Studies on the efficiently of some polymers based on octadecyl acrylate as crude oil additives

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Abstract: This paper describes synthesis and evaluation of polymeric additives for improving the flow properties of crude oil. The additives were prepared by the free-radical initiated polymerization of octadecylacrylate with methyl methacrylate and acrylic acid or styrene. Polymerization was performed by initiator dibenzoyl peroxide in xylene at 90°C. The influence of additives structure, composition and molar weigh. Crude oil samples from the fields of QURON located in EGYPT were used for evaluation of the prepared additives. It was found that the additives efficiency was considerably influenced by their properties.

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1.Introduction

The natural of crude oil creates many problems during its transporting through long distance pipelines. At lower temperature crystallization of waxes creates problem in restorability of crude oil after shutdown the pipeline. The waxes generally crystallize as trapping the remaining oil in cage -like structures (1) and cause the blockage of pipeline. Crude oil having high wax content causes many problems during production, storage and transportation (2). The high wax crude oils are characterized by high pour point, high viscosity, high gel strength and abundant wax deposits (2). When the waxy crude temperature is below the wax appearance temperature (WAT), wax crystals separate out from oil solution. If being continually cooled, more and more waxy crystals appear and interlock to form a network, which entraps the liquid oil, resulting in the gelation of crude oil ⁽³⁾. This decreases the flow of the crude and increases the resistance to its flow, overloads the pump, reduces productivity and possibly plugs the well. For this reason, the inhibition of paraffin deposits is required to maintain the necessary well productivity (4). Several options are available including stream heating, blending with lighter cutter stocks, mechanical scraping and use of chemical additives (5-6). The preferred option is the use of chemical additives referred to as wax crystal modifiers, also known as pour point depressants (PPD), flow improvers (FI) and paraffin inhibitors⁽⁵⁾. Pretreatment with PPD is an attractive solution for transportation of waxy crude oil via pipelines. Polymeric additives which satisfy most of the requirements to act as pour point depressants and flow improvers for crude oil were synthesized and evaluated such as; poly long alkyl methacrylate, alkyl naphthalene copolymer (7), esters of oleic acid-maleic

anhydride copolymer⁽⁸⁾, copolymers of maleic anhydride and esters of n-alkyl alcohols with oleic acid and methacrylic acid ⁽¹⁾, poly(styrene-co-alkyl methacrylate) ⁽²⁾, styrene-alkyl itaconate copolymer ⁽⁹⁾, hexa-triethanolamine oleate esters ⁽¹⁰⁾, poly-α-olefins (11) and (12), ethylene–vinyl acetate copolymers (EVA)⁽¹³⁾, terpolymers like styrene/ dodecyl methacrylate / octadecyl methacrylate having different molar ratios (11) and (14). Also some diblock copolymers e.g. polyethylene-polyethylene propylene (PE-PEP) (15-16) and poly (ethylene-co-butene) (PEB)⁽¹⁷⁾ and ⁽¹⁸⁾ are reported as good PPD. PPD modify the size and shape of crystals and inhibit formation of large wax crystal lattices (5,19-20). Polymeric additives known as flow improvers or pour point depressants are generally used to lower the pour point, viscosity and yield stress of crude oil. In pipeline transportation, these additives improve the fluidity of waxy crude and reduce the extra pumping cost ⁽⁸⁾. The designing of better additives requires a good understanding of the crystallization behavior of the paraffin molecules in the hydrocarbon fluids. Any additives which are as effective as PPD may be ineffective to reduce the viscosity and yield stress and enhance the flow ability⁽⁹⁾. All PPDs are structured so that the part of the molecules is like the paraffin wax crystals, this part functions by providing the nucleation sites and co-crystallizing with the paraffin waxes, while the other part of the structure, dissimilar to the wax crystals, blocks the extensive growth of the wax matrices ⁽²¹⁾. PPD additives may work by number mechanisms ⁽²²⁾ and ⁽²³⁾. They may serve as nucleating agents if they self-assemble or aggregate above the precipitation temperature of the wax. Facilitating nucleation can lead to a higher wax precipitation temperature. However, a lower precipitation

temperature may result if there are many small nucleation sites that are not large enough to be detected. PPD may also bind to larger crystals and prevent particle-particle interactions from forming aggregates, volume spanning networks, or deposits. This can be termed steric stabilization or adsorption. PPD additives do not work by changing the amount of wax that comes out of solution (22,24-25), but by altering the crystal growth and structure. The following factors play an important role in the efficiency of pour point depressants (26-27); (a) the number of pendant alkyl side chains and the length and distance between them are the important factors, (b) the solubility of the additives (which are generally polymers) in crude oil which depends on their average molecular weights, (c) if additives are copolymers then monomer to monomer ratio should be taken into consideration. (d) amorphous and crystalline parts of additives are very important in determining their efficiency, and (e) physical and chemical stability of additive (28). The first object of this work is to prepare Octadecylacrylate from stearyl alcohol and acrylic acid by using of Para toluene sulphonic acid and then copolymers with methyl methacrylate, acrylic acid and/or styrene. The second object is to evaluate their efficiency toward decreasing the pour point of the waxy crude oil. The third object is to perform these polymers as flow improvers via dynamic viscosity and the rheological parameters.

2-Experimental

2.1. Materials

The following chemicals were used; Methyl methacrylate monomer (MMA) and styrene was supplied by Navol; and stearyl alcohol from SASOL; Acrylic acid from BASF; Dibenzoylperoxide (DBPO) was obtained from Merck; Xylene, Toluene and methanol solvents were obtained from Carloerba reagents; p-toluene sulfonic acid and hydroquinone which were supplied from Sigma Aldrich chemicals company.

2.2. Crude oil used

Egyptian waxy crude oil was submitted from Qarun Petroleum Company. Its physicochemical properties are listed in Table-1. Their properties and *n*-paraffin distribution are shown in Fig. 1, Table- 2.

2.3.1. Preparation of Octadecyl acrylate:

Octadecyl acrylate were prepared by the reaction of acrylic acid and stearyl alcohol in a molar ratio 1.2:1, using toluene as solvent, p-toluene sulfonic acid (PTSA) as catalyst, hydroquinone as inhibitor, water was separated a zeo tropically using Dean–Stark apparatus ⁽²⁹⁾. The alcohol, toluene and hydroquinone were added to a four-necked flask, then heated to 60 °C. After the alcohol completely dissolved, acrylic acid and PTSA were added, and

gradually heated to $115-120\,^{\circ}\text{C}$. The reaction was stopped after a theoretical amount of water separation. Reaction products were washed several times with an aqueous solution of Na_2CO_3 (5 % m/V) until the under layer liquid became clear, then washed by distilled water, at last vacuum distilled using a rotary evaporator and vacuum dried.

Table 1: Complete Analysis for untreated Crude Oil.

Test	Method	crude
Zone		L.B
Field		Qarun
Specific Gravity @ 60/60 °F	ASTM D-	0.8343
	1298	
API Gravity @ 60 °F	ASTM D-	38.1
	1298	
Total Water Content %		1.6
Free Water %	IP 74/70	Nil
Emulsified Water %		1.6
Pour Point °C	ASTM D-97	21
Kinematics Viscosity @ 40 °C	ASTM D-	7.6843
cSt.	445	
Conradson Carbon residue % Wt	IP 13/94	3
Ash content %Wt	IP 4/94	0.012
Sediment by extraction % Wt.	IP 24/55	0.02
Sulfur Content %Wt	ASTM D-	0.3676
	4294	
Asphaltene Content% Wt	IP 143/57	1.05
ASTM Distillation		
I.B.P °C		48
Recovery @ 100 °C ml		5
Recovery @ 125 °C ml		11
Recovery @ 150 °C ml		17
Recovery @ 175 °C ml	IP 24/25	23
Recovery @ 200 °C ml		29
Recovery @ 225 °C ml		34
Recovery @ 250 °C ml		40
Recovery @ 275 °C ml		46
Recovery @ 300 °C ml		52

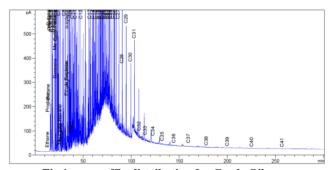


Fig 1: *n*-paraffin distribution for Crude Oil

Table 2: Distribution of n-paraffin fractions in the Crude Oil sample :

Crude On Sample.					
Fraction	Weight %				
Ethane	0.0413				
Propane	0.2213				
I-Butane	0.1922				
n-Butane	0.4541				
i-Pentane	0.3401				
n-pentane	0.7346				
N-Hexane	2.9116				
Me-cyclo-Pentane	1.4992				
Benzene	0.0554				
C7	2.4960				
Toluene	0.0939				
C8	9.5161				
Ethyle Benzene	0.1549				
m,p-xylene	1.5601				
o-xylene	0.1138				
C9	5.6966				
C10	3.9881				
C11	5.6393				
C12	5.0039				
C13	3.7021				
C14	4.0304				
C15	4.4288				
C16	4.0219				
C17	4.4978				
C17	3.3699				
C19	3.0019				
	2.8206				
C20 C21					
C21	2.9425				
C22 C23	3.3108				
C23	2.3564				
C24 C25	2.0631				
C25	3.9349				
C26	1.5406				
C27	1.3919				
C28	1.5405				
C29	1.7358				
C30	1.5503				
C31	1.8129				
C32	1.3596				
C33	1.0327				
C34	0.6130				
C35	0.7897				
C36	0.5465				
C37	0.1883				
C38	0.2027				
C39	0.1050				
C40	0.3973				

2.3.2 Polymerization of additives

All additive samples were prepared by free radical solution polymerization in xylene using dibenzoyl peroxide as the initiator⁽³⁰⁾. Polymerization was performed in a reaction flask equipped with a condenser, mechanical stirrer, and temperature controller. Polymerization reaction was carried out in nitrogen atmosphere at 90°C for 5 h, under continuous stirring. During that time, more than 95% of monomer

was polymerized. The resulting homogeneous product was then cooled down to room temperature and used as additive without any further treatment. To determine monomer-to-polymer conversion and additive composition, we isolated the ultimate polymer by repeated precipitation with methanol, filtering, and dissolving in xylene. The final product was vacuum dried at 60 °C. The ¹H NMR spectra indicated the absence of unreacted monomer in the isolated polymer.

2.4. Characterization of the prepared compounds.

The average number of carbon atoms in octadecyacrylate esters and additive compositions were obtained from Proton Nuclear Magnetic Reonance (H- NMR) Spectra using a 300 MHZ Varion NMR300 SPECTROMETER using DMSO as a solvent, at room temperature with TMS as internal standard.

Molar weights based on calibration with monodispersed polystyrene standards (Polymer Laboratories) were determined by GPC (Varian HPLC, Model 8500) using a set of 4-μ styrogel columns with THF as solvent, at room temperature. Testing of flow improver additives was carried out by measurements of pour point (ASTM D 97)..

Viscosity and flow curves (rheograms) were measured by means of the BROOKFILD UV-III ULTRA rheograms rotating viscosimeter.

Paraffin distribution in crude oil sample was performed by means of Agilent Technologies 7890A GC System.

3. Results and discussion

Flow improver additives of defined and controlled structural characteristics were prepared by solution polymerization of Octadecylacrylate with methyl meth acrylate (MMA), styrene (St) and/or acrylic acid (AA). We studied the influence of polymerization conditions on additive composition and properties, and consequently the effects of additives on the pour point and rheological behaviour of various crude oil samples.

3.1. Polymerization conditions and additive composition

polymer samples of Octadecylacrylate with mthyl methacrylate and acrylic acid, were prepared. For comparison, we also prepared Octadecylacrylate with methyl methacrylate and Styrene; and prepared Octadecylacrylate with methyl methacrylate, Styrene and acrylic acid. Experimental conditions and additive composition are given In Table 3.

Additive properties are presented in Table 3. The chemical structure of the synthesized compounds is shown in Fig. 2.

B
$$\begin{array}{c}
CH^{-}CH_{2} \\
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Fig 2: Structural representation of (A) octadecylacrylate(OCDA); (B) polymer of OCDA -MMA-AA; (C) polymer of OCDA-MMA -ST and (D) polymer of OCDA-St-MMA -AA.

Figure options

The structural determination of the polymer was carried out by recording the 1 H NMR spectra using DMSO as solvent and TMS as internal standard. The peak assignments for the polymers are as follows: (a) octadecyl acrylate polymer, δ : 3.9 ppm, $-\text{OCH}_{2}$ – groups; 1.2 ppm, $-\text{CH}_{2}$ – groups of the side chain; 0.9 ppm, and $-\text{CH}_{3}$ groups of the side chain; (b) styrene polymer, δ : 6.9–7.2 ppm, $-\text{C}_{6}\text{H}_{5}$ groups; 1.1–2.4 ppm, $-\text{CH}_{2}$ – Groups; (c) acrylic acid polymer, δ : 10 –13.2 ppm, -CH–COO– groups; 1.5–2.1 ppm, $-\text{CH}_{2}$ – groups and (d)methyl methacrylate polymer, δ : 1.4–2.1 ppm, $-\text{CH}_{2}$ – and $-\text{CH}_{3}$ groups. Polymerization conditions, conversion, polymeric additive compositions, molar weight of additive, and pour point of the untreated crude Oil and that treated with of prepared additive, including one commercial additive, are shown in table-2.

3.2. Characterization of the chemical structures of additives

Table 3: property of additive composition and molar weight

Sample code	Monomer compo	Conv. (%)	Molar weight (g mol 10 ⁻³)				
	OCDA	St	AA	MMA		$M_{ m w}$	$M_{\rm n}$
В	0.8		0.2	0.15	97.84	18162	8388
С	0.8		0.6	0.15	97.71	21900	9130
D	0.8	0.2		0.15	97.5	27411	10981
Е	0.8	0.6		0.15	96.91	24845	7789
F	0.8	0.3	0.2	0.15	97.52	26331	10958
G	0.8	0.2	0.3	0.15	97.3	27036	10024

The chemical structures of the prepared polymers were studied by means of infrared spectrometry using Perkin.

Elmer IR spectrophotometer model IR1420. The IR spectra of octadecylacrylat and apolymer prepared shows the absorption peaks at 1730 cm⁻¹, 1630 cm⁻¹, 1722 cm⁻¹, 929 cm⁻¹,1118 cm⁻¹, 2840 cm⁻¹,1467 cm⁻¹, 1165 cm⁻¹,1250. The average molecular weight, number average molecular weight, and molecular distribution were determined using a gel permeation chromatographic analysis and the results are listed in table 3.

3.3. Comparison of structures of wax crystals in waxy crude oil beneficiated with and without a PPD

In Fig. 3, micrograph of wax crystal in virgin crude oil is shown in Fig. 3(a) while Fig. 3(b) display the wax crystal aggregates in oil beneficiated with PPD. While in Fig. 3(a) wax crystals become bigger and have tended to aggregate as, the particles which increase and more wax precipitated, so which will affect the flow behavior of waxy crude oil.

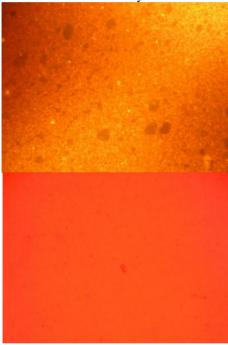


Fig 3: Polarized microscopy images of (a) virgin crude oil (b) crude oil beneficiated with C additive at 1000 ppm concentration.

3.4. Pour point

All the waxy crude oils eventually become non-fluid on chilling. This is caused by the precipitation of wax crystals as thin plates and needles. These depressants obstruct the lateral crystal growth as a result of their adsorption on the precipitating $\max_{(31,32)}$.

The results of pour point were presented in Table -4 as pour point reductions in relation to the pour point of the virgin (oil without any additive) crude oil minus pour point of crude oil with additives.

Table 4.: Influence of additive on crude oil pour point

Additive	concentration	in	Pour point reduction °C					
ppm			В	C	D	E	F	G
300			12	9	18	15	18	18
500			9	6	15	12	15	15
1000			6	3	9	6	12	9

The data in Table 4 indicates that all the polymeric additives are quite effective in reducing the pour point of crude oil. Additive D is most successful in reducing the pour point from 21 °C to 3 °C (reduction in pour point by 18 °C) at 1000 ppm dose and to 6 °C at 500 ppm. F has lowered the pour point by 3 °C at the concentration of 300 ppm and at 1000 ppm dose reduction is up to 12 °C.

The higher dispersing activity of these additives may be due to the combined effect of carbonyl oxygen of acrylice acid and polarity group in the structure.

3.5. Rheological studies

The viscosity of crude oil was determined at different doses of concentration and temperature using BROOKFIELD viscometer, results are given in Table -5 which shows that as the concentration of additive polymer increases the viscosity decreases. Rheograms of the virgin and PPD beneficiated crude oil shows that the rheogram of shear rate vs. shear stress, that waxy-paraffinic crude oils, a high pour point and possess the non-Newtonian flow properties at the temperature equal to or lower than the pour point, due to wax crystallization and gel formation of their heterogeneous matrix. Thus, a yield stress rises and increase of viscosity occurs. The rheological properties cease to be constant and viscosity varies as a function of shear rate (33-34). The strength of the generated gel depends on the temperature and cooling rate of the crude oil condensate. The lower cooling rate means the higher gel strength and viscosity. It is important to ascertain whether cooling occurs under static or dynamic conditions. A low cooling rate under static conditions results in pronouncedly unfavourable flow properties for the particular oil, because paraffin has all thermodynamic prerequisites for generating a solid crystal structure. This means that undisturbed transportation of fluid with increased wax content requires a modification of flow properties. In this study, the additives influence on the rheological behaviour of crude oil was established by measurements of viscosity and rheograms (shear rate/shear stress) of the untreated crude oil and treated crude oil selected additives (using the rotating viscosimetry method).

Table 5: Brook field viscometric data for crude oil

Code	Additive concentration (ppm)	Temperature (°C)	Apparent viscosity (cPs)	Plastic viscosity (cPs)	Yield value (D/cm ²)
Blank	00	5	2411.49	58.9	31.83
		10	452.90	45.9	7.3
		15	146.97	22.5	4.33
		5	216	45	5.5
	500	10	46	32	1.5
В		15	35	26	0.44
		5	44.99	25.9	1.02
	1000	10	26	13.2	0.33
		15	23,9	18.98	0.24
		5	216	45	1.8
	500	10	52	27	1.3
С		15	34	19	0.55
C		5	54	38.99	0.41
	1000	10	48	21	0.23
		15	27	15	0.22
	500	5	45	89	1.1
		10	36	35	0.98
D		15	27	27	0.48
ט	1000	5	90	82	0.38
		10	83.5	76	0.28
		15	41	53	0.20
	500	5	173.5	24	4.75
		10	42	22	0.94
		15	18	19	0.35
Е	1000	5	140	26	5.56
		10	33	19	1.3
		15	18	18	0.3
F	500	5	240	46	6.2
		10	96	37	5
		15	58	33	2.1
		5	180	28.6	4.8
	1000	10	78	24	3.34
		27	49	20	1.22

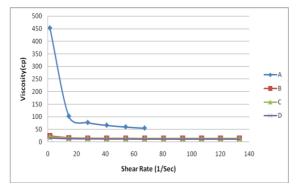


Fig 4: Viscosity versus shear rate for Crude oil Untreated (A), treated Crude oil by (B, C, D) with 1000 ppm of additives at 10 $^{\circ}$ C.

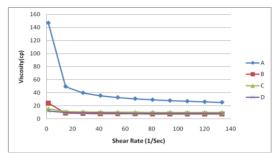


Fig 5: Viscosity versus shear rate for Crude oil Untreated (A), treated Crude oil by (B, C, D) with 1000 ppm of additives at 15 $^{\circ}$ C.

Flow curves of the untreated crude oil samples suggest the non-Newtonian fluid properties. It was found that the additives reduced the viscosity of both condensate samples to about the same extent and that a decrease was significant at low shear rates. Graphic presentation of shear rate/shear stress curves of the crude oil untreated and treated samples are shown in Fig. 6 and Fig. 7.

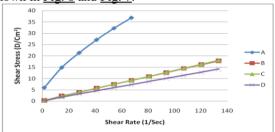


Fig 6: Shear stress versus shear rate for crude Oil untreated (A) treated Crude oil by (B, C, D) with 1000 ppm of additives at $10\,^{\circ}$ C.

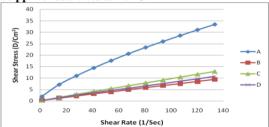


Fig 7: Shear stress versus shear rate for crude Oil untreated (A) treated Crude oil by (B, C, D) with 1000 ppm of additives at 15 $^{\circ}$ C.

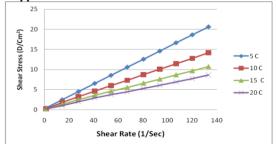


Fig 8: Shear stress versus shear rate for crude Oil treated Crude oil by (C) with 1000 ppm of additives at 5° C, 10° C, 15° C, 20° C.

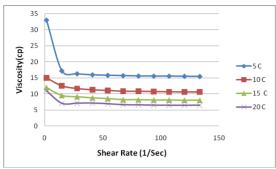


Fig 9: Viscosity versus shear rate for treated Crude oil by (C) with 1000 ppm of additives at 5° C, 10° C, 15° C, 20° C.

From the rheological data shown in Fig.4 to Fig. 9, it is obvious that additives reduce the shear stress with minimum yield values (τ_0), which are different for the crude oil. The yield value in both condensate samples containing additives is lower in comparison with the untreated samples.

4. Conclusions

Crude oil flow improver additives were prepared by the free-radical initiated polymerization of octadecyl acrylate with methyl metha acrylate, styrene and/or acrylic acid in aromatic solvents (xylene) using dibenzoyl peroxide as initiator. The evaluation of additives was carried out by their application to the crude oil. The additive efficiency depends on their composition, molar weight and crude oil properties. The terpolymers of octadecyl acrylate with methyl methacrylate and acrylic acid are very efficient flow improvers crude oil. From the obtained rheological data it is obvious that additives reduce the shear stress with minimum yield value which is lower in comparison with the untreated crude oil sample.

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