# Petroleum and Coal

## Synergistic Effect Between Some Copolymers and Organic Solvents to Improve the Flow Properties of Waxy Crude Oil

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**Abstract**

Wax deposition is a common problem in oil pipelines and production systems. The modification of the wax crystal habit is useful in the transportation and processing of crude oil at low temperatures. Various pour point depressant (PPD) additives can help with this modification via a variety of mechanisms. Hexadecyl acrylate and NAFOL 1822B acrylate are synthesised in this work by reacting a hexadecyl alcohol/NAFOL 1822B alcohol blend with acrylic acid, and two copolymers are synthesised by copolymer hexadecyl acrylate with vinyl acetate and another copolymer NAFOL 1822B acrylate with methyl methacylate. They characterized by IR spectroscopy and GPC was used to calculate their molecular weights. These copolymers were tested with various solvents for their efficacy as a crude oil pour point depressant and flow improver. The solvents utilised in this study were xylene, n-hexane, kerosene, and triethanolamine (TEA) for the wax dispersing wax operation. The effect of different percentages of these solvents with copolymer additions in the crude oil sample on the crude oil flow characteristics was assessed. All of the solvents tested enhanced crude oil flow properties. Blends of kerosene and triethanolamine (TEA) with the copolymer NAFOL 1822B acrylate (NA) -methyl methacrylate (MMA) were more effective than the other solvents as a crude oil pour point depressant.

*Keywords*: Copolymers; Solvents; Additives; Pour point depressant; Viscosity; Crude oil.

## 1. Introduction

Transportation of crude oil is considered as a difficult and highly technical operation. To deal with the high viscosity, which is one of the main challenges in pipeline transportation [1-2], more efficient and commercial methods are required It is well known that wax crude oils have a high viscosity and a high pour point, making production, transportation, and refining more difficult, especially at low temperatures [3-4]. As a result, different crude oil flow improvers have been used to improve heavy oil [5-6]. Dilution with lighter crudes or alcohols, as well as heating, are some of the common methods, but it depends on the feedstock of the lighter crudes, and the cost of using alcohols is very high. In general, these additives can be classified as crude oil flow improvers as long as they improve the flowability of crude oil. Pour point depressant and flow improver are the two most common types of crude flow improver. Pour point depressant and viscosity reduction are the two most prevalent types of crude flow improvers [7-8].

Polymers with a wax-like paraffinic part and a charged component, such as polyalkyl acrylates and methacrylates, ethylene vinyl acetate copolymers, copolymers and homo of alpha olefins, alkyl fumarate-vinyl acetate copolymers, and alkyl esters of styrene-maleic anhydride copolymers [9-11], are commonly used chemical additives. According to [12], the process of oil refinement contains negative factors such as large molecular weight, long molecular chain, and high thermo stability of these polymers.

In this case, crude oil is small molecular additives are required to act as a viscosity reduction. The structural features of gelled waxy crude oils have recently been studied in order to resume oil pipeline transit [13-14]. The first goal of this research was to use a copolymer ingredient to improve the flowability of waxy crude oil and lower its pour point. As a result, a hexaacrylate-

vinyl acetate copolymer and a NAFOL 1822B acrylate-methyl methacrylate copolymer (hexaacry-late-vinyl acetate copolymer and NAFOL 1822B acrylate-methyl methacrylate copolymer) were prepared. The second goal was to investigate the rheological behavior of two copolymers in different solvents, as well as the pour point depression of this molecule when mixed with waxy crude oil.

## 2. Experimental

## 2.1. Materials

Aldrich chemical provided hexadecyl alcohol, methyl methacrylate (MMA), acrylic acid, vinyl acetate, p-toluene sulphonic acid, and toluene as analytical reagents, which were used exactly as received. Benzoyl peroxide, the initiator, was re-crystallized from methanol. CONDEA chemical company provided a linear long-chain alcohol blend and (NAFOL 1822 B) with the typical analysis listed in Table 1. Qarun Petroleum Company submitted Egyptian waxy crude oil (CO). Table 2 lists their physicochemical properties. Gas chromatography analysis was used to determine the n-paraffin distribution of the isolated waxes in accordance with ASTM D 2887 standards.

Table 1. Typical analysis of linear long chain alcohol blends (NAFOL)

| Properties | NAFOL 1822B | |
|---|---|---|
| Alcohol composition (wt.%) | $C_{16 - OH}$ | 0.1 |
| | $C_{18 - OH}$ | 5.0 |
| | $C_{20 - OH}$ | 16.5 |
| | $C_{22 - OH}$ | 77.6 |
| | $C_{24 - OH}$ | 0.8 |
| Average carbon number (calculated) | $C_{av} = 21.5 \sim 22$ | |
| Density (g/cm$^3$) approx. | at 80$^o$C = 0.802 | |
| Solidification point ($^o$C) approx. | 64 | |
| Flash point ($^o$C) approx. | 204 | |
| Ester No. (mg KOH/g) | 0.14 | |
| Acid No. (mg KOH/g) | 0.05 | |
| Iodine No. (mg I/100 mg) | 0.31 | |
| Water (Wt.%) | 0.04 | |

Table 2. Physical characteristics of waxy crude oil

| Properties | Method | Crude oil |
|---|---|---|
| Specific gravity at 60/60$^o$F | IP 160/87 | 0.8557 |
| Kinematics viscosity at 35$^o$C (cSt) | IP 71/80 | 15 |
| Pour point (pp), $^o$C | IP 15/67(80) | 25 |
| Sulfur content (wt%) | IP 266/87 | 0.21 |
| Total paraffins content (wt%) | Urea adduct | 20.75 |
| n-paraffin (wt%) | GLC | 20.05 |
| Iso-paraffins (wt%) | GLC | 0.70 |
| Asphaltene content,wt.% | IP 143 | 00.728 |
| Wax content,wt% | UOP 46/64 | 10.920 |

## 2.2. Synthesis of additive

### 2.2.1. Synthesis of monomers

Synthesis of n-alkyl acrylic acid (0.3 mol) was esterified with a series of n-alkanols with increasing number of carbon atoms (hexadecyl alcohol C16, and NAFOL 1822 B (Cav= 22) (0.2 mol) in a round bottomed flask equipped with a Dean and Stark trap to remove the reaction water azeotropically as it formed. The reaction was carried out at reflux temperature

in the presence of toluene (100 mL) as a solvent P-toluene sulfonic acid was used as a catalyst, and hydroquinone was used as a polymerization inhibitor. The two prepared monomers (hexadecyl acrylate and NAFOL 1822 B acrylate) were then purified by distilling off the solvent at the end of esterification at low pressure. The reaction mixture was then extracted with n-hexane, washed with distilled water to remove the catalyst and any unreacted acid until the washing became neutral, and the organic layer separated.

### 2.2.2. Preparation of copolymers

Two copolymers, P1 copolymer additive of hexadecyl acrylate (HA)-vinyl acetate (VA) and P2 copolymer additive of NAFOL 1822B acrylate (NA)-methyl methacrylate (MMA), were pre-pared by free radical polymerization in the presence of dibenzoylperoxide (0.5 wt percent) as an initiator, which was dissolved in toluene and added to the reaction mixture in equal metered portions every 0.5 hour during the course of the reaction. At the start of polymerization, the reaction mixture was nitrogen flushed for twenty minutes before being gradually heated to reflux temperature under a nitrogen blanket while adding the first portion of initiator and vigorously stirring. The reaction was allowed to run for 6 hours. They were purified by precipitating them in an excess of methanol and then filtering them. Purification was completed by dissolving in toluene and precipitating with methanol, followed by filtration and vacuum drying at 40°C .

### 2.2.3. Method mixed the solvent

The pour point test and kinematic viscosity were used to determine the efficacy of xylene, n-hexane, kerosene, triethanolamine (TEA), and their blends with copolymer additives as flow improvers for crude oils. Each improver, copolymer additives (P1,P2), xylene, n-hexane, ker-osene, and triethanolamine (1 percent by volume) were added to the crude oil at room tem-perature (28°C). Each blend contains the following proportions: blend 1 is a mixture 0.5 mL of P1/P2 , 0.5 ml of xylene and 0.5 mL of n-hexane, blend 2 contains 0.5 ml of P1/P2 ,0.5 mL of triethanolamine and 0.5 ml of kerosene), and blend 3 is a mixture of 0.5 ml of P1/P2 ,0.5 ml of xylene, 0.5 mL of n-hexane and 0.5 mL of triethanolamine. Each blend was added to the crude oil at room temperature (28°C).

### 2.3. Characterization of copolymers

In the technology laboratory, the structures of the prepared mono-esters hexadecylacrylate /NAFOL1822B acrylate and copolymers hexadecylacrylate with vinylacetate/NAFOL1822B acrylate with methylmethacrylate, abbreviated as P1 and P2, were confirmed using infrared (IR) spectroscopic analysis. The KBr technique was used to measure the infrared spectra with a model Genesis series (USA) infrared spectra-photometer. Table 3 depicts the characteriza-tion of the prepared copolymers.

Table 3. Molecular weights of the prepared copolymers

| Sample | Designated | Mol. wt. | PDI |
|---|---|---|---|
| Copolymer hexadecyl acrylate (HA)-vinyl acetate (VA) | P1 | 35,841 | 1,164 |
| Copolymer NAFOL 1822B acrylate (NA) - methyl meth-acrylate (MMA) | P2 | 24,588 | 1,3784 |

The molecular weight was determined in the technology laboratory using Gel permeation chromatography (GPC), which included a Waters 515 HPLC pump, a Waters temperature con-trol module, and 2410 refractive index detector from Water Company. And further supplement using viscosity (Brookfield viscometer, USA).

### 2.4.Evaluation method

### 2.4.1. Pour point measurement (ASTM D 97-96)

Effective the effectiveness of xylene, n-hexane, kerosene, triethanolamine (TEA), and their blends with the copolymer P1/P2 as flow improvers for crude oils was determined using the pour point test shown in Table 4, Figure 1. Each improver, xylene, n-hexane, kerosene, and

triethanolamine (1 percent by volume), was added to the crude oil along with the copolymers P1 and P2. . Blend 1 contains (0.5 mL of xylene, 0.5 mL of n-hexane, and 0.5 mL P1/P2), Blend 2 contains (0.5 mL of triethanolamine and 0.5 mL of kerosene), and Blend 3 contains (0.5 mL of xylene, 0.5 mL of n-hexane, 0.5 mL of triethanolamine, and 0.5 mL of P1/P2). At a low temperature, each blend was added to the crude oil.

Table 4. Effect of combined crude oil, copolymer additives and different solvents on pour point of crude oil

| Additives (%) | Pour point ($^oC$) | Pour point depressant ($^oC$) |
|---|---|---|
| Crude oil | 25 | 0 |
| Crude oil+1%P1 | 20 | 5 |
| Crude oil+1% P1+1% xylene | 21 | 4 |
| Crude oil+1% P1+1%n- hexane | 20 | 5 |
| Crude oil+1% P1+1% kerosene | 15 | 10 |
| Crude oil+1% P1+1% TEA | 16 | 9 |
| Crude oil+1% P1+1% xylene +1% n- hexane | 18 | 7 |
| Crude oil+1% P1+1% TEA +1% kerosene | 14 | 11 |
| Crude oil+1%P2 | 15 | 10 |
| Crude oil+1% P2+1% xylene | 19 | 6 |
| Crude oil+1% P2+1%n- hexane | 18 | 7 |
| Crude oil+1% P2+1% kerosene | 12 | 13 |
| Crude oil+1% P2+1% TEA | 13 | 12 |
| Crude oil+1% P2+1% xylene +1% n- hexane | 15 | 10 |
| Crude oil+1% P2+1% TEA +1% kerosene | 10 | 15 |
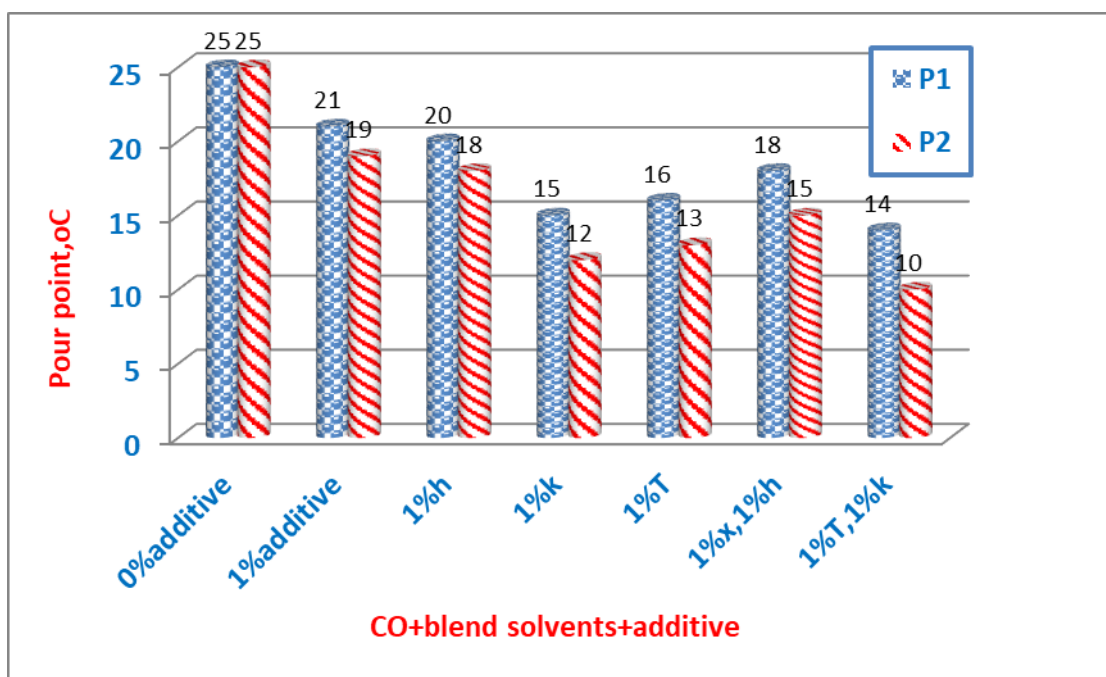


Figure1. Effect of combined crude oil, different additives and different solvents on pour point of crude oil

### 2.4.2. Determination of viscosity

The viscosity was calculated according to ASTM D 445-06. The kinematic viscosity of crude oil P1/P2 and the blend with different solvent was determined at 25$^o$C and 35$^o$C was used to containing the copolymers study the effect of different solvent, type of copolymer, and crude oil on kinematic viscosity. All of the experimental data is presented in Table 5 and Figures 2

and 3. The procedures were repeated for crude oil samples copolymer (P1,P2) without any solvent and treated copolymer additives (P1,P2) with 0.5 mL of xylene,0.5 mL n- hexane,0.5mL kerosene, and 0.5 mL triethanolamine (TEA), and then with the blends blends1 (0.5 mL of xylene and 0.5 mL of n-hexane),  blends 2 (0.5 mL of kerosene and 0.5 mL of triethanolamine) and blends 3( 0.5 mL of xylene, 0.5 mL of n-hexane and 0.5 mL of triethanolamine). Each blend was added to the crude oil with the all the synthesized additive (P1 and P2).

Table 5. Effect of combined crude oil, copolymer additives and different solvents on viscosity of crude oil at different temperatures

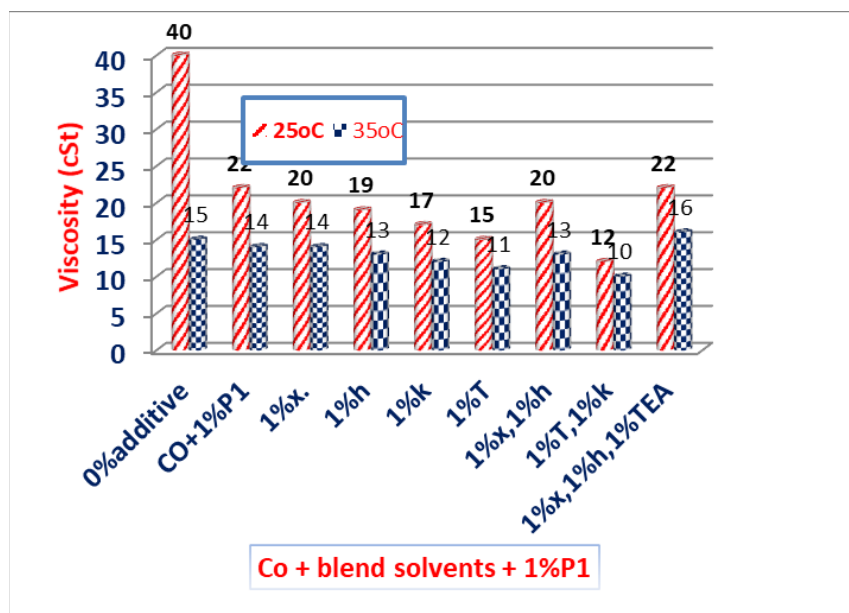| Additives (%) | Viscosity,cSt | |
|---|---|---|
| | At 25°C | At 35°C |
| Crude oil | 40 | 15 |
| Crude oil+1%P1 | 22 | 14 |
| Crude oil+1% P1+1% xylene | 20 | 14 |
| Crude oil+1% P1+1%n- hexane | 19 | 13 |
| Crude oil+1% P1+1% kerosene | 17 | 12 |
| Crude oil+1% P1+1% TEA | 15 | 11 |
| Crude oil+1% P1+1% xylene +1% n- hexane | 20 | 13 |
| Crude oil+1% P1+1% TEA +1% Kerosene | 12 | 10 |
| Crude oil+1% P1+1% xylene +1% n- hexane+1% TEA | 22 | 16 |
| Crude oil+1% P2 | 20 | 14 |
| Crude oil+1% P2+1% xylene | 18 | 13 |
| Crude oil+1% P2+1%n- hexane | 17 | 12 |
| Crude oil+1% P2+1% kerosene | 16 | 12 |
| Crude oil+1% P2+1% TEA | 13 | 10 |
| Crude oil+1% P2+1% xylene +1% n- hexane | 15 | 12 |
| Crude oil+1% P2+1% TEA +1% Kerosene | 10 | 7 |
| Crude oil+1% P1+1% xylene +1% n- hexane+1% TEA | 18 | 12 |

Figure 2. Effect of temperature on viscosity of crude oil with P1 additive and different solvents
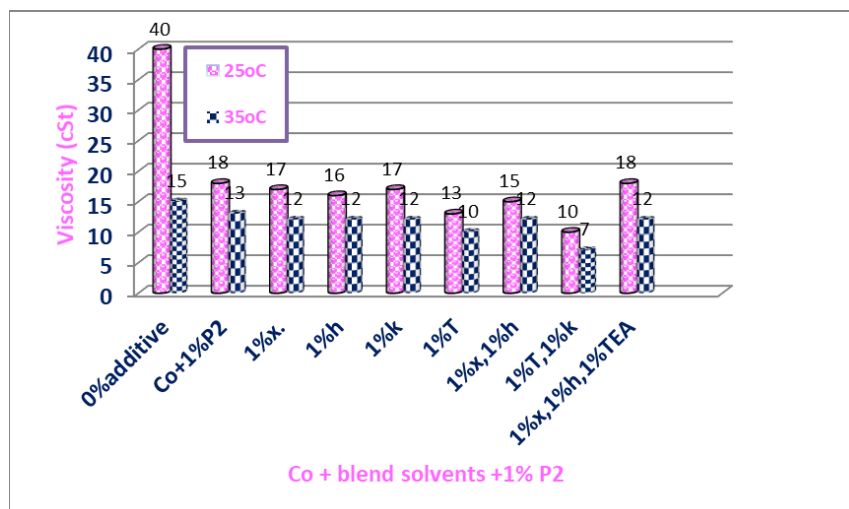
Figure 3. The effect of temperature on viscosity of crude oil blends with P2 additive and different solvents

# 3. Results and discussion

## 3.1. Analytical methods

### 3.1.1. Molecular weight analysis

Table 3 shows the experimental values of average molecular weights (Mw) and polydispersity index data (PDI) of the prepared copolymers (P1 and P2). This result also indicates that polymer P2 is less polydispersed than polymer P1, implying greater uniformity. The values show that as the alkyl chain length of the alcohol used in esterification increases, the Mw values decrease while the PDI value increases.

### 3.1.2. Spectroscopic analysis

**- Ester monomers**

Infrared spectral analysis was used to investigate the chemical structure, which revealed similar patterns for the two esters. The OH stretching band of the mono carboxylic group absorbed a small broad band at 3443 cm$^{-1}$ as a result of the partial esterification reaction in a representative IR pattern for NAFOL 822B acrylate monomer. The stretching vibrations of the CH$_3$ and CH$_2$ groups were strongly absorbed at 2913–2845 cm$^{-1}$, the ester C=O appeared as a strong absorption band at 1707 cm$^{-1}$ , and the presence of ester functionality was further supported by the appearance of a C=O stretching vibration at 1154–1188 cm$^{-1}$. The characteristic C=C stretching band was detected at 1636 cm$^{-1}$, while the CH in plane deformation vibrations of acrylate ester monomers were detected at 1323 cm$^{-1}$. A strong absorption of the bending 713 cm$^{-1}$.

**-Copolymers**

In IR spectra was illustrated absence of peaks above 3000 cm$^{-1}$ indicates absence of C=C unsaturation which supports complete polymerization in all the cases. The characteristic C=C peak of alkyl acrylate vanished completely. Therefore, the P1 and P2 copolymers were synthesized successfully. Lack of peaks over 3000 cm$^{-1}$ in IR spectra demonstrates the absence of C=C unsaturation, which confirms full polymerization in all circumstances. As a result, the P1 and P2 copolymers were successfully produced.

## 3.2. Evaluation tests

### 3.2.1. Effect of copolymer additives and solvents on the pour point

The depression of the crude oil pour points is shown in Table 4 and Figure 1. P1 with crude oil lowered the pour point to 20$^o$C, while P1 with Xylene lowered the pour point of crude oil to 21$^o$C from 25$^o$C. P1 with n-hexane decreased the pour point to 20$^o$C, while P1 with kerosene

decreased it to 15°C, P1 with triethanolamine (TEA) reduced the pour point to 16°C, P1 with xylene and n-hexane blends reduced it to 18 °C and P1, triethanolamine, and kerosene blends reduced it to 14°C. While crude oil with P2 reduced the pour point to 15 °C, the additives may co-crystallize with the paraffin wax and modify their crystal structure. It was also discovered that there is a direct correlation between chain length and efficiency, with efficiency increasing as chain length increases, so the prepared additives were ranked as P1 <P2 [15].

The prepared additives reduced the pour-point temperatures from 25°C to 15°C at the maximum pour-point reduction of 1000 ppm of P2. The pour point reduction [16] was calculated using the following equation: pour point reduction $(\Delta PP) = PP_{pure} - PP_{add}$ ; where $PP_{pure}$ is the pour point of the crude oil without any additives (blank) and $PP_{add}$ is the pour point of the treated crude oil .

P2 with xylene reduced the pour point of the crude oil to 19°C, while P2 with n-hexane reduced it to 18°C. P2 with kerosene lowered the temperature to 12°C . P2 with triethanolamine (TEA) lowered the temperature of the oil to 13°C from 25°C. P2, xylene, and n-hexane blends reduced it to 15°C, while P2, triethanolamine, and kerosene blends reduced it to 10°C. The decrease in pour point is primarily caused by wax crystal modification. Pour point depressants molecules are adsorbed on different crystal faces P2 than P1, reducing the interlocking forces in P1 additive between two nuclei of wax molecules and deforming regular crystal growth [17].

When compared to copolymers,P2 additive has a higher efficiency as PPD. It could be because P2 has a higher polarity than P1 additive. The highly polar polymers are highly adsorbed by the wax crystals present in crude oil, improving their efficiency as polymer PPD improvers [18]. When present in crude oil, the pour point depressant changes the wax crystal shapes from thin extensively interlocking plates to more compact crystals by co-crystallizing with the wax [19]. The triethanolamine (TEA) actively reduced the samples' pour points and wax deposition potentials after doping. The oxygen-containing group in triethanolamine inhibits wax growth and poisons it via an adsorptive surface poisoning mechanism [20]. The waxes then formed in the crude oil as small particles and were unable to form the net-like structure required for solidification and deposition.

### 3.2.2. Effect of copolymer additives and solvents on the crude oil viscosity

Table 5, Figures 2 and 3 show how effective the added flow improvers are on the viscosity of the crude oil as well as their blends. At room temperature (25°C), the kinematic viscosity of the crude oil reduced from 40 cSt to 20 cSt and 18cSt for the crude oil containing P1 and xylene, P2 and xylene respectively, reduced to 19 cSt and17 cSt for crude oil containing P1 and n-hexane, P2 and n-hexane respectively, reduced to 17cSt and 16 cSt for crude oil con-tainingP1 with kerosene, P2 with kerosene respectively  and reduce to 15 cSt and 13 cSt for crude oil containing P1 and triethanolamine, P2 and triethanolamine, respectively. On adding copolymer additives with solvents blends, blend 1 (mixture of 0.5mL P1,0.5mL xylene and 0.5mL n-hexane/   mixture of 0.5 mL P2, 0.5mL xylene and 0.5 mL n-hexane), blend 2 (mixture of 0.5mL P1, 0.5mL triethanolamine and 0.5mL kerosene/mixture of 0.5mL P2,0.5mL triethanolamine and 0.5mL kerosene) and blend 3 (mixture of 0.5mL P1,0.5mL xylene,0.5mL n-hexane and 0.5mL triethanolamine/mixture of 0.5mL P2, 0.5mL xylene,0.5 mL n-hexane and 0.5mL triethanolamine) to the pure crude oil, the viscosity of the crude oil at 25°C re-duced. Blend 1 reduced the viscosity from 40 cSt to 20 cSt and 15 cSt, blend 2 reduced it to 12 cSt and 10 cSt blend 3 increased it from 40 cSt to 22cSt and18 cSt respectively.

As temperature increased, the viscosity of the crude oil reduced  Table 5, Figures 2 and 3. This is in line with the reports by [21], that crude oil response differently with the same additive at different temperature due to the changes in rheological properties of the crude oil. The Table 5 shows that the additive plays an important role in affecting the viscosity of the crude oil. The viscosity of the crude oil can be improved by adding requisite amount of flow improver, the appropriate type P1 or P2 and volume of the additive has to be added for effectiveness. From the results in Table 5 triethanolamine (TEA) is a very good wax deposition inhibitor with P2 additive than P1 additive. The reduction of viscosity on the addition P2 additive of these solvents is due to the dissolution of paraffin wax, which shows the effectiveness of these

additives, also effect of chain length where efficiency increase as chain length increase so P2 showed high viscosity reduction. Reduction of viscosity depends on the temperature type of additive and solvent**.**

## 3.3. Mechanism analysis

The low-temperature flow ability of crude oil is improved to better understand the P2 additive, and the possible working mechanism is depicted in Figure 4. Because n-alkanes have a high melting point in untreated crude, the wax crystals gradually precipitate, agglomerate, and interact with each other to form a three-dimensional network structure, losing fluidity at low temperatures. The long-chain alkyl of P2 additive inserts and co-crystallizes with n-alkanes in crude oil, providing an abundance of crystallization sites, thereby delaying the starting precipitation temperature, modifying the growth orientation of wax crystals, and preventing the formation of large crystals in P2 additive treated crude oil. As a result, the crystallization behavior and morphology of the crystal are modified, resulting in a better cold flow property illustrated in Figure 4.
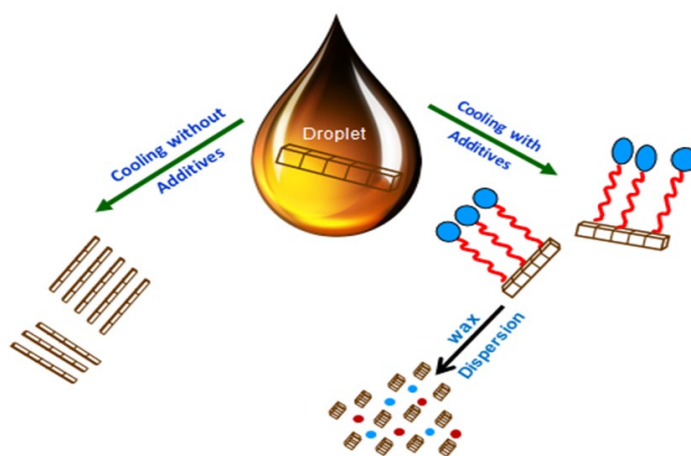


Figure 4. Illustration for wax crystallization process of treated with P2 additive

## 4. Conclusion

For crude oil, the copolymers can act as effective pour point depressants and flow improvers. Because of its longer alkyl chain, copolymer NAFOL 1822B-methyl methacrylate (P2) outperformed copolymer hexadecyl - vinyl acetate as a pour point depressant and viscosity reducer. The effects of different solvents xylene, n-hexane, kerosene, and triethanolamine on the pour point and viscosity of crude oil were investigated. All of the additives tested in this study were effective at lowering crude oil is pour point and viscosity of the crude oil. At different temperatures, triethanolamine and kerosene with P2 additive were more effective than triethanolamine and kerosene with P1 additive (25$^{o}$C, 35$^{o}$C).

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