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AN OVERVIEW OF DIFFERENT POUR POINT DEPPRESANT SYNTHESIZED AND THEIR BEHAVIOUR ON DIFFERENT CRUDE OILS

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Abstract: Wax deposition in crude oil during transportation and production causes serious issues such as flow reduction and blockage in pipeline adversely impact pipeline performance. Different mechanical and chemical methods have been used to overcome this issue. Out of all these methods, pour point depressant has been found to be an effective remedy to wax deposition. Pour Point Depressant has been synthesized through different methods and used widely for minimizing the deposition of wax in the crude oils lines. Besides these, the PPD are also used to reduce the freezing point of crude oil thereby reducing the flow resistance during transportation of oil. It is used as the flow improver which increases the liquidity of the crude oil. The various factors affecting the performance of the crude oil in presence of synthesized PPDs have been studied among which many of the PPD synthesized are Oleic acid based or maleic anhydride based polymeric additives through esterification and Friedel Craft Alkylation. Different types of PPDs having high molecular weight copolymers show better results than other low molecular weight polymers.PPD synthesized were examined on the the crude oils of the Limbodra, Nada, Langhnaj, Akholjuni of Gujarat region and the results were investigated throught SARA Analysis, Fourier Transform Infrared Spectroscopy(FTIR), Gel Permeation Chromatography(GPC).

Index Terms – Pour Point depressant, crude oil, flow improvers.

I. INTRODUCTION

Petroleum and its product are the most important substances for today's modern society. It is used as raw material or as a fuel for the production of energy and in industries and transportation. Petroleum, also called as crude oil is a naturally occurring world's treasures which is toxic, flammable liquid consisting of complex mixtures of the hydrocarbons of various molecular weights. They are formed beneath the earth surface due to the debris of plants and animals which had settled down at the bottom of the ancient oceans. These remains or sediments accumulated and buried by layers of mud and sand and gradually, over the millions of years due to the pressure and heat get converted into the rocks. The living organism (once living) between these sediments get converted into a waxy substance which further converts into oil and natural gas or coal and other types of fossil fuels because of chemical and biological transformation.

Hence, Petroleum is not a uniform homogeneous material but its composition may vary with the location and age of the oil field, depth and its depth in the well. It is a mixture of hydrocarbons with some organic compounds containing other elements. Thus, the Crude oil varies greatly in appearance depending on its composition for example it may be black or dark brown or yellow to black thick sticky liquid. This Crude oil contains hydrocarbon of various molecular weights. The most commonly found molecules are paraffin or wax, naphthalene's, aromatics and chemicals like asphaltenes. Each of the Petroleum Variety obtained from different wells or locations has a unique mix of molecules, which define the physical and chemical properties like color, viscosity, fluidity, cloud point and pour point, etc

Table 1.1: Elementary composition of crude oil

Sr. No.	Element	%by wt.
1	Carbon	83.0-87.0%
2	Hydrogen	10.0-14.0 %
3	Nitrogen	0.1-2.0%
4	Oxygen	0.05-1.5%
5	Sulphur	0.05-6.0%
6	Metals	<1000 ppm

The crude and heavy fuel oils are complex mixtures of various hydrocarbons ranging from paraffins, aromatics, naphthene's, and resins to asphaltenes. These long chained paraffins tend to crystallize when cooled down, which ultimately lead to the severe sedimentation. Since wax normally makes up only a small percentage of the hydrocarbons in oil, the wax structures contain large amounts of trapped liquid, resulting in a matrix that is quite fragile. Virtually all the paraffinic mineral oil base stocks contain small amounts of waxy materials. As the temperature of the oil is decreased, some of the waxy components come out of solution as tiny crystals (needles), and the solution begins to appear hazy to the naked eye. The temperature at which this occurs is called the cloud point.

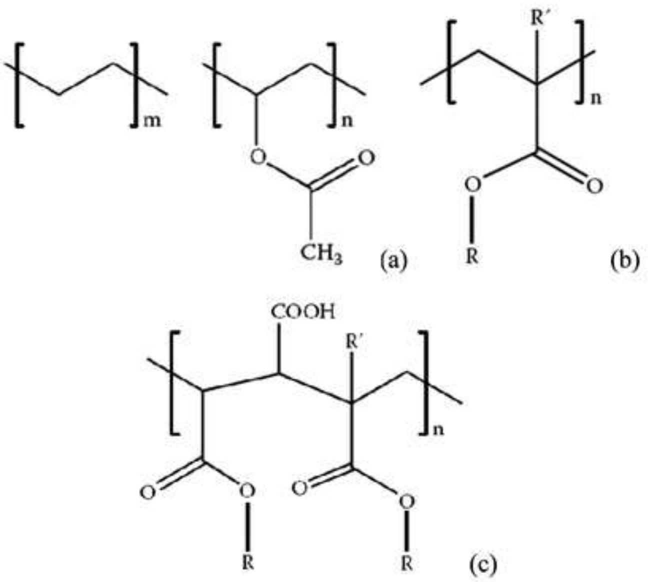
As additional wax precipitates, the crystals grow into plates and, finally, if the temperature is decreased further, the plates will grow to form a three-dimensional network which completely reduces or stops the mobility of the oil (gelation). The lowest temperature at which the oil is fluid is called the pour point.Thus, in order to prevent the sedimentation or the blockage of pipelines, Pour point deppresants are added to keep the crude or heavy fuel oil moving.

In most high wax content crudes, high rates of cooling will generally favour the formation of a large number of small crystals. Rapid cooling causes the formation of many nuclei that result in smaller crystals.
The crystallization of paraffins will occur because of the variation in the cloud-point/melting point of the various paraffinic components. In general, the higher molecular weight paraffins will tend to come out of solution before the lower molecular weight paraffins. On rapid cooling, the high and low molecular weight paraffins can come out of solution simultaneously.

II. Pour Point Depressants

The pour point of a crude oilis the lowest temperature at which the oil will pour or flow when it is cooled, without stirring, under standard cooling conditions. It represents the lowest temperature at which oil is capable of flowing under gravity. If the temperature is less than the pour point of a petroleum product, then the crude oil cannot be stored or transferred through a pipeline. The pour point of crude oils is related to the paraffin content of the oil: the higher is the paraffin content, the higher is the pour point of the crude oil. . To reduce wax deposition or to lower pour point of crude oil, a chemical compound having polymeric structure is added which is known as pour point depressant or flow improver.
Prior to 1930s, there were very less options to deal with the cold flow problems, but a reasonable alternative solution was to increase the solvency power of the fluid portion of the oil by addition of some solvent. But all these methods were also creating flow problems at high temperature as it decreases the viscosity of the oil. The other alternative sources were also experimented by using the naturally occurring materials which were sometime reasonabley effective. Because these materials depress the pour point of the crude oil, they were termed as Pour Point Depressants. Based on this many synthetic materials which can behave similar to these naturally occurring PPD were synthesized and experimented on the crude oil.

In 1931, alkylated naphthalenes containing the linear waxy paraffinic structures were introduced and in 1937 the first polymeric pour point depressants, polyalkylmethacrylates (PAMAs) based on waxy alkyl groups. Over the years a wide variety of synthetic materials have been synthesized as pour point depressants. The most commercial products used as PPDs were moderate to high molecular weight polymers such as polymethacrylates, polyacrylates, acrylate-sttyrene copolymers, esterified loefin or styrene maleic anhydride copolymers, alkylated polystyrene and vinyl acetate fumarate copolymers. In order to mitigate and prevent the wax deposition in the pipeline Different polymeric inhibitors have been utilised in the oil and gas field.



Molecular structure of EVA copolymers (a), poly(meth) acrylate ester (b), and (c) monoester of maleic anhydride-(meth) acrylate ester copolymer. R = long alkyl chain, R′ = H or CH_3.

While measuring the pour point, it is 3^0C (5^0F) above the temperature at which the oil shows no movement when a lab sample container is held horizontally for 5 seconds. The pour point is an indication of oil's cold-temperature properties.

III. Chemical structure of PPDs

Most polymeric additives contain a nonpolar moiety as well as a polar moiety, with the exception of crystalline-amorphous copolymers. Via nucleation, adsorption, co-crystallization and solubilization interactions, non-polar moieties of polymeric additives which usually contain long alkyl chains, alter the morphology and interface of precipitated wax crystals, inhibiting wax deposition, improving flow, and inducing gel formation. On the other hand, the crystal growth was interested by polar moieties such as esters, vinyl acetates, anhydrides, etc. Most polymeric additives fall into the categories of crystalline-amorphous copolymers, ethylene-vinyl acetate copolymers, comb polymers and nanohybrids.

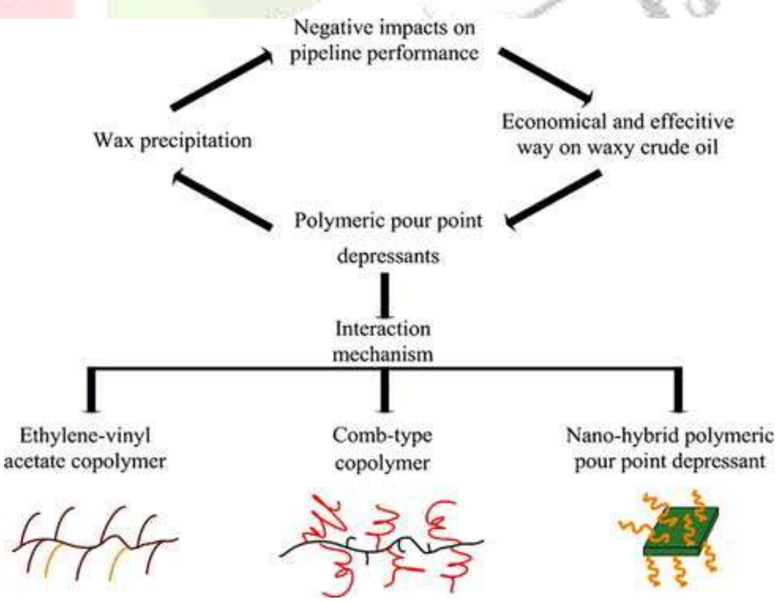


Fig – 3.1 Effect of Pour Point Depressants

Over many years, a wide variety of synthetic materials has been introduced commercially as pour point depressants. Chlorinated wax is the most notable example of small molecule chemistry, but most commercial products are moderate to high molecular weight polymers, such as polymethacrylates, polyacrylates, acrylate-styrene copolymers, esterified olefin- or styrene maleic anhydride copolymers, alkylated polystyrene, and vinyl acetate-fumarate copolymers. Alkyl aromatics and aliphatic polymers are two types of pour point depressants that are commercially available. Most commercially available pour point depressants are organic polymers, but nonpolymeric substances such as phenyl tristearyloxysilane and pentaerythritol tetra stearate may also be effective

IV. Mode of Action:

Crystallization of paraffin waxes in crude oil takes place in three stages: nucleation, growth and agglomeration. Nucleation occurs below WAT where wax molecules attach and to form a stable cluster.

The crystal growth then takes place in which the clusters attach in a plate-like or lamellar structure. The Pour Point Depressant acts as a flow improver or wax crystal modifier by modifying the size and the shape of the wax crystals. Hence, the wax crystals are not inhibited to grow but modified not to precipitate. PPDs Consist of Polar component with large polymer molecules which inhibit the formation of crystal growth. Thus the performance of the PPD does not depend on the concentration and distribution of paraffin but also influenced by the presence of asphaltenes and other component of the crude oil.

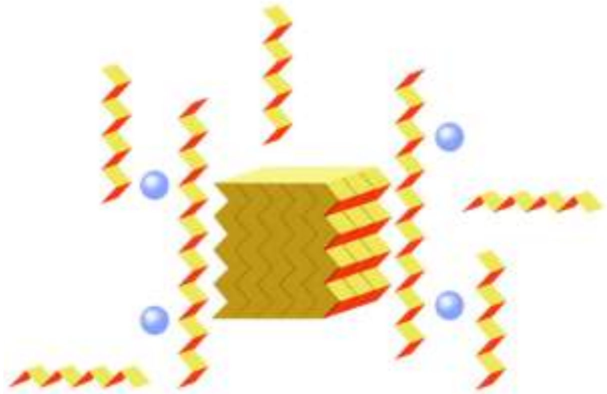


Fig – 4.1 Crystal Modification of a wax by Pour Point Depressants

At temperatures below the WAT, pour point depressants co-crystallize with wax molecules or adsorb on growing surfaces of precipitated wax crystals which inhibits the growth and ultimately inhibiting wax deposition and improving flowability. Pour Point Depressants (PPD) are the polymers which prevent the formation of wax fractions in the base oil, thereby reducing the forming of large crystal networks which inhibit lubricant flow at cold temperatures. It modifies the interface between the crystallized wax and the oil.

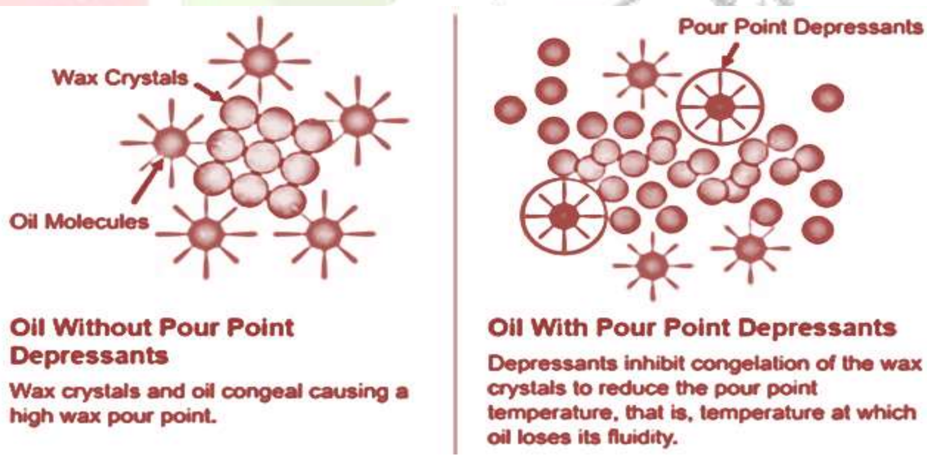


Fig 4.2 Effect of PPD with and without oil

Reduction in the pour point of oil depends on the both the composition and properties of the oil as well as the type of pour point depressant used. The other factors which affect the pour point of the oil are the relative molecular weight of the crude oil, its chemical composition and the concentration of the oil. If the concentration of the pour point depressant is too high the viscosity of the oil will be affected at higher temperatures.

The PPDs are only effective on refined oils. Non refined oils contain polyaromatic hydrocarbons and resins which act as antagonists against these laboratory synthesized PPDs. Generally, the PPDs are effective on the thinner oils.

The presence of asphaltenes significantly impacts wax crystal morphology and interfaces, thus influencing the mechanism of polymeric additives. Factors influencing polymeric efficacy include molecular structure, fluid composition, and pipeline transport conditions.

Most of the wax is removed during the oil refining due to which the pour point depressants are added in order to allow mineral oils to efficiently function at low temperatures, which also keeps the viscosity same at higher temperature. Many good polymeric additives can lower the pour point as much as 40^0 C, which alter the wax crystal growth and keep the bulk oil in a liquid state. The Difference in the structure of the viscosity improver and pour point depressant is in the backbone chain and the branches of the PPD.

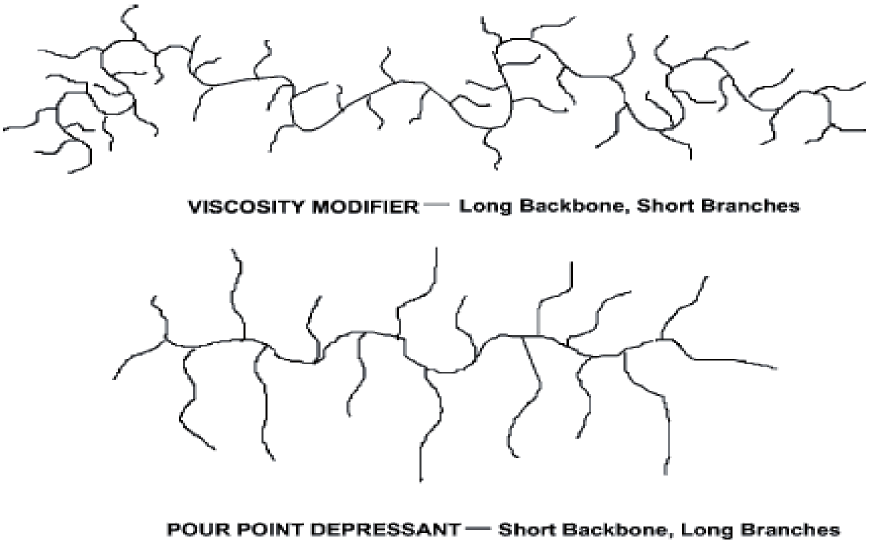
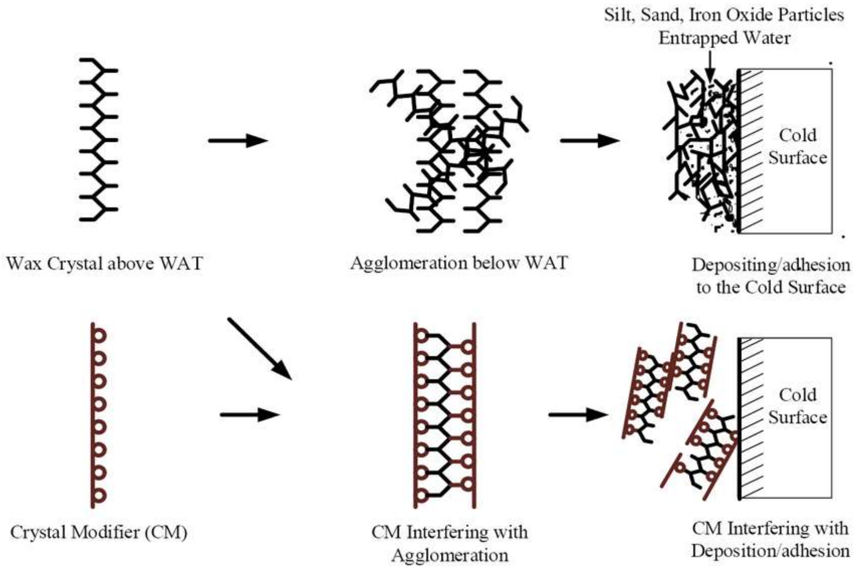


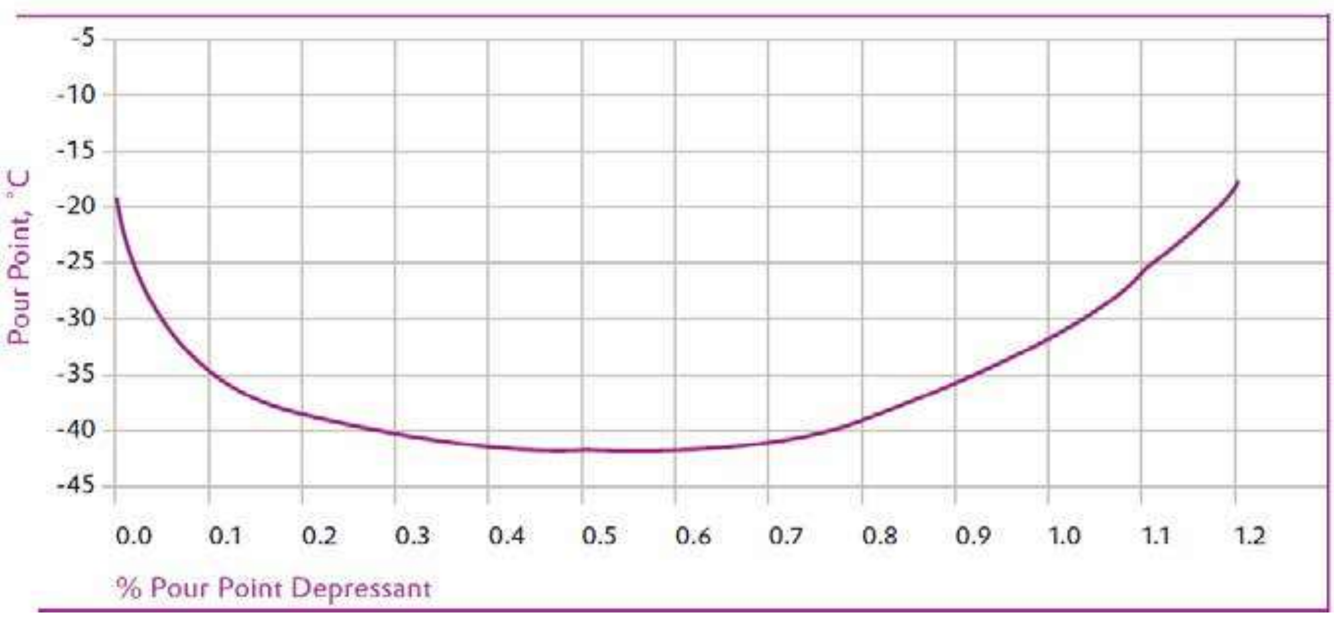
Fig – 4.3 Difference between the viscosity modifies and PPD

The PPD works on the wax crystal growth by creating the distance between the agglomeration of wax (WAT) which in turn increases the gap between the crystals and thereby reduces the formation of Crystals.



The PPD works as per the structure formed and depend on the polymerization of the monomer used to synthesize PPD. The molecular structure of the polymers gives the flexibility to create the distances between the crystals. Since many years' different types of PPDs have been synthesized which have different structure and their mechanism of interaction with the wax crystal growth and its hindrance on the growth of crystals have been studied.

The PPD activity does not wholly depend on the polymer molecular weight and the degree of polymerization.



V. PPD selection

The selection of PPD and use is highly important for ensuring high performance and prolong lifecycle of oil formulations by preventing equipment damages at low temperatures. The correct dosage selection of the PPD for the crude oil provide enhancement of low temperature performance even at low concentration. But if the concentration of PPD is raised than an additional improvement is also seen up to minimum extent. These flow improvers themselves behaves as waxy material hence while adding the calculated dosage of PPD, the wax accumulation should under control otherwise the reverse performance takes place

The function of wax is time and thermal history dependent. The wax solution if formed will behave like a supercooled liquid in which wax crystallization takes place over a period of time thereby converting the wax into gel form.

The Factors which affects the Pour point Depressant and its rate of Treatment- are

1. Base Stock.
2. Other waxy component present in the oil
3. The amount of Detergents and the Modifiers added
4. Viscosity Modifiers
5. Methods used for testing the different specifications
6. Performance at low temperature and its reverse effect
7. Ageing of oil and its pumpability requirement
8. Role of Other Additive components having hydrocarbon structure
9. Agglomeration of Polar molecules in Aged oil

A pour point depressant cannot be selected just on the basis of the single test but it is test as per the different temperature requirement of the crude oil. The PPD"s not only has to be tested on the fresh oil but it should also be properly tested on the aged oil because the aged oil is already oxidised and contains polar molecules which can form structure at cold temperatures and at this condition also the PPD"s must be able to control the agglomeration of these polar molecules.

VI. Experimental Work Statistic for PPD

Some of the experimental work on the synthesis of the Pour Point Depressant had been discussed in the detail with the graph and the overview of the different monomer used in the synthesis of PPD with its reaction condition and the result is summarized in the table

Sinsakulroj et. al (2012) worked on Synthesis of pour point depressant (PPD) from sunflower oil. PPD used to improve cold flow property of palm biodiesel by four pour point depressants was synthesized by transesterification of sunflower oil with methanol, ethanol, 2-propanol and 2-butanol. On working experiment, they added Palm biodiesel with PPDs at various concentrations: 10,000, 100,000 and 300,000 ppm after that the mixture solutions were measured CP (cloud point) and PP (pour point) according to the ASTM D-2500 for CP and ASTM D-97 for PP. It was observed that, at 300000 ppm, cloud point of palm biodiesel was decreased from 21°C down to 14.2°C, and the pour point was decreased from 19°C down to 13.6°C, when using 2-butanoate ester.

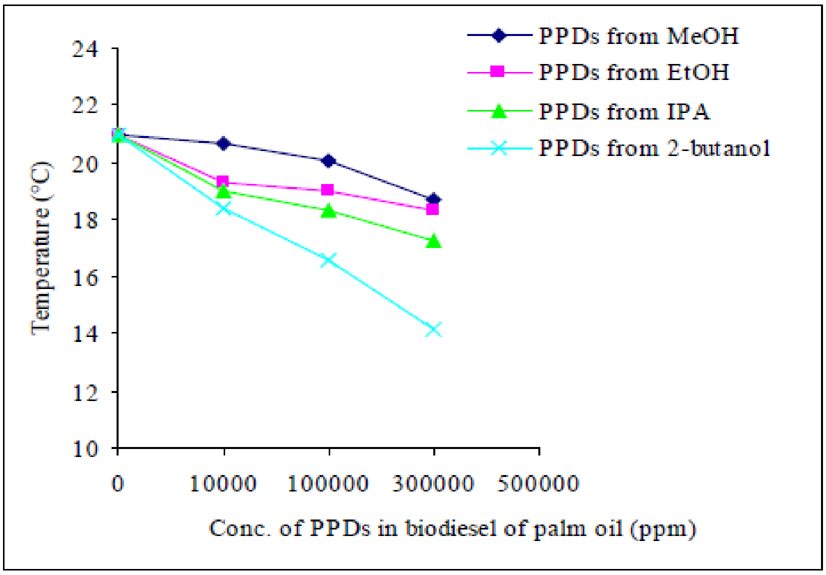


Figure 6.1: Conc. of PPDs in biodiesel of palm oil (ppm) vs. Temperature

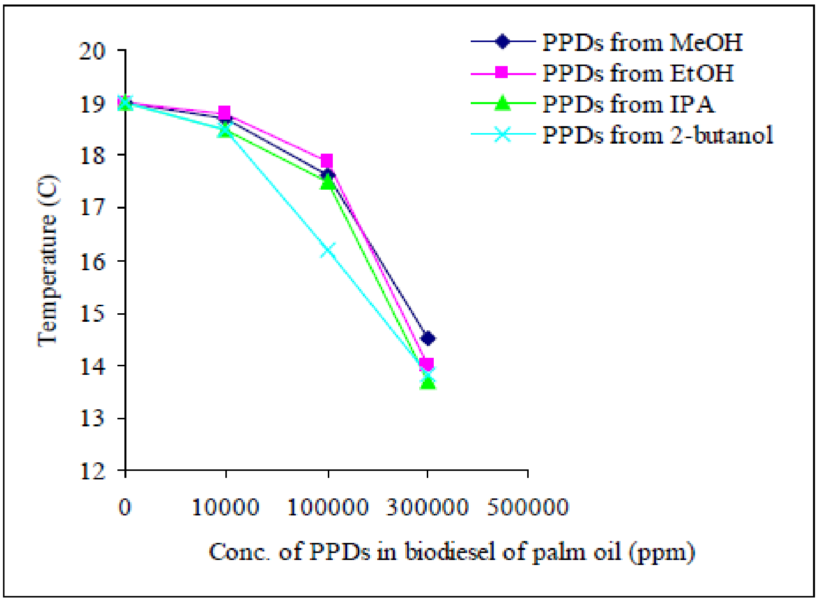


Figure 6.2: Conc. of PPDs in biodiesel of palm oil (ppm) vs. Temperature

Ghuiba et. al. (2014) conducted studies on preparation and evaluation of some surfactant as pour point depressants in oil field. Where three surfactants additives were synthesized by esterfication of polyethylene glycol with adibic, phythalic and citric acid then esterfication of polyethyleneglycol adipate acid (A), phythalate acid (P) and citrate (C) with triethanolamine by them. The three prepared additives were tested as pour point depressants through pour point determination according to ASTM D-97 procedure at concentration 300, 700, 1000, 1500, 2000 ppm and results shows in Figure. It was observed that all additives achieved moderate pour point depression at concentration 1000ppm and the activity of these additive as pour point depressant has the following the order A>P>C.

They evaluated the prepared surfactants as surfactants by surface tension and critical micelle concentration. It has been discovered that an additive is more efficient as pour point Δpp 2000ppm = 12°C than phythalate acid (P) and citrate (C), Δpp 2000ppm = 9°C and 6°C. Mixes between the best of additive adipate acid (A) with commercial flow improver CFI and Natural wax dispersant NWD were done and evaluated as pour point depressants (500:1000:500), Δpp 2000ppm = -21°C[3].

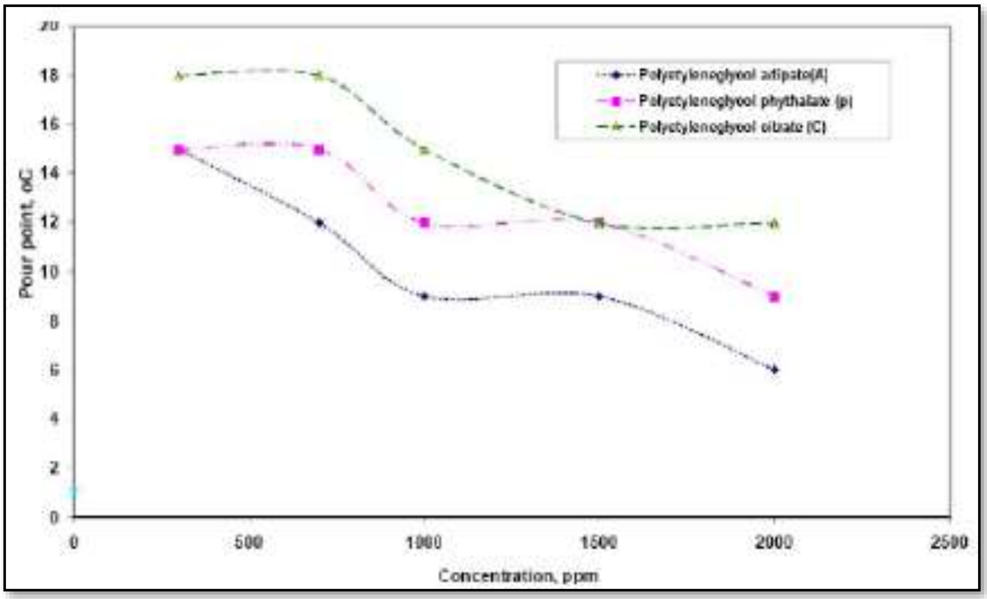


Figure 6.3: Conc. of three PPDs (ppm) vs. Temperature

Tung et. al. (2001) had done investigations of paraffin deposition process and the noteworthy changes in the crystal structure of waxes brought about the improvement of a few copolymer paraffin gem modifiers. They observed that mixtures with surfactants have caused strong viscosity reduction in Various Vietnam high paraffin crudes. Likewise it was discovered that the adjustments in structure and size of paraffin crystals due to co crystallization between effective pour point-viscosity depressants and crude oil waxes, were examined and the outcomes were recorded by method of scanning electronic microscopy (SEM).

The mechanisms of pour point and viscosity reductions were inspected using Infrared and Raman spectroscopes by them. It was found that Ethylene vinyl acetate copolymers can lessen the pour point and viscosity of Dragon and White Tiger crude oils, Copolymer A seems to be more effective than copolymer B. "A"'s structure is similar to the paraffin crystal structure of Vietnam crude oil. Subsequently, copolymer A easily incorporates and co crystallizes with the paraffin crystals. While surfactants AN and NI are effective in deposit dispersion and in the water wetting of paraffin particles, shielding them from flocculating and depositing on the walls of tubing, flow lines and pipelines.

They have used Scanning Electronic Microscopy is an effective analytical tool in studying the shape and size of paraffin crystal morphology. Chemical additives which produce more clusters or more crystal with smaller size and rounder shapes are viable synthetics in crude oil pour point and viscosity reduction. Likewise the IR technique is useful for providing the basic data in studying the mechanism of viscosity reduction. The Raman technique is helpful for investigating the change of the crystalline state of paraffin during the chemical additive treatment process[6].

Wilson et. al. (2010) worked on Novel Polymer Modifications Lead to Next-Generation Pour-Point Depressants. PPDs with blended (linear and branched) alkyl esters were synthesized and applied to model oils. They observed that by introducing a 2-alkyl branching into the polymer structure, the solubility of the polymer in xylene or high-flash-point aromatic solvents could be almost doubled and the final solvent based PPDs were still liquid at ambient temperature. It was found that their viscosities ran somewhere in the range of 1.3 and 2.7 Pa.s at an active content of 70%. Noteworthy decrease of pour point and gel strength, accompanied by low-temperature viscosity, was seen in two high-paraffinic model oils. These types of highly concentrated solvent based PPDs enable the oilfield and refinery industry to overcome the challenge of gelling or blocking in the production, transportation, and storage of high paraffinic crude oils.

Figure 6.4: Effect of PPDs with blended alkyl esters to model oils

It was found that BEM series of PPDs have a good pour point reducing/viscosity-reducing effect for crude oils transported by Luning, Zhongluo, Pulin, Weijing, Donghuang and Donglin etc. long distance pipelines in China, at the dosage of 50 mg/kg, pour points of above-mentioned crude oils can be reduced by 19°C, 19.5°C, 18°C, 13.5°C, 13°C and 20°C respectively, and viscosities of above-mentioned crude oils can be reduced by 84.6%, 94.9%, 97.5%, 93.0%, 15.7% and 23.0% respectively. After BEM series of PPDs were used in above-mentioned pipelines, a tremendous economic benefit was obtained (more than 40 thousand tons fuel was saved per year, and more than 28 million yuan's RMB was saved per year after deducting cost of PPD), safety and flexibility of above-mentioned pipelines operation were remarkably enhanced too. Above waxing out temperature, pump shear and pipe shear have no influence on the rheological behaviour of the crude oil.

It was observed that main reason for above-mentioned phenomenon is that above waxing out peak temperature there are a very small number of wax crystals in the crude oil, thus pump shear and pipe shear couldn't almost alter structure of wax crystal. According to them during waxing out peak temperature, pump shear can deteriorate the rheological behaviour of the crude oil, but pipe shear can improve the rheological behaviour of the crude oil. The main reason for above-mentioned phenomenon is that during waxing out peak temperature there are a great number of wax crystals in the crude oil, structure of wax crystal was destroyed by violent pump shear and became the smaller wax crystals with new active centres, then smaller wax crystals interlocked by the active centres on wax crystals, and formed network structure with the larger strength, but weak pipe shear improved probably interaction between PPD molecules and wax crystals[4].

Hao et. al. (2019) reviewed the Mechanism and Role of Wax Inhibitors in the Wax Deposition and Precipitation. As indicated by their review it is imperative to grasp how WIs and PPDs, even in small dosage, serve as an effective preventive measure in the transportation pipelines, considering the critical flow assurance problems that are caused by the paraffin waxes. They provide an overview on the major mechanisms of these WIs and PPDs in delaying the formation of wax crystals as well as their ongoing applications. It was found that the mechanisms of WIs are largely similar, which are governed by various factors, such as the composition of crude oil, the structure of WIs, the length of alkyl side chains, the average molecular weight, and the carbon number of alkane chain in the crude oil.

According to their review the parameters heavily depend on one another to achieve optimum co-crystallization and surface adsorption for these wax crystals. In this regard, the most common mechanism is the co-crystallization, which highly emphasizes on the compatibility of the length of alkyl side chain to increase the interaction and solubility of WI in the structure of paraffin wax. Despite the documented effectiveness of these polymeric WIs across different oilfields at the global scale, these polymeric WIs have several significant limitations, such as their high molecular weight (that impedes the decomposition in the refinery), the deposition of harder wax in the production pipelines, and the uneconomical cost of polymeric WIs. Meanwhile, it is imperative to critically explore the selectivity of these polymers WIs that is highly dependent on the nature of crude oil. Considering that, it is recommended to seek for cleaner and universal inhibitor for waxy crude oils with small molecules.

It was noticed that the use of surfactants should be critically explored in the laboratory research or field application settings. These surfactants possess similar co-crystallization ability and the emulsifying ability with the presence of water to form O/W emulsion, which ease the pigging operation. Besides that, their wax dispersion ability places these surfactants as a desirable WI compared to the conventional polymeric WI. According to them certain non-toxic bio-surfactants have lower cost of extraction with the ability to recover in the refining process, which leads to a greener and more economical flow assurance industry. Apart from that, it is recommended for future research to further assess the development of surfactant-polymer hybrid despite the proven results of enhanced performance in prior studies. Moreover, the surfactant nano-hybrids were found to be inadequately explored, which should be comprehensively addressed in the future research, as well[e].

Soni et. al. (2004) performed synthesis of chemical additives and their effect on Akholjuni crude oil (Gujarat, India). They synthesized four chemical additives HS-1 to HS-4 for improving the flow properties of Akholjuni crude oil and characterized their effect on pour point and rheological properties of the crude oil. It was found that HS-1 to HS-4 satisfies the most of requirements to act as a pour point depressant and flow improvers. Length of pendant side chain of additives HS-1 to HS-4 increases but their numbers remains same. They concluded that the numbers of pendant alkyl chains do not affect very much in changing the pour point and rheological properties of the Akholjuni crude oil but optimum length of pendant chains definitely affects in reducing such properties. From all four additives, HS-3 matches all such requirements and performs best at the conditions of experiment. Also it was notice that resins and asphaltenes present in the crude oil plays a significant role on flow properties of the crude oil. They finally concluded that property designed copolymers can serve as better PPD[1].

Table 6.1 Summary of the PPD Synthesized

Sr. NO .	Oil Type	PPD	Parameters	Monomer	Solvent	Result
1	Langhnaj, North Gujarat Crude OIl	MPO8, MPO10 MPO12	Refluxing temperature for 8-10 hours	n-alkyl Oleate and fatty alcohol	Toluene	MPO8- 3^oC Drop MPO10, MMPO12 – 6^OC
2.	Akholjuni, Gujarat Crude Oil	HS-1 HS-2 HS-3 HS-4	60-70^O C, 7-8 Hours	Esters of N – alkyl Alcohol, Maleic Anhydride, Unsaturatted acid	Xylene	HS-1 & HS-2 – 6^oC Drop; HS-3 & HS-4 - 6^oC increase.
3.	Gandhar Crude oil	DO-16, DO-18 DM-16, DM-18	65^o C – 70^o C 16-18 hour N_2 atm	Esters of N – alkyl Alcohol with oleic acid, methacrylic acid, maleic anhydride	Dry Benzene and Xylene	DO-16 & DO-18 – 9^oC drop; DM-16 & DM-18 - 6^oC drop
4.	Synthetic Crude oil	Emulsified acrylic polymer PD90	50^O C 500 RPM 1hour	Emulsified pD90 and Solvesso 150	Ethylene Glycol Distilled water and diethanolamine	-20^o C
5.	Kathana Crude Oil	Comb Shaped Polymer 22 CMR 22 MMR	145^0 C – 150^o C 15 h	n- alkyl esters, methyl ricinolate	Xylene	3^o C 6^o C

		22 UMR 22 OMR				9° C 12° C
6.	Model Oils	Styrene Maleic Anhydride Copolymer 1. PSMA-1822 2. PSMA-1822/I32-70/30 3. PSMA 1822/I32-30/70	95° C to 98° C 6-8 hours	Styrene and Maleic anhydride	Benzoyl Peroxide and Xylene	30° C 15° C -3°- C
7.	Khalda Petroleum Co. Product 1. Sumpetco Base Crude Oil 2. Salam Base Crude Oil	PEAA-DcA Copolymer	140° C N_2 gas	Ethylene Acrylic acid ,Vinyl acetate Polymer, 1-Docosanol	Xylene	3° C 6° C 9° C 12° C 15° C (Depending upon dose)
8.	Mevad Oil field	Natural Surfactant	48 hours	MadhucaLongifolia fruit (Raffinate)	Ethyl acetate	7°- C
9.	Egytian Waxy Crude oil	PPD	-	Octadecene and Styrene	Alkylation, Esterification , Amidation	6° C

VII. Conclusion

The Synthesized PPD were tested and experimented with the crude oils of different region and sector and had reduced the pour point of the crude oil by 3-21° C. Some natural Surfactant had also been used as flow improver which had reduced the pour point by 6° C. The PPD has been used as flow improvers or Wax Inhibitors in all the case which retards the crystallization of wax in the pipeline.

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