investigate the wax deposition at various aging times. The experiment was repeated by utilizing the additives at the concentrations of 0.1, 1, and 10%. For this experiment, the crude oil sample was preheated to 60 °C. The additive was added to the crude oil sample, and the mixture was stirred until homogeneous.

For each run of the experiment, three oil tanks of the cold finger system are utilized at the same time and average amounts of deposited wax are used to calculate the paraﬁn inhibition efﬁciency. The paraﬁn inhibition efﬁciency (PIE %) of each chemical additive was determined using eq 1.
pour point of the crude oil (WAT). Hence, cross-polarized microscopy (CPM) setup shown in Figure 14 is used to determine the WAT of the chemical inhibitor. Therefore, cross-polarized microscopy (CPM) is known as the WAT or cloud point of the sample.

4.4. Determination of Wax Appearance Temperature.

The wax appearance temperature (WAT) or cloud point of the crude oil depends on the wax content of the crude oil and is a parameter of interest when evaluating the efficiency of the wax chemical inhibitor. Hence, cross-polarized microscopy (CPM) functions on the basis that crystallized wax is able to rotate the polarized light plane (anisotropic) and the utilization of two prisms in the apparatus permits the microscope field of investigation to initially appear black but as wax crystallizes, white or light brown spots will be visible. The temperature when this occurs is known as the WAT or cloud point of the sample.

In addition, to ensure a constant decrease in the temperature gradient, liquid nitrogen is used to cool down the sample to the pour point of the crude oil (−18 °C).

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Notes
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NOMENCLATURE

API American Petroleum Index
CPKO crude palm kernel oil
CPO crude palm oil
CSO castor seed oil
EVA ethylene vinyl acetate
JSO Jatropha seed oil
PIE paraffin inhibition efficiency
RSO rubber seed oil
SARA saturate, aromatics, resins, asphaltene
TEA triethanolamine
WAT wax appearance temperature

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