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Wax inhibitor based on ethylene vinyl acetate with methyl methacrylate and diethanolamine for crude oil pipeline

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Abstract. Wax precipitation and deposition is one of the most significant flow assurance challenges in the production system of the crude oil. Wax inhibitors are developed as a preventive strategy to avoid an absolute wax deposition. Wax inhibitors are polymers which can be known as pour point depressants as they impede the wax crystals formation, growth, and deposition. In this study three formulations of wax inhibitors were prepared, ethylene vinyl acetate , ethylene vinyl acetate co-methyl methacrylate (EVA co-MMA) and ethylene vinyl acetate co-diethanolamine (EVA co-DEA) and the comparison of their efficiencies in terms of cloud point¸ pour point, performance inhibition efficiency (%PIE) and viscosity were evaluated. The cloud point and pour point for both EVA and EVA co-MMA were similar, 15 ^{o}C and 10-5 ^{o}C, respectively. Whereas, the cloud point and pour point for EVA co-DEA were better, 10 ^{o}C and 10-5 ^{o}C respectively. In conclusion, EVA co-DEA had shown the best % PIE (28.42%) which indicates highest percentage reduction of wax deposit as compared to the other two inhibitors.

1. Introduction

Paraffinic crude oil has been a huge historical challenge for operators in the oil and gas industry. The principle problems arise when the equilibrium is disturbed during production or transportation which leads to alteration in pressure, temperature and composition and eventually results in extensive problems such as the precipitation and deposition when the crude oil solidifies on the flow lines of the production system causing the internal diameter to decrease as well as restricts the flow of the fluid. The main composition of crude oil are saturates, asphaltenes, aromatic and resins. Paraffins are the main saturates that contribute to the formation and deposition of wax during the transportation process whereby the solubility decreases as the temperature and operating pressure decrease [1-3]. This phenomena leads to the wax formation and deposition where it tends to create crystalline capable of entrapping liquid crude oil [4,5]. The temperature difference between the pipelines walls and the crude oil driven by molecular diffusion mechanism results in the wax formation and deposition on the surface of the pipelines walls. Wax deposition process can eventually results in significant reduction of the internal diameter of the tubular, casing , perforations, pump strings, rods, pipelines and at worst case causing complete blockage to the flow lines that leads to tremendous profit loss [6,7].

Due to consequential problems faced as a result of wax deposition many treatment methods were developed, such as mechanical, thermal, chemical, and biological treatments that are usually implemented to clean and disperse the deposits based on its nature, location, cost and other influential factors [8]. All these methods are applicable to solve the problem after the wax has been precipitated in the pipeline. In addition to that , there are also several wax prevention techniques such as increasing the operating pressure which promotes the shear rate, insulation the flow lines or even coating the inside of the flow lines with a non-adhering material. Unfortunately the abovementioned techniques come in high cost and usually require partial or complete shutdown of production system in order to be implemented.

Wax prevention techniques have shown an increased interest to the researchers. Many chemical additives were developed to reduce or eliminate this problem. Chemical additives such as wax inhibitors are one of the wax controlling methods that are implemented the industries to help control the deposition and provide flow assurance. Chemical wax prevention methods come in three approaches which are wax dispersants, wax inhibitor (wax crystal modifier) and pour-point depressant (PPD).

Wax inhibitors such as pour point depressants and wax dispersants are used to chemically modify the wax solid structure thus reducing the tendency of the wax crystals to interlock and form three dimensional network growths [9,10]. The main function of wax inhibitors is to impede the paraffin growth. However, these wax inhibitors do not stop the waxing process but it delays the precipitation and hardening process, thus allowing the wax aggregates to be swept away in the oil production steam flow [11].

Even though pour point dispersant and wax dispersants are under the same class that is the chemical additives, but they are different from one another. Pour point dispersant is a wax crystal modifier which functions by co-crystallizing into paraffin structure through van der Waals interactions leaving the polar moiety end tail to create a steric hindrance to interfere with alignment of new incoming wax molecules. Whereas, the wax dispersant functions by absorbing itself into the wax crystal and prevent further sticking of the molecules. Recently, wax dispersants are blended with wax inhibitor to enhance their performance in reducing wax deposition [12-15].

Ethylene vinyl acetate (EVA) is an ester group which can weaken the solid deposition of the wax in the crude oil through van der Waals bonding in which it results in an easy removal of the deposition by sheer force of the flow stream [16]. The influence of the EVA on the crude oil's pour point and cloud point has been investigated by several researchers, 30 wt% of EVA was found to be the most efficient copolymer as a pour point depressant compared to other copolymers [2,17,18]. Methyl methacrylate is the other class of comb polymers commonly used as wax inhibitors. Methyl methacrylate (MMA) is made up from acrylic acid monomer whereas the ester group are made up from long chain of alcohols. EVA and MMA are saturated compounds and the refractive index between both these wax inhibitors is very close ,making them easily blended together [19]. Diethanolamine (DEA) comprises of hydroxyl groups and also contains wax-like portions that allow the molecules to incorporate into the wax deposition structure. The aim of this study was to evaluate the performance of two pour point dispersants (MMA, and DEA) when blended with EVA.

2. Experimental methods

2.1. Preparation of ethylene vinyl acetate co-methyl methacrylate (EVA co-MMA) and co-diethanolamine (EVA co-DEA) wax inhibitor

EVA (40 wt% melt) & DEA (reagent grade ≥ 98.0%) was purchased from Sigma Aldrich, USA, MMA (≥ 99.5%) & cyclohexane (≥ 99.5%) was purchased from Merck KGaA, Germany. All these reagents were supplied by Merck Sdn. Bhd, Selangor, Malaysia. Cyclohexane was used as solvent for EVA. For the preparation of the inhibitor, the reaction was conducted on a hot plate at a temperature of about 50-60℃ with continuous stirring using magnetic stirrer. A hot plate was used instead of an oven to allow space to work while preparing the chemicals. The individual chemicals, EVA, MMA, DEA and cyclohexane were measured separately of its respective volume and weightage in accordance to the manipulated percentage composition. EVA was measured in grams of mass, meanwhile MMA, DEA and cyclohexane were measure in mL.

The EVA co-DEA and EVA co-MMA was prepared by using a blend polymerization method. For both of the inhibitor formulations 1 g of (30 wt%) EVA was measured using electronic mass balance, and then added to 10 mL of cyclohexane using a micropipette. For the first formulation 5 mL of MMA was added, meanwhile for the second formulation 5 mL of DEA was added to the mixture. Finally the two mixtures were heated in the measuring cylinder at 50-60 ℃ to melt the copolymer. When the inhibitors were completely melted, the crude oil was poured into the measuring cylinder. In order to obtain the cloud and pour point of the crude oil, cooled down the crude oil with the wax inhibitor from 50 ℃ to 0 ℃ with decrement of temperature by 3 ℃ for every 2 to 3 hours. The experiment was carried out for different amount of the EVA with the fixed amount of DEA and MMA. This is to observe the effect of EVA mass on the viscosity of the crude oil.

2.2. Determination of pour point and cloud point temperature

Pour point and cloud point temperature of both treat and untreated crude oil samples were determined using ASTM D97 method and ASTM D2500 method respectively [20-22,16,17]. To find the pour point ,at which the crude oil fluid becomes semi solid and losses its flow characteristics ,100 mL of the crude oil sample was heated to 50 ℃ in an oven and kept overnight to ensure that the wax crystals have completely dissolved [22]. The 4 samples were placed in a pour point test jar which was closed tightly by the cork carrying the thermometer in order to monitor the temperature drop .After the sample was heated to 50 ℃ using the water bath, the water bath temperature was decreased gradually and observation of the temperature decrease was noted. Every 3 ℃ temperature drop the movement of fluid was checked until the crude oil showed no movement when being held in the horizontal position for exactly 5 seconds. The temperature at which the fluid stops flowing is recorded from the thermometer. That temperature represents the pour point of the fluid. Cloud point measurement was done using similar procedure of pour point except the cloud point temperature is recorded when smallest cluster of hydrocarbon crystals was first observed then the temperature on the thermometer was recorded.

2.3. Determination of viscosity

Viscometer was used to evaluate the viscosity of the crude oil samples. The viscometer used is the Brookfield Programmable Viscometer DV-III + Rheometer. The standard settings of the equipment shown in table1.

Table 1. Standard settings of viscometer

Spindle size	7
Rotational speed	100 rpm
Units	cgs
Reading	Viscosity (cP) Torque (%)

The viscometer was left to warm up for about 10 minutes after start up, and then, auto-zeroed without the spindle and at a levelled position. Each set of samples was tested of its viscosity from 50 ℃ to 5 ℃ at every 5 ℃ intervals. Firstly, the control experiment is conducted which is the blank crude oil at the absence of inhibitor. The crude oil was removed from the oven and allowed to cool down to 50 ℃, then measure of the viscosity using the viscometer. The temperature of the sample was observed and controlled using a temperature probe. Then, the sample was allowed to cool down to 45 ℃ before the viscosity is measured again. The step is repeated at every 5℃ interval until the sample temperature drops to 5 ℃. To cool the sample below the room temperature, the sample is immersed into an iced water bath. Care was taken to avoid upsetting the spindle. The procedure was repeated for each of the other samples that contain the other inhibitor. Before starting a new sample each time, the spindle and temperature probe is lightly cleaned to avoid contamination of the samples. Again, the spindle is handled with care in order to maintain the calibration.

2.4. Determination paraffin inhibitor efficiency (PIE)

After the wax precipitation has been observed from the crude oil sample at the first sight, decreased the temperature from 10 to 5 ℃ for 8 hours, and ensured that the crude oil continuous to flow by stirring the sample for every 10-15 minutes. After that, the specimen was heated to increase its temperature so that the wax from the crude oil sample was separated. The wax out from the crude oil was filtered by using filter paper and dried at room temperature for 1 day. The mass of the crude oil was weighed by using the electronic balance and the paraffin inhibition efficiency is determined by using the formula [16,23]:

$$PIE(\%) = \frac{W_f - W_t}{W_f} x\, 100$$

where W_f is the reference amount of wax deposit without chemical treatment in gram (g) and $W_t$is the amount of paraffin deposit with chemical treatment in gram (g).

3. Results and discussion

3.1. Effects of the inhibitors on the pour point and cloud point of the crude oil

Pour point and cloud point of samples were measured by following the ASTM procedures (ASTM D-97 and ASTM D-2500) respectively. These two tests were crucial to evaluate and compare the efficiency of the inhibitor formulations proposed to inhabit the wax formation of the crude oil. Table 2 clearly shows that the untreated crude oil solidified at the temperature of 35 ºC. Meanwhile, crude oil treated with EVA , EVA co-MMA and EVA co-DEA solidified at temperature of 15 ºC, 15 ºC, 10 ºC respectively, which confirms the efficiency of the these inhibitor formulations to reduce the temperature of wax formation. This proved the statement that reported by Vieira *et al* [24] whereby it was stated that the WAT is related to the molar mass and structure of the chain in which the waxes that are more linear and have higher molar mass crystallized at higher temperature, meanwhile less linear waxes with lower molar mass crystallized at lower temperature [25]. The molar weight of DEA, MMA and EVA is 105.14 g/mol, 100.12 g/mol and 114.14 g/mol respectively. Since

MMA has the lowest molar weight, it had crystallised at a lower temperature followed by DEA and then EVA. But, since the structure of DEA is less linear compared to MMA, causes the DEA to be crystallised at lower temperature compared to the MMA. The difference in the molar mass between MMA and DEA is quite small making it possible.

Table 2. The effect the inhibitors on the wax precipitation on the crude oil in terms of temperature

Temperature, °C	Crude oil only	Crude oil + EVA	Crude oil + EVA + MMA	Crude oil + EVA + DEA
50	X	X	X	X
47	X	X	X	X
44	X	X	X	X
41	X	X	X	X
38	X	X	X	X
35	X	X	X	X
32	/	X	X	X
30	/	X	X	X
25	/	X	X	X
20	/	X	X	X
15	/	/	/	X
10	/	/	/	/
5	/	/	/	/

whereby, X sign indicates that there is no precipitation of wax and / Sign indicates that there is precipitation of wax.

3.2. Effect of temperature on viscosity for the crude oil with and without wax inhibitor

Figure 1 shows the relationship of between viscosity and temperature for the 4 samples. It can be observed that the viscosity of the crude oil is has a steadily decreased with the temperature decreased whether it is mixed with wax inhibitor or not. The untreated crude oil showed the highest viscosity value at 5 °C that is 1460 cP followed by the treated crude oil with EVA co-MMA, EVA co-DEA and EVA. The viscosity of the treated crude and untreated oil does not have any significant differences between them at low temperature, however it can be observed that the viscosity decline of the samples that contained EVA and EVA co-DEA was very clear compared to the other samples. Therefore, it can be concluded that the sample with EVA co-DEA wax inhibitor shoes best response compared to the other three samples of untreated crude oil, crude with EVA inhibitor and crude with EVA co-MMA inhibitor.

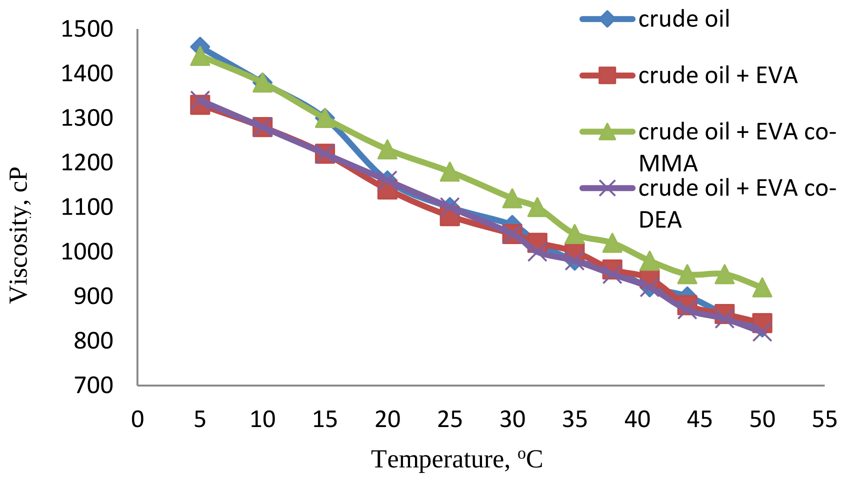
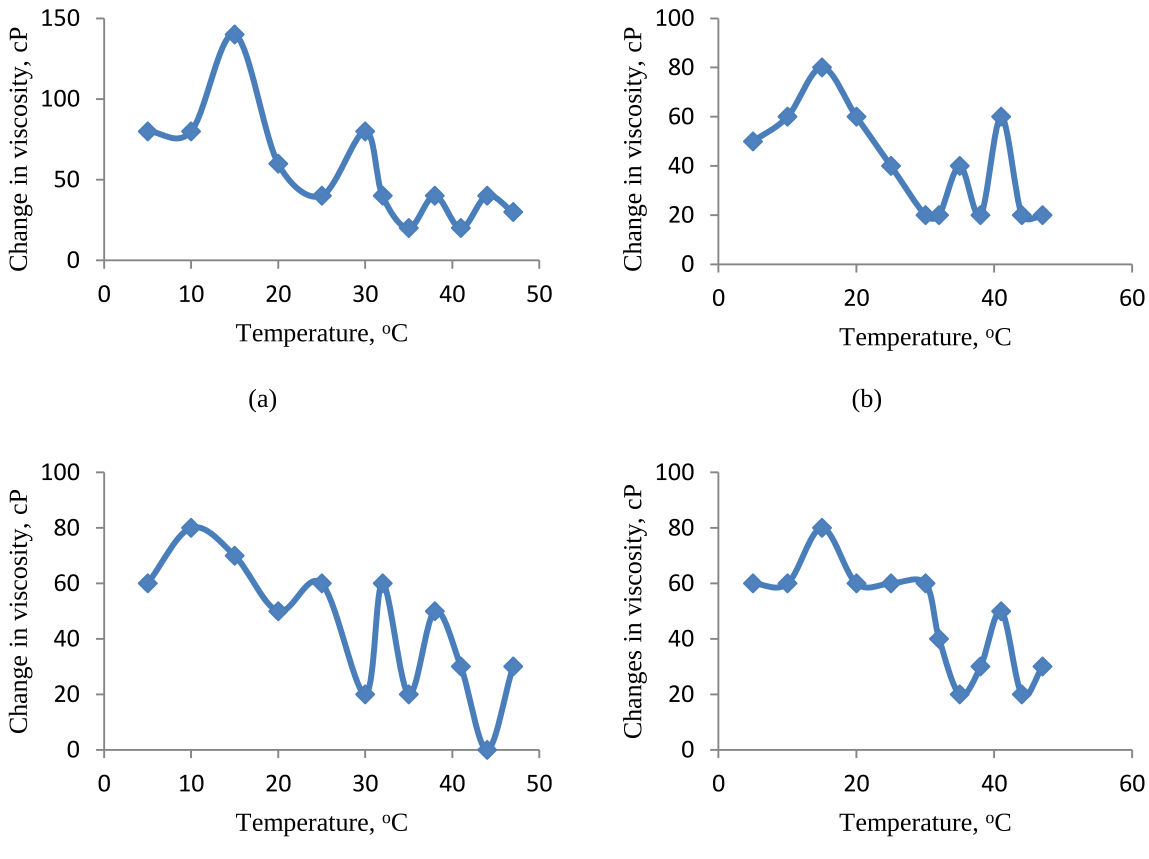


Figure 1. Viscosity of the crude oil

Figure 1 also shows that the crude oil with EVA co-MMA has the highest viscosity compared to the others in the range of 50 °C to 10 °C. The reason for this is that crude oil at high temperature behaves as a Newtonian fluid showing viscosity-temperature dependence. As the temperature of the crude oil falls below the WAT, precipitation of the wax crystals will rapidly increase the viscosity entering into the non-Newtonian fluid region. The yield stress of a wax-solvent mixture is believed to be a function of wax composition, and thermal and shear histories. A typical waxy crude oil would normally exhibit a pseudo-plastic with thixotropic properties [26]. The untreated crude oil is found to behave similarly to pseudo plastic fluid, wherein with increasing shear rate, the viscosity tends to decrease.

Figure 2 (a-d) shows the changes of the viscosity for the sample of the crude oils at different temperature with and without the wax inhibitor. The most significant value of viscosity was when the temperature decreased from 20 ℃ to 15 ℃ for the untreated crude oil sample, crude oil with EVA, and crude oil with EVA co-DEA. The viscosity changes for these three types of samples are 140, 80 and 80 cP respectively. Whereas, the highest viscosity recoded of the crude oil with EVA co-MMA was 80 cP, when the temperature was reduced from 15 ℃ to 10 ℃. From the trend of figure 2, it can be observed that at low temperature the viscosity has changed for both treated and untreated crude oil the viscosity at low temperature was higher compared to the viscosity at high temperature. This is because, the wax inhibitor reduces the change in viscosity of the crude oil below 25 ℃ and 23 ℃ since adding a wax inhibitor will change the fluid properties of the of crude oil [26-28].

Figure 2 (a-d) also shows that the crude oil with EVA has the lowest change of viscosity as the temperature is dropped from 25 °C to 5 °C, followed by the crude oil with EVA co-DEA, crude oil with EVA co-MMA. The highest change in the viscosity is observed when the temperature is dropped without inhibitor.



(a) (b)

(c) (d)

Figure 2. Change of viscosity at different temperatures (a) crude oil (b) crude oil with EVA (c) crude oil with EVA co-MMA (d) the crude oil with EVA co-DEA

3.3. *Determination of the crude oil pour point*

Table 3 shows the pour point temperature of the crude oil with and without wax inhibitor. From table 3, it can be concluded that at temperature 5-10 °C, the crude oil, crude oil with EVA, and crude oil with EVA co-MMA tend to harden or stop flowing. Whereas, for the crude oil with EVA co-DEA, it stops flowing at a lower temperature than the other at 0-5 °C. These values have confirmed that different types of inhibitor enhances pour point property of crude oil.

There was no significant difference in the pour point for the untreated crude oil, crude oil treated with EVA and crude oil treated with EVA co-MMA, this might be due the small amount of crude oil sample used. However, the crude oil sample treated with EVA co-DEA has a slightly lower pour point compared to the other samples , this is mainly because at lower temperature the crude oil with EVA co-DEA still have low viscosity and it may be due to the characteristic of EVA co-DEA in which it has structure similar to the nucleation site and co-crystallizing with the paraffin waxes ,therefore preventing the formation of waxes which increase the viscosity of the oil [29,30]. It can be concluded that EVA co-DEA wax inhibitor has proven to have better inhibition characteristics compared to the others. In addition to that ,it has shown the strongest Van Der Waals interaction between wax and the functional group in the wax inhibitor that causes the decrease in the WAT and increase of wax solubility.

Table 3. Pour point temperature

Temperature, °C	Crude oil only	Crude oil + EVA	Crude oil + EVA co-MMA	Crude oil + EVA co-DEA
50	/	/	/	/
47	/	/	/	/
44	/	/	/	/
41	/	/	/	/
38	/	/	/	/
35	/	/	/	/
32	/	/	/	/
30	/	/	/	/
25	/	/	/	/
20	/	/	/	/
15	/	/	/	/
10	X	X	X	/
5	X	X	X	X

whereby, X sign indicates that the crude oil stopped flowing when diverted horizontally for 3 to 5 second, meanwhile / sign indicates that the crude oil still can still flow.

3.4. Effect of EVA mass on the viscosity of the crude oil

In this section the EVA mass was increased to evaluate its effect on the viscosity of the crude oil as the temperature is decreased. Table 4 below illustrates the results. It was observed that increasing the amount of the EVA in the wax inhibitor formulation results in increased viscosity of crude oil. As the viscosity of the crude oil increases with the increase of EVA mass the cloud point of the crude oil remains unchanged, this proves that increasing wax inhibitor dose doesn't affect the wax precipitation temperature. Even if the amount of wax inhibitor is higher than it supposed to be, it will not affect the temperature of the crude oil to form a wax since the wax precipitation will occur when it reached certain temperature. In other words, the amount of the wax inhibitor will have no effect on the WAT of the crude oil except if the amount of the crude oil is increased drastically.

Table 4 shows that the crude oil with EVA co-MMA inhibitor still has the highest viscosity compared to the other inhibitor. The trend figure 2 (a-d) shows that the increasing in viscosity is also affected by the increasing in the concentration of the wax inhibitor and decreasing in temperature. As the viscosity increases the pour point of the crude oil also increases, however table 3 shows that the pour point does not increase as the viscosity of the crude oil sample is increased. This may be due to the concentration of the crude oil does not exceed the point where it changes pour point temperature to stop flow at high temperature.

Table 4. The effect of EVA mass on the viscosity of the crude oil

Temperature, °C	Viscosity, cP					
	Crude oil + EVA (3 g)	Crude oil + EVA (5 g)	Crude oil + EVA (3 g) + MMA	Crude oil + EVA (5 g) + MMA	Crude oil + EVA (3 g) + DEA	Crude oil + EVA (5 g) + DEA
50	860 ± 10	920 ± 12	940 ± 12	1000 ± 15	880 ± 10	900 ± 10
47	900 ± 10	960 ± 12	980 ± 12	1020 ± 15	900 ± 10	940 ± 10
44	920 ± 12	1000 ± 15	1000 ± 15	1040 ± 15	960 ± 10	980 ± 10
41	960 ± 12	1040 ± 15	1040 ± 15	1080 ± 15	1000 ± 15	1040 ± 15
38	1000 ± 15	1080 ± 15	1080 ± 15	1120 ± 18	1020 ± 15	1080 ± 15
35	1040 ±15	1140 ± 18	1120 ± 18	1180 ± 18	1060 ± 15	1140 ± 18
32	1080 ± 15	1180 ± 18	1160 ± 18	1240 ± 18	1120 ± 18	1200 ± 18
30	1140 ± 18	1200 ± 18	1220 ± 18	1300 ± 20	1180 ± 18	1260 ± 18
25	1200 ± 18	1240 ± 18	1300 ± 20	1340 ± 20	1240 ±18	1300 ± 20
20	1260 ± 18	1300 ± 20	1360 ± 20	1420 ± 20	1300 ± 20	1340 ± 20
15	1320 ± 20	1380 ± 20	1440 ± 22	1500 ± 22	1360 ± 20	1400 ± 20
10	1380 ± 20	1460 ± 22	1500 ± 22	1560 ± 22	1440 ± 22	1460 ± 22
5	1420 ± 20	1540 ± 22	1580 ± 22	1620 ± 25	1480 ± 22	1500± 22

3.5. Determination of the performance inhibitor efficiency

Table 5 shows the performance inhibition efficiency (%PIE) of EVA, EVA co-MMA and EVA co-DEA. From table 5 it can be concluded that the EVA co-DEA has the highest %PIE of 28.42% compared to the others.

Table 5. The results of performance inhibitor efficiency

Performance inhibitor efficiency (%PIE)		
EVA	EVA co-MMA	EVA co-DEA
25.32 ± 0.60	24.83 ± 0.50	28.42 ± 0.80

4. Conclusion

EVA co-DEA inhibitor has proven to be the best inhibitor that reduces the wax formation based on paraffin inhibitor efficiency value, cloud point, and pour point that is 28.42% at 10 ℃ and 5 ℃ respectively. This study explains the effected of the reduced molar mass of the inhibitor and its ability of reducing the pour point temperature of the crude oil. This can be explained in terms of the changes of the crude oil characteristic from the non-Newtonian to a Newtonian as the temperature decreases. Furthermore this study has also proven that the EVA can act as the main wax inhibitor to be mixed with other wax dispersant to form higher quality wax inhibitor however, not all wax inhibitors are suitable for such reaction. Therefore, it can be concluded that EVA co-DEA is better wax inhibitors compared to the EVA and EVA co-MMA. Meanwhile, EVA co-MMA has shown a lower PIE value and higher viscosity of crude oil compared to EVA that proves that, not all mixture of wax inhibitor with wax dispersant are able to enhance the performance of a wax inhibition compared to the primary wax inhibitor.

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