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SCIENTIFIC PAPER

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NOVEL ALLYL-ESTER-BASED POLYMERS AS FLOW IMPROVERS FOR WAXY CRUDE OIL

Article Highlights

- Preparation and polymerization of para-decyloxy allyl benzoate (I10)
- The homo- (HI10) and copolymer (MHI10) were evaluated as effective pour point depressants
- MHI10 exerted the highest effect on the pour point depression and rheological parameters
- The efficiency of the additives increased by increasing their concentrations from 1000 to 5000 ppm

Abstract

The deposition of paraffin wax from crude oil at low temperatures due to wax deposition, high pour point, high viscosity, and weak flow capability is among the critical and persisting challenges faced by the petroleum industry. In this study, a new para-decyloxy allyl benzoate (I10) was prepared, polymerized into HI10, and copolymerized with dioctadecyl maleate into MHI10 via the free-radical polymerization method employing azobisisobutyronitrile and toluene as the initiator and solvent, respectively. The prepared monomer was characterized by spectroscopic analyses (Fourier-transform infrared (FT-IR)) and proton nuclear magnetic resonance. Further, the polymers were characterized by FT-IR, and their average molecular weights were determined by gel permeation chromatography. The prepared compounds were taken in different concentrations and then tested as flow improvers of Qarun waxy crude oil using pour point depression and rheological parameters. The results of this test indicated that MHI10 exerted the highest effect on pour point reduction and the rheological parameters (yield value and apparent viscosity). Moreover, an increase in the efficiency of the additives was observed after increasing their concentration from 1000 to 5000 ppm.

Keywords: flow improver, pour point depressant, allyl benzoate, waxy crude oil.

As temperature decreases in the winter season, paraffin crystals begin to grow and create a crystalline net that traps the molecules of liquid hydrocarbon; this process continues until the crude oil cannot flow [1-3]. The precipitation of paraffin is one of the main challenges in the production and transport of crude oil via pipelines. The wax crystals of paraffins are generally irregularly shaped (platelet- or needle-like) and can form a continuous three-dimensional (3D) network at relatively low concentrations (~1 wt.%) [4,5].

Paraffin deposition significantly affects the production process as it entails removing the formed deposits, maintenance, increased costs of production and transport, well shutdown, and the failure of equipment [6]. Among the different available techniques and methods for eliminating or alleviating problems associated with waxy crude oil transportation and/or production, using flow improvers as additives is the most used method to change the rheological behaviors of petroleum. There are several types of improvers for improving crude oil flow. Among them, there are improvers based on surface active agents [7,8]. These improvers employ the adsorption mech-

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anism of wax crystal surfaces such as petroleum sulfonate, polyoxyethylene, and alkylamine. Polymer-type-based additives such as alkyl acrylate copolymer and vinyl acetate copolymer and their derivatives [9,10] are the main additives used for improving the flowability of high-wax-content crude oils at low temperatures. Notably, various crude oils are not affected in a similar fashion by polymer additives, and not all polymers are necessarily effective on all crudes [11]. It is presumed that effective additives have a good match between the polymer and crude for parameters such as composition, structure, and content. The inhibition of wax crystallization occurs by cocrystallization or adsorption and nucleation in the presence of a polymer [9]. Many copolymers are utilized as inhibitors of wax precipitation [7,12,13]. Further, poly-(ethylene-co-vinyl acetate) (EVA), poly(ethylene-co-butene) [14,15], poly(α-olefins), poly(alkyl acrylates) [16], poly(alkyl methacrylates) [17,18] and maleic anhydride copolymers containing vinylic esters are some of the most efficient commercially available pour point depressants (PPDs). Allyl compounds comprise a large group of ethylenic compounds exhibiting unique reactivities. Allyl methacrylate (AMA) is an ester of methacrylic acid; it is used as a flow improver in oil additives. In this work, we aim to synthesize a para-decyloxy allyl benzoate ester homopolymer (HI10) and a copolymer (MHI10) with dioctadecyl maleate (DM). The synthesized compounds were evaluated as flow improvers for Egyptian waxy crude oil. Through rheological improvement and pour point depression, the yield point and plastic viscosity values were correlated to the pour point results.

EXPERIMENTAL

Materials

Stearyl alcohol (99%), dicyclohexylcarbodiimide (DCC), 4-(dimethylamino)pyridine (DMAP) and 4-hydroxybenzoic acid were obtained from Sigma Aldrich; maleic anhydride (99%) was obtained from Acros; toluene and ethyl alcohol were obtained from Morgan; 2,2′-azobisisobutyronitrile (AIBN, 98%) was obtained from Merck Chemicals; and 1-bromodecane (97%) was obtained from Alfa Aesar. Crude oil was obtained from the western desert of Egypt (QN field - Qarun Company).

Preparations

Preparation of para-decyloxy benzoic acid

First, 1-bromodecane (0.033 mol) was added drop-wise to a solution of ethyl-4-hydroxy benzoate (0.033 mol) with stirring in 50 mL of ethanolic potassium hydroxide (0.04 mol). The stirring proceeded for 2 h at 60 °C and then overnight at room temperature. The resulting ester was directly hydrolyzed by adding potassium hydroxide (0.33 mol) and heating under reflux for 2 h. Then, the cold solution was added to cold dilute hydrochloric acid under stirring. The resulting acids, after being crystallized twice from glacial acetic acid, were confirmed to be pure by thin-layer chromatography and exhibited phase-transition temperatures that agreed with the temperatures reported in literature [19].

Preparation of para-decyloxy allyl benzoate (I10)

Molar-equivalent quantities of both allyl alcohol and para-decyloxy benzoic acid were dissolved in methylene dichloride. To the resulting solution, DMAP was added as a catalyst and then DCC was added, and the solution was left to stand overnight at room temperature with stirring. Thereafter, the solution was filtered, the solute was distilled, and the residue was recrystallized by acetic acid [19].

Syntheses

Synthesis of (DM)

The dioctadecyl maleate (DM) was prepared using molar equivalent quantities of maleic anhydride and stearyl alcohol in the presence of toluene, which was the solvent, and *p*-toluene sulfonic, which was the catalyst. The reaction was performed for 3.5 h at 95 °C [20].

Synthesis of para-decyloxy allyl benzoate polymer (HI10)

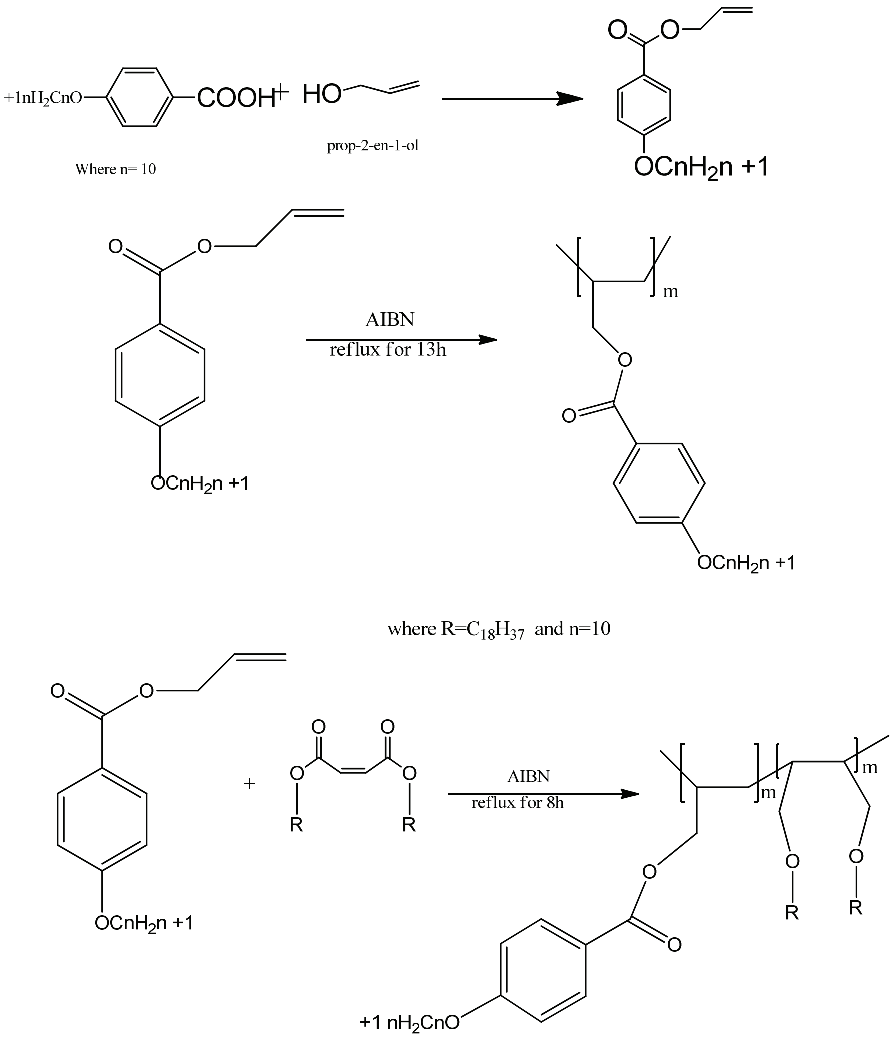
The homopolymer was synthesized *via* free-radical polymerization in a nitrogen atmosphere with constant stirring. The polymerization was conducted in a toluene solution for 13 h at 120 °C utilizing AIBN (0.1 wt.%) as the initiator. The solid polymer was separated and purified, and the synthesis process used for the polymer is shown in Scheme 1.

Synthesis of para-decyloxy allyl benzoate-DM copolymer (MHI10)

The copolymer was synthesized via free-radical polymerization in a nitrogen atmosphere with constant stirring. A mixture containing an equal amount of the monomer ((I10) and DM in 30 mL toluene was prepared, and then, the initiator (AIBN, 0.1 wt.%) was added. The reaction proceeded with continuous stirring for 8 h at 120 °C. The solid polymer was separated and purified, and the synthesis process is shown in Scheme 1.

Characterizations of the prepared compounds

The prepared polymers (HI10, MHI10) were characterized using Fourier-transform infrared (FT-IR)



Scheme 1. Syntheses of HI10 and MHI10.

spectroscopy. The spectra were recorded on a PerkinElmer Spectrum 100 FT-IR spectrometer utilizing a KBr cell; four scans were taken, and the wavenumber range was 450-4000 cm^{-1}. The proton nuclear magnetic resonance (^{1}H-NMR) spectra were recorded on a Varian 300 (Cairo University) spectrometer utilizing tetramethylsilane (TMS) as the internal stander.

Evaluation tests

The effects of the prepared polymers as flow improvers (QN field) on the Egyptian waxy crude oil were evaluated through the rheological parameters and pour point depression. The pour point was measured according to the American Society for Testing and Materials Standard Test Procedure for Pour Point of Crude Oils (ASTM D97), employing the Koehler cloud and pour point refrigerator at different concentrations (1000-5000 ppm) of the prepared additives in the crude oil. The rheological measurements were conducted with a Brookfield DV-III Ultra Rheometer that was equipped with a thermostat cooling system and temperature control. The Brookfield rheometer was employed to measure the dynamic viscosity of virgin and additive-treated crude oils, which was prepared using optimum doses of additive, at different temperatures below and above the pour point (20, 24 and 32 °C), and the yield stress and dynamic viscosity values were determined. The microscopic structures of the crude oils untreated/treated with the prepared additives were observed using polarized-light microscopy; an Olympus BX41-P polarizing microscope was used to observe the wax crystal morphologies.

RESULTS AND DISCUSSION

Characterizations of the crude oil

Table 1 shows the physicochemical properties of the studied crude oil.

Table 1. Physicochemical properties of the crude oil - analysis of the QN field oil

Test	Method	Result
Specific gravity @ 60/60 °F	ASTM D1298	0.8217
API gravity @ 60 °F	ASTM D1298	40.7
BS & W, vol.%	ASTM D4007	0.1
Water content, vol.%	IP 74/70	0.1
Pour point, °C	ASTM D97	24
Viscosity kinematics @ 40 °C cSt.	ASTM D445	5.72
Wax content, wt.%	UOP 46/64	10.08
Conradson carbon residue, wt.%	IP 13/94	1.1
Ash content, wt.%	IP 4/94	0.005
Sediment by extraction, wt.%	IP 24/55	0.001
Sulfur content, wt.%	ASTM D4294	0.38
Average carbon number (*n*)	IP372/85 (GLC)	18.16
Resin content, wt.%	ASTM 5062-09	3.56
Asphaltene content, wt.%	IP 143/57	0.74

Confirmation of the structures for the prepared compounds

IR spectroscopy

In Figure 1a, the C-H stretching vibrations of the CH_3 and CH_2 groups were observed as strong absorption bands at 2918 and 2850 cm^{-1}. The carbonyl ester stretching bands appeared at 1718 cm^{-1} [21] because of the π-bond characteristic of the carbonyl group, the C-O stretching vibration was observed at 1253 cm^{-1}, and the ν(C=C) aliphatic band appeared at 1643 cm^{-1}. Regarding DM, ν(C=C) appeared at 1641 cm^{-1} and the carbonyl ester stretching bands appeared at 1729 cm^{-1}.

Figures 1b and c show that the C=C aliphatic band disappeared in the homopolymer and copolymer. This indicated the conversion of the monomer into a homopolymer. The carbonyl ester stretching bands of DM and I10 coappeared as a broad band at 1719 cm^{-1}.

^{1}H-NMR spectroscopy

Figure 2 shows the peaks, which are assigned as follows: δ at 0.8 ppm for the CH_3 protons; δ at 1.25 ppm for the $(CH_2)n$, (k) (l); and δ at 7.7 and 6.8 ppm for the benzene ring.

Molecular weight measurements

Molecular weights and polydispersities of the prepared homopolymer and copolymer were measured, and the results are presented in Table 2.

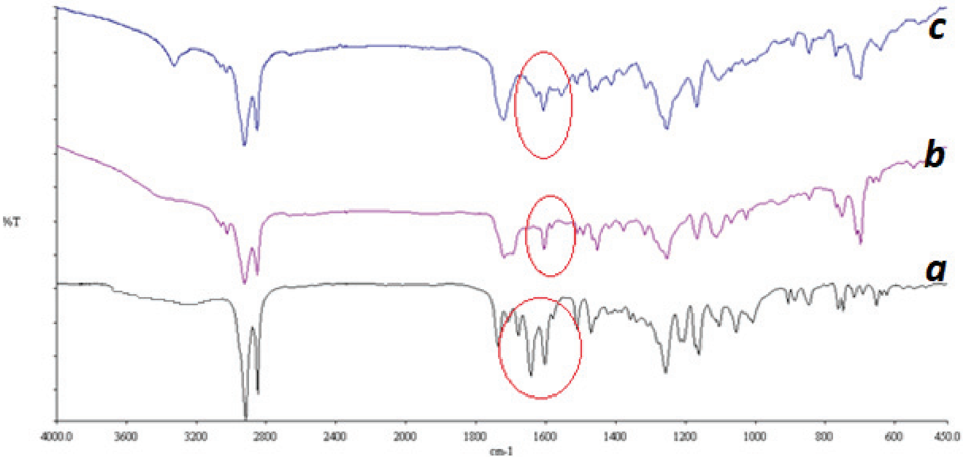


Figure 1. IR spectra of: (a) I10, (b) HI10, and (c) the copolymer (I10, HI10, and MHI10).

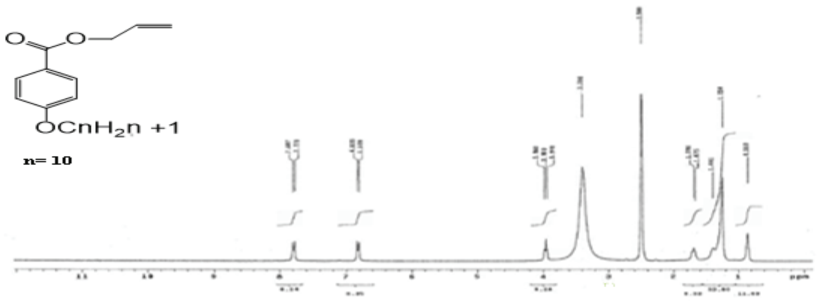


Figure 2. ^{1}H-NMR of I10.

Table 2. Molecular weights and polydispersities of the polymer and copolymer

Additive	M_n	M_{Z+1}	M_w (g/mol)	Polydispersity
HI10	6100	6920	6684	1.096
MHI10	9080	15750	10415	1.147

Evaluations of the prepared compounds as flow improvers

Pour point

The pour point is the lowest temperature at which crude oils can be stored or handled without congealing. The pour point is generally 10-20 °C lower than the cloud point (the temperature at which wax appears).

The data in Table 3 shows that all the prepared additives were effective in reducing the pour point of Qarun crude oil. The pour point depression was directly proportional to the concentrations of the prepared additives in all the additives. Additive concentrations increased from 1000 to 5000 ppm as the pour point temperature decreased. The maximum pour point depression was attained at a concentration of 5000 ppm. The pour point further decreased as the concentrations of the additives were further increased, implying that the additives could crystallize with the paraffin wax and modify their crystal structures within this concentration range. The prepared additives reduced the pour point temperatures from 24 to 9 °C (ΔP = +15 °C). The copolymer was more efficient because it contained a long aliphatic DM side chain that increased the cocrystallization with the wax molecules during the formation of the wax crystals, thereby directly affecting the efficiency of the polymer additive. Further, increasing the polar part (the two ester groups) inhibited the further agglomeration of wax, thus reducing or (in the best cases) preventing the wax crystal formation process.

Table 3. Effects of the structures and concentrations of the prepared compounds on the pour points of the crude oil

Polymer	Dose (ppm)				
	0	1000	2000	3000	5000
HI10	-	21	18	18	15
MHI10		18	15	15	9

The highest reduction in the pour point, *i.e.* a pour point depression from 24 to 9 °C (ΔP = +15 °C), was achieved with MHI10, where the interaction of the long alkyl groups (C18) with the paraffin fraction of the oil occurred through an adequate matching between them as the average number of carbons in the paraffin wax of Qarun crude oil is 18 (Table 1) [22]. The pour point depression efficiency was enhanced by the presence of a benzene ring in all the additive structures. The benzene ring matches well with the aromatic resins and asphaltene, thus increasing the solubility of the additive and interaction between the crude oil paraffin and additive [23].

Viscosity measurements

The chemical treatment of crude oils with PPDs is a general practice in the oil industry to maintain an acceptable state of fluidity in the crude oil even at low temperatures. The prepared additives interacted with the crude oil; consequently, they modified the morphology and crystal structure of the wax, and the formed crystal aggregates were easily destroyed when the oil was subjected to shear test and produced particles with shapes and sizes that were different and conducive to a different rheological behavior [24,25].

The performances of the additives as flow improvers in the tested crude were evaluated through rheological measurements at optimum concentrations (5000 ppm). The tests were performed for treated and untreated crude oils at temperatures below and above their pour points: 20, 24 and 32 °C. The shear revealed the effects of the additives on the viscosities and rheological behaviors of the waxy oils [26,27]: an increase in the shear rate reduced the apparent viscosity of the waxy oils [28]. Generally, lowering the temperature of crude oils increases their pour points, yield stress, viscosity, and progressive wax deposition [29]. The yield stress measurements were determined from the relationship between the shear rate and shear stress values, which were measured using the Brookfield viscometer. The yield value is an essential viscosity parameter, which offers beneficial information about the flow of crude oils below their pour point.

The shear stress-shear rate and viscosity-shear rate data of the untreated and additives-treated crude oils are plotted in Figures 3-5, which illustrate the fitted experimental rates, and an interception with the *Y*-axis represents the yield value. For the untreated crude oil yield stress and plastic viscosity and the values of the treated crude oil, it was observed that the shear stress sharply increased at all the tested temperatures by increasing the shear rate so that the cold flow pattern follows non-Newtonian pseudoplastic rheological behavior.

Increasing the shear stress sharply by increasing the shear rates of the treated and untreated crude oils caused a partial breakdown of the gel wax structure in the crude oil matrix, which caused a steep increase in the shear stress. The additives reduced the dynamic viscosity, and the decrease was more significant at lower shear rates than at higher ones. The apparent viscosity was constantly obtained at high shear rates, where the equilibrium steady-state was also obtained. When the optimum concentrations of the selected additives were applied, they precipitated with the wax crystals and modified their morphologies, thereby inhibiting their aggregation and depressing the pour point of the crude oil. This behavior is reflected by the significant drop in the yield values. Figures 3-5 indicate that all the cold-flow properties were improved by increasing the concentration of the additive in the sample. Further, an increase in the chain length [30] also corresponded to improved efficiency. The decrease in the viscosity depended on the temperature and additive concentration.

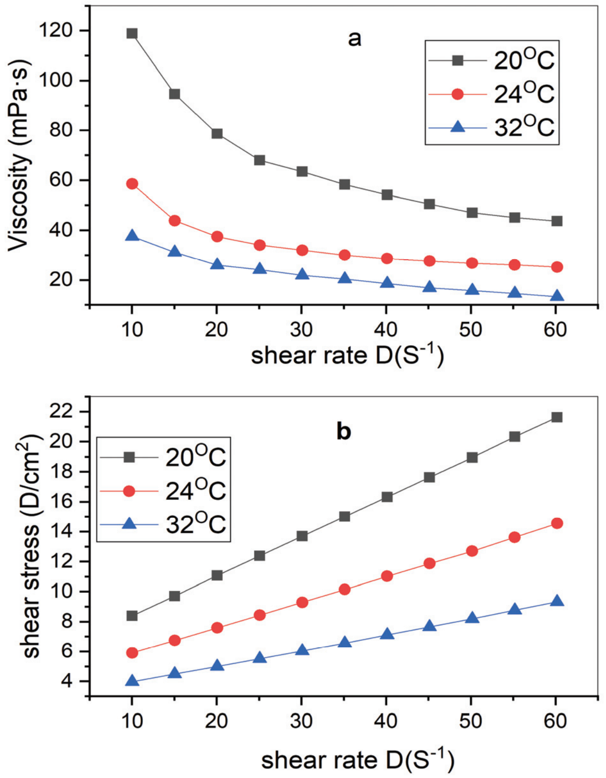


Figure 3. a) Relation between the shear rate and viscosity of the untreated crude oil; b) relation between the shear rate and shear stress of the untreated crude oil.

The depression in the yield stress was reflected by the apparent viscosity values, which were notably depressed compared to those of the untreated crude oil (Table 4). The obtained results of the viscosity and pour point correlated well. This can be explained in terms of the ability of the additives to establish a strong interaction with the wax crystals to modify their structure and form small crystals. Furthermore, the additives inhibited the formation of the agglomeration of wax crystals, thereby depressing the pour point.

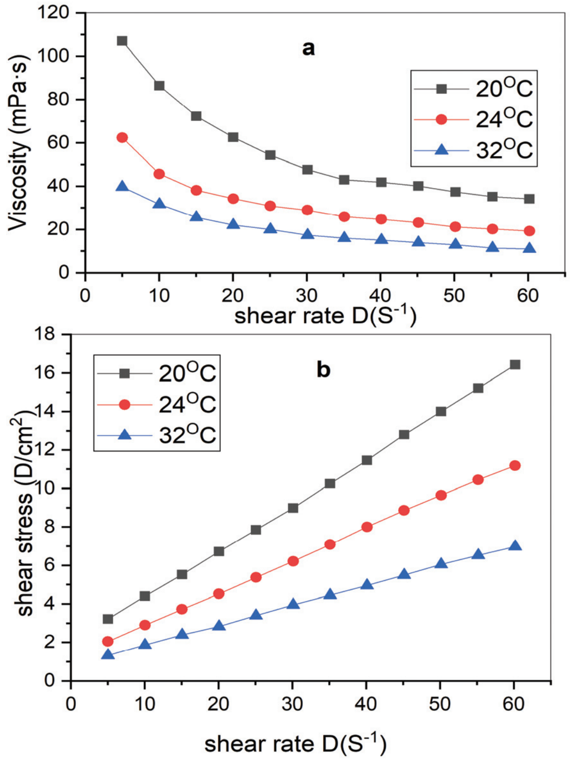


Figure 4. a) Relation between the shear rate and viscosity of HI10; b) relation between the shear rate and shear stress of HI10.

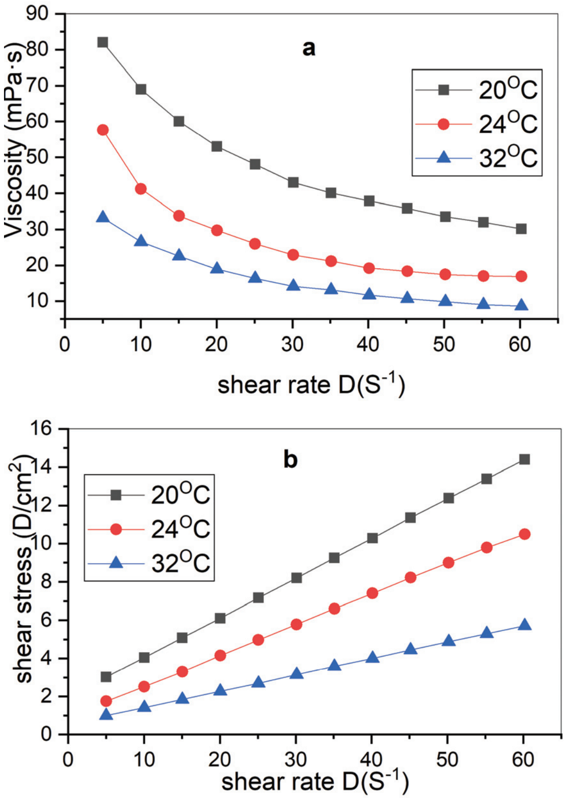


Figure 5. a) Relation between the shear rate and viscosity of MHI10; b) relation between the shear rate and shear stress of MHI10.

From the previously discussed results, MHI10 achieved better rheological enhancement than HI10. This can be explained as follows: the increase in the high polarity of oxygen in the ester groups of DM along the polymer chain might have prevented the agglomeration of the wax crystals in the crude oil. Additionally, the interaction of the long alkyl groups (C18) with the paraffin fraction of the oil occurred through an adequate matching between them, because the average number of carbons in the paraffin wax of QN crude oil is 18 (Table 1) [22]. Therefore, the flow improvers (additives) reduced the pour point, viscosity, and yield stress by cocrystallizing with normal paraffin molecules to inhibit the growth of wax crystals, implying a balance between the rates of crystal growth and cocrystallization [31].

Optical microscopy analysis

Photomicrography is a fast and simple evaluation tool that can reveal the modification that was induced by the wax dispersants and flow improver according to the type of waxy crude oil. When the waxy crude oil was cooled below its cloud point, its wax constituent tended to separate from the liquid phase and crystallize.

Photomicrography analysis is also applied to understand the flow characteristics in terms of the modification of the morphology of the wax crystal through additional laboratory tests. The decrease in the size and morphology of the wax crystals is due to the action of the PPD molecules that acted as nucleation centers in a manner that could induce the formation of many small crystals (Figure 6).

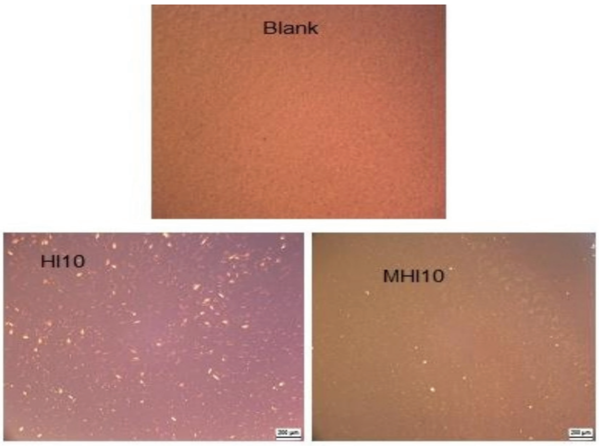


Figure 6. Photomicrographs of the untreated crude oil and the crude oil that was treated with 5000 ppm of the prepared polymers.

This might be because the additive cocrystallizes with the wax crystals and thus prevents the growth of the wax crystals, thereby keeping crystals small and apart. Thus, the waxy modifier correlated well with the improved flow parameters and pour point.

CONCLUSIONS

From the results obtained in this work, the following conclusions can be derived:

- The prepared materials could function as flow enhancers of petroleum crude.
- The higher the concentration of the additive, the lower the pour point and viscosity of the crude oil.
- Co-polymerization with DM resulted in a very effective polymer that was more effective than the homopolymer and ester.
- The result indicated that the most effective additive quantity for all prepared polymers is 5000 ppm.

Photomicrography, as a simple and rapid analytical tool, was employed to elucidate the changes in the wax structure that were induced by the PPD additives. It confirmed that the wax crystals were generally dispersed according to the concentration of the additive.

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NAUČNI RAD

NOVI POLIMERI NA BAZI ALIL-ESTRA KAO POBOLJŠIVAČI PROTOKA ZA VOSKASTU SIROVU NAFTU

Taloženje parafinskog voska iz sirove nafte na niskim temperaturama, zbog visoke tačke stinjavanja, visokog viskozitet i slabe sposobnosti tečenja je među kritičnim i stalnim izazovima sa kojima se suočava naftna industrija. U ovom radu, pripremljen je novi para-deciloksi alil benzoat (I10), polimerizovan u HI10 i kopolimerizovan sa dioktadecil maleatom u MHI10 metodom polimerizacije slobodnih radikala koristeći azobizizobutironitril i toluen kao inicijator i rastvarač, redom. Pripremljeni monomer je okarakterisan spektroskopskim analizama (FT-IR) i protonskom nuklearnom magnetnom rezonancom. Dalje, polimeri su okarakterisani FT-IR, a njihove prosečne molekulske mase su određene gel permeacionom hromatografijom. Pripremljena jedinjenja su uzeta u različitim koncentracijama i zatim testirana kao poboljšači tečenja Karunove voskaste sirove nafte korišćenjem smanjenja tačke tečenja i reoloških parametara. Rezultati ovog testa su pokazali da je MHI10 imao najveći efekat na smanjenje tačke tečenja i reološke parametre (granično smičuće naprezanje i prividni viskozitet). Štaviše, povećanje efikasnosti aditiva je primećeno nakon povećanja njihove koncentracije sa 1000 na 5000 ppm.

Ključne reči: poboljšivač tečenja, depresor tačke tečenja, alil benzoat, voskasta sirova nafta.