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Effect of solid contents and the ratio of EVA/ Octadecylacrylate blends on Paraffin Inhibition and pour point temperature of waxy crude oil

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Abstract

The best performing, most economical and safest is the most desirable triangle in the products used in the petroleum industry. Optimizing the performance of PPD increases the economy of the process; however, excess PPD may have adverse effect. Most of the PPDs have two parts; an oleaginophilic part that co- crystallizes with wax forming components and a polar component that limits the degree of co- crystallization. The present work has main target to study the effect of additives solid contents and composition on the flow characteristics of wax crude oil at low temperature below pour point temperature and paraffin inhibition (PI%). In this respect, different amount of octadecyl acrylate and ethylene vinyl acetate to give different blends with different solid content as follow: PPD-X1(1: 1.4), PPD-X2 (1:0.88), PPD-X3 (1: 1.15), PPD-X4 (1: 1.31), PPD-X5 (1:2) were prepared. These polymeric materials were characterized by FTIR and GPC. The performance of these additives as pour point depressants and flow improver for Egyptian waxy crude oil was evaluated through measurements of pour point paraffin inhibition. It was observed that the PPD-X5 the highest solid content and octa ratio multiple EVA ratio which leads to more effective performance that decrease pour point from 24 to 6 °C with 1200 ppm dose, good handling and highest paraffin inhibition (85 %).

Key words: solid contents, Paraffin Inhibition, pour point temperature, waxy crude oil.

1. Introduction

Millions of dollars are funded to prevent and remove wax deposition in the petroleum industry. Phase changes occur during the production and shipping of crude oil [1]. Physical properties of heavy oils, which contain a wide range of hydrocarbon components, differ greatly among which the flow property plays a significant role in production, transportation, storage, and refining [2, 3]. Wax deposits on pipeline walls cause several severe problems such as reducing the effective pipeline diameter and even pipeline clogging [4]. Thus, it is economically important to reduce the wax deposition effect. It is essential to maintain the Paraffinic crude oil temperature by heating or insulation that increases the energy consumption to prevent crude oil treatments [5]. Several previous studies have clarified that the PPDs effectiveness as flow improver based on different factors such as pendant chains and polymer backbones. Hence, by studying the compatibility of the chemical nature of PPD and the rheological behavior of crude oil it will lead to a better knowledge of the flow characteristics [5] Also, the molecular structure and Polydispersity index of PPD affect its performance [6]. Typically, PPDs contain polar and non-polar moieties [7]. The non-polar moieties are long-chain linear alkyl compounds containing 14-25 carbons that co-crystallize along with wax-forming compounds, while the polar moieties such as acetates or acrylates limit the degree of co-crystallinity. The ratio of monomer to monomer will be considered in the co-polymerization of wax and PPD. The crystalline and amorphous parts of PPDs are essential in determining compatibility [8]. Thus, analysis of PPD structural and molecular weight is critical to understanding flow assurance. Gel permission chromatograph (GPC) is used to determine the average molecular weight of the polymer, also H1NMR spectra and Fourier transform infrared spectroscopy (FTIR) is used to confirm the chemical structure of PPD, whereas the crystalline property is determined by X-ray diffraction [5, 9, 10, 11, 12].

Ethyl-Vinyl Acetate (EVA) Typical materials that are usually chosen to act as crystal modifiers are polymeric compounds. Ethyl-vinyl

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acetate (EVA) has been widely used as it has the ability to control the size of the wax crystals formed [13]. Haohao et al., [14] have studied the effects of EVA20, EVA30, EVA40 and EVA80 on the oil viscosity and the pour point at 4oC. It was found that EVA30 and EVA40 showed a higher viscosity reduction in comparison to EVA20 and EVA80 at the optimum condition and EVA80 has the lowest efficiency. On pour point reduction property, EVA40 exhibits the higher efficiency than EVA30 as the pour point depressant for the Brazillian crude oil. Ashbaugh et al., [15] have proposed that the efficiency of the additive to improve flowability decreases with the carbon number of the wax crystals. Jafari et al., [16] used scanning electron microscopy (SEM) to observe the wax crystal morphology. It was observed that EVA copolymers could greatly reduce the size of wax crystals and rearrange plate-like shape to a spherical and denser morphology [16]. Anisuzzaman et al., [17] have evaluated the performance of EVA with methyl methacrylate (MMA) and diethanolamine (DEA) and found that EVA co-DEA reduced the pour point of the crude oil by 5ºC and the paraffin inhibition efficiency at 28.4%. However, the viscosity of the crude oil did not reduce sufficiently. Alkyl Acrylate Copolymer A solid form of polymer acrylate polymer has also been used as a pour point depressant by Admiral et al., [18]. They found that the use of acrylate polymer could enhance the ability of a crude oil to flow in the pipeline. Alkyl acrylate that contains various sets of alkyl group with carbon number ranging from C14 to C26 is one of the most extensively explored classes for paraffin depressants. Previous studies have suggested that combining alkyl acrylate with the other co-monomers could further reduce the pour point of crude oils than using only alkyl acrylate polymer [19]. It has been proposed that the efficiency is influenced by the alkyl chain length and the polarity of the polymer [20]. Deshmukh and Bharambe [21] synthesized five different composite of n-alkyl acrylates-co-Nhexadecylmaleimide to investigate the rheological properties of Nada crude oil. Their result showed that the crude oil viscosity was reduced from 90.8 mPa.s to 11.27 mPa.s at 27 oC with additive used. However, the reduction was minimal at 21ºC. Indeed, there was an increased in viscosity for three types of the additives. However, the effectiveness of the additives was enhanced in reducing the pour point of the crude oil when the concentration and the alkyl chain were increased.

In this research, we studied effect of solid contents and the ratio of EVA/ Octadecylacrylate blends on paraffin inhibition and pour point temperature of waxy crude oil and its improvement in the process of production and transport to reduce the problems caused by high viscosity of crude oil and wax accumulation to improve production process technically and economically.

2. Experimental

2.1. Materials

General chemicals: Acrylic acid, octadecyl alcohol, *p*-toluenesulfonic acid (PTSA), hydroquinone, methanol, NaOH, xylene, benzoyl peroxide.

Ethylene vinyl acetate (EVA), the characterization of EVA is listed in table 1 .

Crude oil: Waxy crude oil was collected from Neag, Badr eldin Petroleum Company (BABETCO) Western Desert, Egypt. The physicochemical properties of waxy crude oil as a fingerprint are listed in Table 2.

Notice: All chemical used were commercial degree.

2.2. Preparation and characterization of poly octadecyl acrylate

2.2.1. Esterification of acrylic acid and octadecyl alcohol

Acrylic acid and octadecyl alcohol were added to round-bottom flask connected to a deanstarck system by 1:1 molar ratio in the presence of *p*-toluenesulfonic acid (PTSA) (0.5 mol %), and hydroquinone (3% w/w, in relation to acrylic acid). The temperature of the reaction medium is adjusted to 120 °C and kept under magnetic stirring for 8 h. the product was purified in methanol and washed several times with an aqueous solution of NaOH (5% m/V) then washed by distilled water and dried with anhydrous magnesium sulfate.

2.2.2. Polymerization of octadecyl acrylate monomer

In a two-necked round-bottomed flask, octadecyl acrylate was dissolved in a xylene solution and polymerization was performed by using benzoyl peroxide (1 mol%) as the initiator. The reaction was performed at 90 °C for 8 h, under nitrogen atmosphere and magnetic stirring. Afterwards, the xylene was evaporated under low pressure. The poly octadecyl acrylate (PODA) was then purified in chloroform/methanol, and vacuum dried at 75 °C, until constant weight.

2.3. Preparation of different blends between poly octadecyl acrylate and ethylene vinyl acetate copolymer

Into (500 ml) three-necked flat bottom flask equipped with a reflux condenser, we dissolved poly octadecyl acrylate in xylene, the solution was stirred on hot plate with magnetic stirrer at 90 °C and then ethylene vinyl acetate copolymer was added on patches for 3 h and the blend still was stirred until the ethylene vinyl acetate was completely soluble. We made these steps by different amount of octadecyl

acrylate and ethylene vinyl acetate to give different blends with different solid content.

2.4. Characterization of poly octadecyl acrylate

2.4.1. Gel permeation chromatography (GPC)

Gel permeation chromatography (GPC) Average molecular weights (Mw and Mn) of resultant copolymers were determined by Waters Model 515/2410 using a gel Permeation Chromatographic technique (GPC, Waters, America) and a Styragel column calibrated with polystyrene standards and series 2410 refractive index detector using THF as mobile phase in a Xylene GPC system, with flow rate: 1 mL min^{-1} and temperature: 40°C at EPRI.

2.4.2. FTIR Characterization analysis Spectroscopic measurements

The chemical structures of the prepared copolymers were confirmed by means of Fourier transform infrared spectrometer (Nicolet IS-10 FTIR spectrometer) at Egyptian Petroleum Research Institute (EPRI) over the wavenumber of 4000–400 cm^{-1}.

2.5. Evaluation and Characterization treated and untreated crude oil

2.5.1. Evaluation of the prepared copolymers as pour point depressants (PPDs) in crude oil [22]

Evaluation of the prepared copolymers was carried out according to Standard Test Method for Pour Point of Petroleum Products (ASTM D97), with some adjustments. This analysis is primarily performed manually, using a thermometer to read the system temperature and check the fluidity of the system at periods of 3 ° C temperature drops until the 'no-flow' temperature is reached (the sample is no longer moving when in a horizontal position for 5 s). The pour point is then stated as non-flow temperature plus 3 ° C. The tests were conducted in triplicate. The calculated error for these analyzes also took into account uncertainties in the thermometer (0.6 ° C) and equilibrium (0.0002 g).

Different dosages (800, 1,000 and 1,200 ppm) of each of the formed esters and amide were injected in the sample of waxy crude oil at 60°C.Then, the sample of treated crude oil was undergone pour point determination according to ASTM D 97.

2.5.2. Paraffin inhibition (PI %)

Copolymer additives efficiency was evaluated by Cold Finger experiments at PPD Laboratory, Chemical Services and Development Center (CSDC), EPRI, which are often used as a simple means to approximate the deposition process in the flow line by simulating the actual field conditions in the laboratory.

2.5.3. Wax content (Wt %)

Wax percentage of the crude oil was measured by the UOP Method (46/64).

2.5.4. Asphaltene content (Wt %)

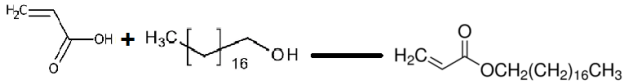
Asphaltene content was isolated using n-heptane according to the IP 143/84 procedure

3. Results and discussion

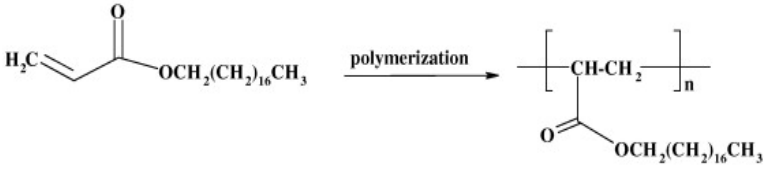
3.1. Preparation and characterization of poly octadecyl acrylate

3.1.1. FT-IR Characterization of PODA, ODA monomer and PPD-X5.

Infrared spectroscopy was used to confirm the chemical structure of the prepared species. Figure 1 show the FTIR spectra of octadecyle acrylates monemr, PODA and PPD-X5. From fig. 1 strong bands appeared at 1731 cm^{-1} attributed to C = O ester expansion, and bands at 2849-2959 cm^{-1} for aliphatic C - H of the alkyl group. C-C bands at 1636 and absorbance at 1384, 1302, 992, 888 and 810 cm^{-1} were absent in the PODA spectrum [19]. The structure of the prepared PODA was confirmed by the strong alky group bands and double bond absence [23].



Eq. (1): Esterification of acrylic acid and octadecyl alcohol



Eq. (2) Polymerization of octadecyl acrylate monomer

3.1.2. Gel permeation chromatography GPC

Table 4 and figure 2. Show average molecular weights, Polydispersity, and the number of molecular weights of the prepared PODA. Commonly, there are two reaction rates for the free radical polymerization reactions: at the beginning, the rate of the polymerization highly increases with the reaction time, then the rate decrease steadily. The data

illustrated from table 1 show that the average molecular weight of the prepared PODA polymer reaches (≈ 38153) and The Polydispersity index is 1.023, that provides conclusive indication of the particle and polymer chain growth during polymerization.

3.2. Evaluation and Characterization of treated and untreated crude oil

3.2.1. Chemical composition analysis of tested crude oil

From data represented in Figure 3, the total carbon average of paraffins is C-11to C-24 with broad molecular weight distribution. This means that n-paraffins have a tendency to form deposits and block the flow of crude oil by forming interlocking networks. Hence, the polymer side chains should have lengths similar to the distributions of paraffin wax to interact with paraffin and prevent the wax networks formation.

The chemical structure of polymeric additives should contains both polar group such as ester, amine, amide and hydroxyl group and non-polar moieties such as long chain hydrocarbon to be efficient as flow improver and wax dispersant. The pour point depressant (PPD) mechanism depending on the ability of PPD to modify the morphology of the wax crystal in crude oil by change crystal shapes from widely interlocking plates to more closed crystals by co crystallizing with the wax. The more combatable the polymer structure to wax components, the higher efficiency and the better ability of the polymer to bind to wax components and form a barrier for networking of wax particles [24].

Furthermore, the performance of PPD affected by crude oil characteristics itself including quantity and type of wax present in crude, the chain length, chain shape (branched or linear) of wax, total wax content.

Table 1: Characterization of EVA

characteristics	Method	Result
Vinyl acetate content, %	WT FTIR (internal method)	38-41
Melt Index, g/10 min	ISO 1133/ ASTM D1238	48-62
Density, g/cm^3	ISO 1183	0.96
Melting point, °C	ISO 11357-3	55
Vicat softening point, °C	ISO 306/ ASTM D1525	<40
Ring & Ball temperature, °C	ASTM E28/ NF EN 1238	97
Elongation at break, %	ISO 527/ ASTM D 638	900-1100
Tensile strength, at break, MPa	ISO 527/ ASTM D 638	
Hardness Shore A	ISO 868/ ASTM D 2240	50

Table 2: The physicochemical properties of waxy crude oil

Test	Method	Result
Density at 20°C (g/cm^3)	ASTM D1298	0.8410
Kinematic viscosity, (c.st.) at 40°C	ASTM D445	5.58
Pour point (PP), °C	ASTM D 97	24
Wax content, wt %	UOP 46/64	28.5
Asphaltene content, wt %	IP 143/84	1.5
n-Paraffins, wt %	ASTM D2887(GLC)	57.9
Average carbon number (n)	IP 372/85 (GLC)	12.34
Water content, wt %	IP 74/70	0.25
Ash content, wt %	ASTM D 482	0.01
API gravity at 60 F	ASTMD-1298	36.75

Table 3: Preparation of different blends with different solid content

Chemical	**PPD-X1**	**PPD-X2**	**PPD-X3**	**PPD-X4**	**PPD-X5**
Soilid content, %	32	40	35	40	40
Dynamic viscosity, cp@50°C	99	XX	514	540	111
Poly Octa, %	18.7	18.7	18.7	22.7	26.7
EVA, %	13.3	21.3	16.3	17.3	13.3
Solvent (Xylene), %	68	60	60	60	60

Table (4): GPC results of PODA

	Retention Time	Mn	Mw	Mp	Mz (Daltons)	Mz+1 (Daltons)	Poly-dispersity
1	22.419	9484	38153	42030	84076	130648	1.023

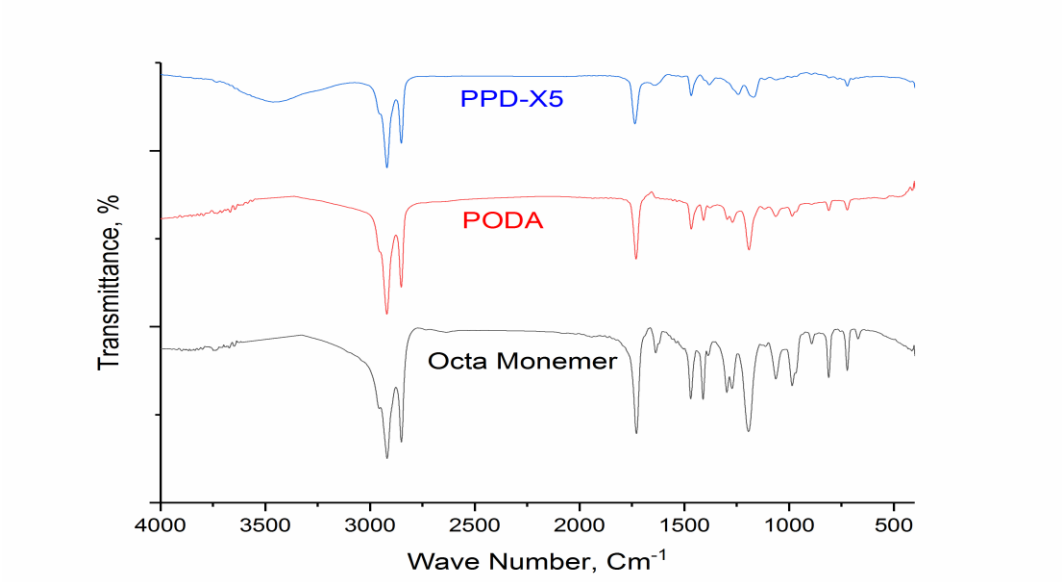


Fig.2: GPC results of PODA

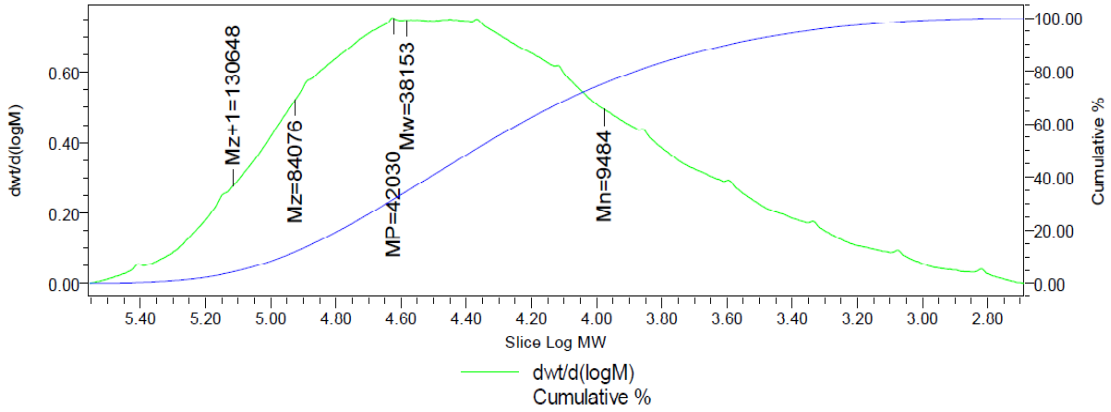


Fig.3: The carbon number distribution of n-paraffin in crude oil.

3.2.2. The evaluation of the prepared copolymer esters as PPD

Different dose from each polymeric additives, namely (800, 1000 and 1200) ppm was injected to sample prepared from crude oil to evaluate the performance of prepared copolymer. The results of pour point measurement are given in Table 4. The data illustrated from (Table 4) explain that the alkyl chain length affects the performance of polymeric additives. In general, the polymeric additive with higher efficiency is PPD- X5 showing a maximum of 9 °C depression in pour point at high dose of additive (800 ppm), that is, low effectiveness as PPD, while in PPD-X1, PPD-X2 and PPD-X3 the extent of depression in pour point reaches about 18°C at the same dose (8000 ppm). These results of pour point also showed that the depressions in pour point increase with solid contents increase. From data in table [4] can found that the ratios of EVA comparing with octa as follow: PPD-X1(1: 1.4), PPD-X2 (1:0.88), PPD-X3 (1: 1.15), PPD-X4 (1: 1.31), PPD-X5 (1:2) i. e the PPD-X5 the highest solid content and octa ratio multiple EVA ratio which leads to more effective performance and good handling.

Table 5: The pour point of untreated crude oil and treated crude oil

No.	Chemical name	Solid Content, %	Viscosity, cP @ 50°C	Performance			Paraffin inhibition, % at 800 ppm
				800 ppm	1000 ppm	1200 ppm	
1	PPD-X1	31.8	99	18	15	12	55
2	PPD-X2	40	XX	18	15	9	59
3	PPD-X3	35	514	18	15	15	62
4	PPD-X4	40	540	15	12	12	68
5	PPD-X5	40	110	9	9	6	85

3.2.3. Paraffin inhibition (PI %)

Wax inhibitor that exhibit characteristics as wax crystal modifiers are chemical compounds that have the same chemical structure as the wax in the crude oil. Typical compounds selected as the modifiers are usually polymeric compounds consist of one or more hydrocarbon chain molecules that are similar to the wax, but with a polar portion. This type of materials will agglomerate with the wax by bonding the hydrocarbon chains of wax molecules on the crystal lattices. In addition, the inhibitor will hinder the wax crystal growths, which in turn lower the cloud point of the crude oil. Wax deposits were tested on a "Cold Finger." About 400 ml of crude oil (before and after additive addition) was kept in a water bath at cloud point temperature and circulated to copper coil in another water bath. Residual adhering oil was removed after 1 h by taking out the finger and putting it in acetone. Finally, the residual wax deposit was removed completely from the cold finger and weighed (Table 4):

$$PI\ \% = \frac{Weight\ of\ blank - Weight\ after\ injection}{Weight\ of\ \ blank} \times 100$$

It is found that the PPD-X5 additive gives high paraffin inhibition about 85% (Table 4). This is attributed to the composition of polymeric PPD added where, the PPD-X5 has the highest solid content and octa ratio multiple EVA ratio which leads to more effective performance and good paraffin inhibition.

4. Conclusion

The present work has main target to study the effect of additives solid contents and composition on the flow characteristics of wax crude oil at low temperature below pour point temperature and paraffin inhibition (PI%). In this respect, different amount of octadecyl acrylate and ethylene vinyl acetate to give different blends with different solid content as follow: PPD-X1(1: 1.4), PPD-X2 (1:0.88), PPD-X3 (1: 1.15), PPD-X4 (1: 1.31), PPD-X5 (1:2) were prepared and evaluated. The data revealed that:

- High molecular weight polyalkyl acrylates are synthesized by esterification of acrylic acid and octadecyl alcohol.
- Different blends from octadecyl acrylate and ethylene vinyl acetate with different solid content of PPD-X1(1: 1.4), PPD-X2 (1:0.88), PPD-X3 (1: 1.15), PPD-X4 (1: 1.31), PPD-X5 (1:2) were prepared and evaluated.
- PPD-X5 has the highest solid content and octa ratio multiple EVA ratio which leads to more effective performance (paraffin inhibition about 85%) and good handling.
- Typical compounds selected as the modifiers are usually polymeric compounds consist of one or more hydrocarbon chain molecules that are similar to the wax, but with a polar portion.
- A difference in solid contents leads to a difference in polymeric PPD pour point values thereby PI efficiency.

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