# Polymethyl Methacrylate-Graphene Oxide nanocomposite Pour Point Depressant to improve the flow of Waxy Crude Oil through Pipelines

Prasenjit Talukdar[1], Ranjan Phukan[2], Joyshree Barman[1], Rituraj Singh[3], Gaurav Himanta Khaklari[2], Bhaskar Jyoti Saikia[1*]

[1]Department of Petroleum Engineering, DUIET, Dibrugarh University, India
[2]Department of Petroleum Technology, Dibrugarh University, India
[3]Government Polytechnic, Budaun-243601, Uttar Pradesh, India
Email: saikiabhaskar@dibru.ac.in

**Abstract:** Waxy crude oil is characterized by both light and heavy hydrocarbons, which pose great challenges in processing, transport, and storage due to the formation of paraffin wax and asphaltene. In order to address these flow problems, a nanocomposite-based polymeric pour point depressant (PPD) was synthesized and evaluated. The crude oils used were medium-heavy, with API gravities of 26.8 and 26.5, having a high pour point of 32–36 °C, thus requiring special treatment to ensure flow without interruption. The additives were checked for efficiency through pour point, rheological, and microscopic studies. Results show that the synthesized additives effectively lower the pour point and enhance the flow behavior. Untreated crude oil has pour points of around 36 °C, which shows poor flowability under cold conditions. The addition of PPD effectively lowers the pour point, with reductions of 6 °C, 9 °C, 12 °C, and 11 °C at concentrations of 250 ppm, 500 ppm, 750 ppm, and 1000 ppm, respectively. The optimum concentration is 750 ppm, achieving the lowest pour point of 24 °C. These results therefore underscore the capability of graphene-based nanocomposite PPD to act as a potentially effective flow assurance agent in the transport and handling of waxy crude oils.

Keywords: Waxy crude oil; Nanocomposite; Pour point depressant; Flow assurance; poly(methyl methacrylate)-graphene oxide(PMMA-GO), Rheological properties; Wax crystal morphology.

## 1. Introduction

The global energy demand, especially in the developing countries, is rising while the light oil reserves are dwindling. As a result, there is increased interest in the generation of highly paraffinic heavy crude oil. However, the production and transportation of such crude oils are problematic, particularly for subsea or uninsulated pipelines in colder climates. Their large temperature drops during transport put them at a considerable risk of wax-related flow assurance problems. This is because when the Wax Appearance Temperature (WAT) is surpassed by the falling crude oil temperature, some molecules of wax precipitate from the liquid phase into deposition on the inner pipelines walls. This wax deposition leads to a progressive reduction in the effective diameter of the pipeline, leading to increased pressure drops, more energy requirements for pumping, and, in extreme cases, complete flow blockages (El-Dalatony et al., 2019). The wax can also form a sort of gel structure in static sections of the pipeline, and restarting operations is extremely difficult and energy-intensive (Aiyejina et al., 2011)(Struchkov et al., 2020)(Chang et al., 1998). Heating and dilution are other methods of thinning the oil but they are expensive and not very effective (Abdurahman et al., 2012). The current studies focus on the application of pour point depressants (PPDs) in the improvement of the flow of crude oil by reducing the pour point (Borthakur et al., 1996)(Chen et al., 2010). The conventional polymeric PPDs have some issues such as low stability for long-distance transportation (Bai et al., 2019). Nanotechnology offers a promising opportunity for overcoming the difficulties that accompany the production and transportation of waxy crude oils, especially through the introduction of nanocomposite-based pour point depressants (Jia et

al., 2022). These novel materials combine the exceptional properties of nanoparticles with the functionality of polymeric additives, leading to a synergistic effect that improves the rheological behavior of the crude oil (Elbanna & Ahmed, 2023)(Sharma, Deka, et al., 2019). Nanocomposites generally consist of a polymer matrix attached to the surfaces of nanoparticles through functionalization techniques (Mao et al., 2020). This method takes advantage of the high surface area, reactivity, and nanoscale interactions of the nanoparticles, making nanocomposites more effective than the traditional polymeric PPDs (Wen et al., 2021)(Li et al., 2018). One of the major advantages of nanocomposite PPDs is their superior performance in modifying wax crystal formation. The nanocomposite's ability to improve dispersion and interaction capabilities allows the disruption of nucleation and growth of wax crystals to reduce the size of the crystals and change their morphology (Yao et al., 2018). This prevents the aggregation and deposition of wax on pipeline walls, lowering pour points greatly and improving flow properties (Mansourpoor et al., 2018). Nanocomposites also demonstrate excellent thermal stability, retaining effectiveness under extreme temperature fluctuations, like those in subsea pipelines or extremely cold environments. Their ability to enhance shear thinning behavior and reduce viscosity ensures smooth crude oil flow, hence less energy consumption and operation costs.

Since paraffin deposition is a major issue in the oil industry, this work presents the paraffin deposition tendency and the flow behavior of the waxy crude oil. The first is to determine the difficulties that are linked to the production and transportation of highly paraffinic crude oil and the second is to determine the solutions to the challenges (da Silva & Coutinho, 2004)(Fasano et al., 2004)(Bennema et al., 1992). In particular, this research aims to synthesize polymeric nanocomposite pour point depressant also known as flow improver to improve the flow characteristics of waxy crude oils in North East India. The work entails characterization of physico-chemical characteristics of Indian waxy crude oil samples and development of new polymeric pour point depressants for enhancing the flow characteristics of crude oil. It also concentrates on the functional alteration and integration of nanofillers such graphene into biopolymeric flow improvers and characterization of the same. Furthermore, the study involves identifying the viscosity, yield stress, microstructure, and wax particles of waxy crude oil systems (Abd et al., 2014).

## 2. Materials and Methods

### 2.1 Materials

Two crude oil samples were obtained from Oil India Limited, Assam and named them as sample I and sample II. Density was measured with a pycnometer, an accurate instrument that employs a liquid of known density. Specific gravity at 40 °C was used to determine crude oil quality by computing API gravity. The water content and sediments were determined using the centrifuge method (ASTM D 96-58 T) where a sample of crude oil and toluene was rotated at 2115 RPM to create a water layer and an oil layer. The pour point which is the temperature at which oil gels due to wax crystallization was determined using ASTM D97-06. The wax content was measured by dissolving the crude oil sample in n-pentane, adding acetone to precipitate the wax, cooling the solution, filtering, and re-dissolving the sample in n-hexane to remove asphaltenes. Rheological characterization was done using the MCR-72 Anton Paar Rheometer after preheating the samples above the Wax Appearance Temperature (WAT). The distribution of SARA (saturates, aromatics, resins, and asphaltenes) was used to explain crude oil composition with emphasis on asphaltenes' impact on viscosity and wax crystallization (Bisht et al., 2013). These methods offer broad information on the physical and chemical characteristics of crude oil, which is crucial for solving the flow and deposition problems.

Table 1 Physico-chemical Characterization of Crude Oil Samples

| Sl. No. | Parameter | Method | Sample I | Sample II |
|---|---|---|---|---|
| 1. | Density | IP 160/64 | 0.879 | 0.896 |
| 2. | API Gravity | ASTM Table | 26.8 | 26.50 |
| 3. | Pour point (°C) | ASTM D-97 | 36 | 32 |
| 4. | Water Content (%; V/V) | IP 74/64 | 2 | 1.5 |
| 5. | Wax Content (%; w/w) | modified UOP 46-64 | 15.5 | 15 |

Table 2 SARA analysis Data of Crude Oil Samples

| Sl. No. | SARA analysis: (%; w/w) | Sample I | Sample II |
|---|---|---|---|
| 1 | Saturates | 52 | 45 |
| 2 | Aromatics | 14 | 15 |
| 3 | Asphaltenes | 0.5 | 1.5 |

| | | | |
|---|---|---|---|
| 4 | Resin | 8 | 7 |
| 5 | Resin Asphaltenes | 11.5 | 14 |

From this analysis shown in Table 1 and Table 2, it was observed that the samples are medium heavy crude oils. Despite the relatively low water content, it was removed using a demulsifier for further studies. The pour point for all crude oil samples was notably high, and they were all highly paraffinic. Pour point has a linear correlation with wax content up to moderate levels while asphaltene content does not have much effect on it. Nonetheless, at very high concentrations, asphaltenes may decrease the pour point through enhancing the solubility of wax in the crude oil. Also, all the samples have very low sulfur content and hence they are classified as sweet crude oils.

For this study, a graphene-based nanocomposite PPD was prepared through several steps. First of all, graphene oxide (GO) was prepared from the graphite powder using the modified Hummer's method by chemical oxidation with potassium permanganate ($KMnO_4$) in sulfuric acid. This was done by combining graphite, $NaNO_3$, and $H_2SO_4$ in a three-necked round bottom flask, then adding $KMnO_4$ and stirring the mixture until it formed a thick paste upon heating. This paste was then dispersed in water and treated with $H_2O_2$ to reduce $KMnO_4$, which produced a bright yellow suspension that was then washed and dried to yield graphene oxide. The GO was then functionalized to produce polymerizable graphene oxide (vinyl-GO). This was achieved by dissolving GO in a water/dioxane solvent mixture and then adding HCl to the solution. To this suspension, acrylonitrile monomer was added and stirred for 4 hours to allow dispersion of the platelets. The solution was then subjected to NaCl to enhance the platelet aggregation at the water/organic interface and the functionalized platelets were then filtered off. This product was washed to eliminate any remaining unreacted acrylonitrile and other components. At last, the poly(methyl methacrylate)-graphene oxide (PMMA-GO) nanocomposite was prepared. Different quantities of vinyl-GO were dissolved in a flask containing dry toluene and subjected to ultrasonication. MMA monomer, which was also dissolved in toluene, was then incorporated into this solution to prepare various concentrations of PMMA-GO. The mixture was sonicated and stirred under nitrogen atmosphere, and then a benzoyl peroxide initiator was added to initiate the polymerization. The reaction mixture was cooled and kept for a while, and then the nanocomposite polymer was precipitated using methanol and dried to get a thin film of PMMA-GO nanocomposite (Sharma, Mahto, et al., 2019). The Tables 3, Tables 4 and Tables 5 outlines the specific amounts of materials used and the boundary conditions for the synthesis of poly(methyl methacrylate)-graphene oxide (PMMA-GO) nanocomposite.

Table 3 Graphene Oxide (GO) synthesis materials and boundary conditions

| Sl. No. | Materials | Concentration/Amount | Boundary Conditions |
|---|---|---|---|
| 1. | Graphite | 5 g | Room temperature, constant stirring |
| 2. | $NaNO_3$ | 2.5 g | Room temperature, constant stirring |
| 3. | $H_2SO_4$ | 100 ml | Room temperature, constant stirring |
| 4. | $KMnO_4$ | 15 g | Add gradually, heat to 40 °C, stir until paste forms |
| 5. | Water | 1 liter + 500 ml (hot water) | Heat to 90 °C with stirring, treat with 30% $H_2O_2$ |

Table 4 Polymerizable Graphene Oxide (Vinyl-GO) synthesis materials and boundary conditions

| Sl. No. | Materials | Concentration/Amount | Boundary Conditions |
|---|---|---|---|
| 1. | GO | 1.5 g | Room temperature, constant stirring |
| 2. | Water/Dioxane Mixture | 75/25: v/v, 50 ml | Room temperature, constant stirring |
| 3. | HCl | 0.5 ml | Room temperature, constant stirring |
| 4. | Acrylonitrile Monomer | 50 ml | Room temperature, constant stirring |
| 5. | NaCl | 5% | Water/organic interface, facilitate platelet aggregation |

Table 5 PMMA-GO Nanocomposite synthesis materials and boundary conditions

| Sl. No. | Materials | Concentration/Amount | Boundary Conditions |
|---|---|---|---|
| 1. | Vinyl-GO | 0.01 g, 0.03 g, 0.05 g, 0.1 g | Room temperature, constant stirring |
| 2. | MMA Monomer | Different amounts to form 0.1%, 0.3%, 0.5%, and 1.0% PMMA-GO | Dispersed in toluene (40 wt%) |
| 3. | Dry Toluene | 15 ml | 5 °C, under sonication |
| 4. | Benzoyl Peroxide (BOP) Initiator | 0.03 g in 1 ml toluene | 75 °C, constant stirring |
| 5. | Methanol | 50 ml | Precipitation, dry in hot air oven at 60 °C for 6 hours |

| | | | |
|---|---|---|---|
| 6. | Reaction Temperature | 75 °C, cool to 55 °C | Nitrogen gas purged through reaction system |
| 7. | Sonication Time | 30 min at 5 °C, additional 15 min after MMA addition | |
| 8. | Reaction Time | 30 min at 75 °C, additional 6 hours at 55 °C | |

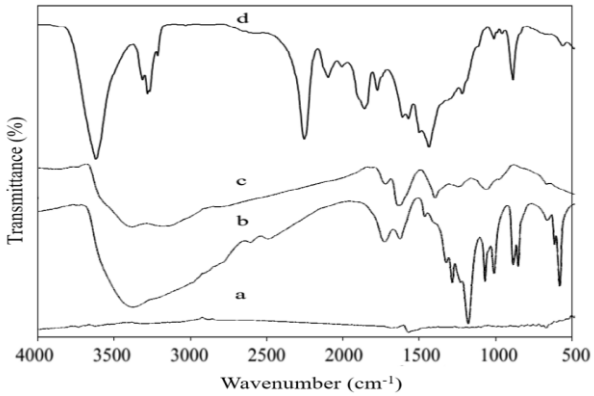


Fig. 1 FT-IR spectra of (a) Graphite, (b) GO, (c) Vinyl-GO and d) PMMA-1%GO

The structure of the nanocomposite was confirmed by FTIR spectroscopy (Fig. 1). Graphite was observed to have weak bands because of the compactness of the structure, whereas GO presented strong absorption bands that validate the successful synthesis of GO from graphite. These bands specify were carbonyl, carboxylic, epoxy, and hydroxyl groups.

The Pinner reaction grafted a vinyl group onto GO, evidenced by the band near 1400 $cm^{-1}$, along with peaks at 1730 $cm^{-1}$ (esterification), and 1626 $cm^{-1}$ (O-H stretching or unoxidized graphene). The suppression of other bands and a reduction in the peak at 3398 $cm^{-1}$ confirmed the participation of hydroxyl groups in the esterification reaction. For the PMMA-1% GO nanohybrid, FTIR exhibited characteristic peaks at 2930 $cm^{-1}$ and 1720 $cm^{-1}$, corresponding to C—H and C=O stretching, along with extra peaks at 2992, 2967, 1731, 1450, and 1179 $cm^{-1}$ for PMMA. Thus, these results have confirmed the successful synthesis of nanocomposites.

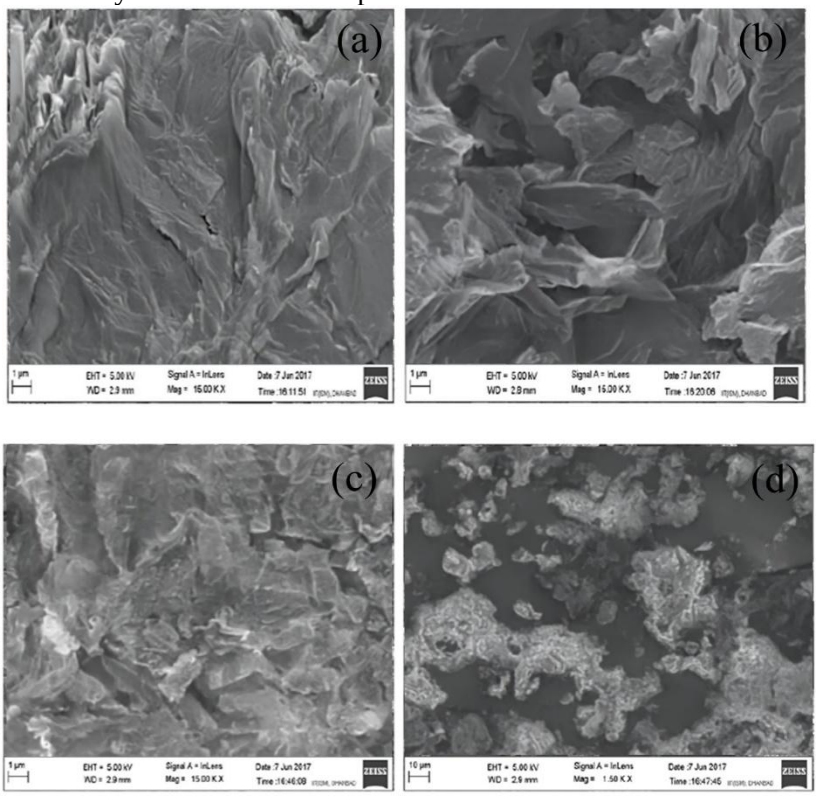


Fig. 2 FESEM images of (a) Graphene oxide, (b) Vinyl Graphene oxide, (c) & (d) PMMA-1% GO

Field-emission scanning electron microscopy (FESEM) (Fig. 2) was employed to investigate the morphological features of Graphene oxide, Vinyl Graphene oxide, PMMA-1% GO. As can be seen in Fig. 2, the vinyl-GO in PMMA-1%GO nanohybrid is well dispersed and showed high dense packed distribution. The vinyl-GO platelets less than 3 μm is observed that shows the MMA monomer is polymerized with vinyl-GO avoids agglomeration of GO sheets.

## 3. Results and Discussion

Experimental investigations were conducted to find the ability of pour point depressants (PPDs) in lowering the pour point of Indian waxy crude oils. Testing followed the standard ASTM method in which crude oil samples were heated at 150 °F, thereby melting waxes with gradual cooling. The fluid movement was then noted after every temperature drop of 3 °C. The lowest temperature at which no fluid movement was observed was recorded as the pour point. For treated crude oils, PPDs were added at certain concentrations and stirred to homogenize them before testing.

The synthesized nanocomposite Graphene-based PPD (G1) was evaluated in this study at the concentration range of 250 ppm to 1000 ppm.

Table 6 Effect of Graphene-based nanocomposite PPD on pour point of waxy crude oil

| Sl. No. | Concentration of PPD (ppm) | Pour point in °C (Virgin Crude Oil) | Pour point in °C (treated with G1) | Δ T (Virgin – Treated) |
|---|---|---|---|---|
| 1. | 250 | 36 | 30 | 6 |
| 2. | 500 | 36 | 27 | 9 |
| 3. | 750 | 36 | 24 | 12 |
| 4. | 1000 | 36 | 25 | 11 |

The synthesized PPD presented significant reductions in pour point shown in Table 6. The PPD successfully lowered the pour point of the crude oil but differed with the result depending upon the concentration. The highest reduction in pour point was shown at 750 ppm. If exceeded this limit, its performance dropped due to the crystallization effects.

The rheological behavior was characterized with a cone-and-plate rheometer (Anton Paar MCR-72) whose diameter of the cone was 50 mm at an angle of 2°, ensuring uniform distribution of stress, shear rate, and temperature. After heating at the temperatures of 70–80 °C to melt the crystalline material, samples were cooled stepwise from 60 °C down to the pour point in 10 °C steps. Wax crystal structures were investigated using a cross-polarized light microscope (OLYMPUS UC-30), and the subsequent evolution was captured by a camera.

3.1 Influence of Temperature on Rheological Behavior

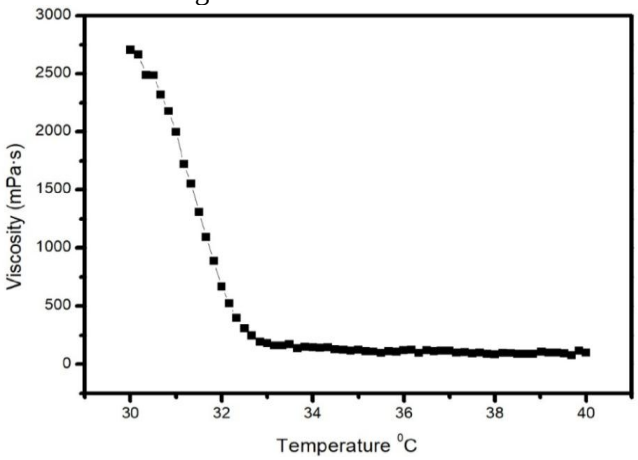


Fig. 3 Temperature effect on crude oil

Temperature has a significant effect on crude oil rheology, especially below the pour point (Fig. 3). With a decrease in temperature, the dissolved wax components saturate and form solid structures that increase viscosity and yield stress. Crude oil shows non-Newtonian behavior at low temperatures. Its apparent viscosity increases with the shear rate. On the other hand, since the structure disintegrates at high temperatures (over the pour point), the oil will behave like a Newtonian fluid and its viscosity will fall linearly. This is because breaking the wax network caused by the applied stress allows continuous flow. The rate of change of viscosity with temperature is faster at lower shear rates.

3.2 Effect of Shear Rate on Rheological Behavior

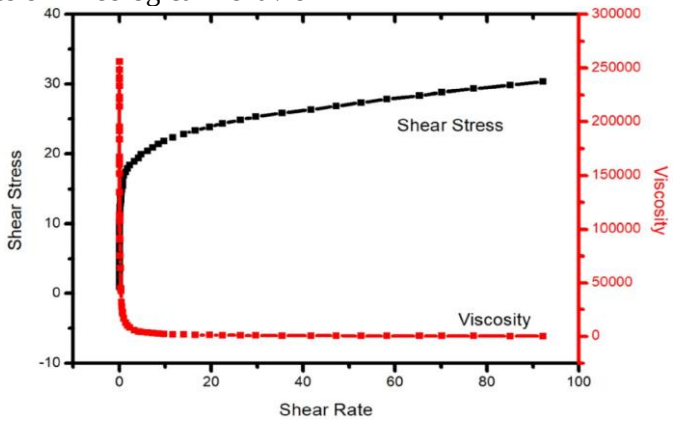


Fig. 4 Effect of Shear Rate on Shear Stress and Viscosity of crude oil

Shear rate affects the viscosity and structural behavior of crude oil (Fig. 4). At low shear rates, wax crystals remain aggregated, and no significant structural change occurs. As shear rate increases, the crystal network begins to break down, reducing viscosity and facilitating flow. Below the pour point, shear stress initially increases with shear rate due to crystal entanglement but eventually peaks and decreases as the network disintegrates. At higher shear rates, viscosity stabilizes, indicating complete structural breakdown. The shear rate effect behaves similarly to flow improvers-breaking up crystal agglomeration and lowering the flow resistance. Temperature and shear rate influence the rheology of Indian waxy crude oils. When temperature exceeds pour point, oil behaves as a Newtonian fluid with fewer viscosity variations. At pour point temperature, it shows non-Newtonian behavior because the creation of wax crystals. Shear rate would play a major role in breaking the wax networks, reducing the viscosity, and providing improvement in flow. Such findings would be crucial for the optimization of flow characteristics of waxy crude oils with different operation conditions.

3.3 Effect of concentration of Polymeric Flow Improvers on the rheological behaviour of Indian waxy crude oils

The impact of polymeric flow improvers on the rheology of Indian waxy crude oils was investigated at different concentrations for synthesized PPD, for which 750 ppm was identified as the optimum concentration for the lowest pour point. Viscosity increased with the decrease in temperature due to wax crystallization but decreased with an increase in shear rates as the wax molecules destabilized. Synthesized nanocomposite PPD significantly reduced viscosity which proved to be effective.

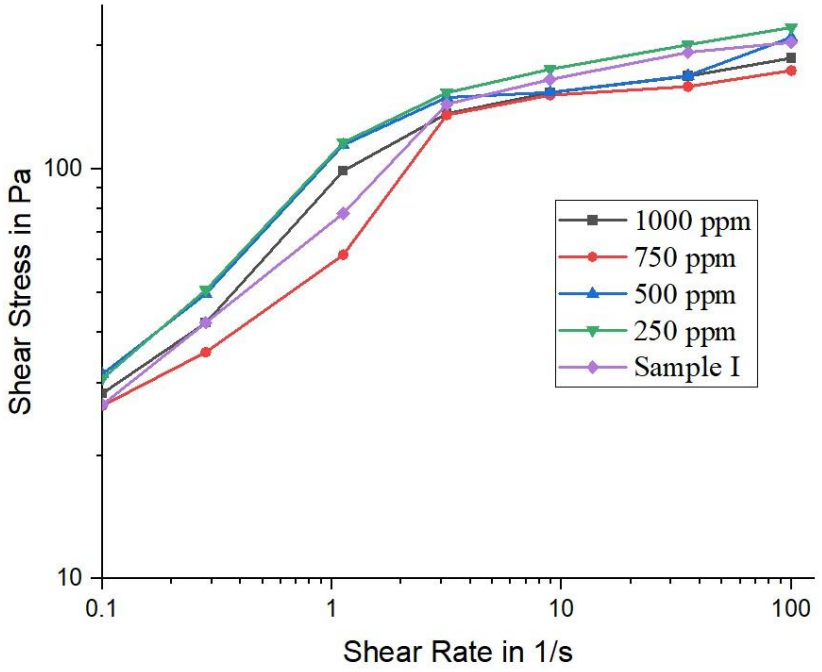


Fig. 5 Rheology of Crude Oil Sample I treated with PPDs at different concentrations

The rheological behavior of Sample I crude oil and PPD-treated crude oil at different concentrations is illustrated in Fig. 5. Shear stress increases with shear rate for all the concentrations, meaning that this system is non-Newtonian. Crude oil with no treatment (Sample I) exhibits the greatest shear stress and thus is considered to be the one having the greatest wax crystal agglomeration and resisting flow most. As the concentration of PPDs increases, the shear stress decreases, especially at intermediate concentrations, with 750 ppm showing the most significant decrease in shear stress. At higher concentrations than this (for example, 1000 ppm), the performance is slightly decreased, which may be due to crystallization effects or reduced efficiency. These results show that PPD can break up wax crystal networks, enhance flowability, and decrease resistance, especially at optimized concentrations.

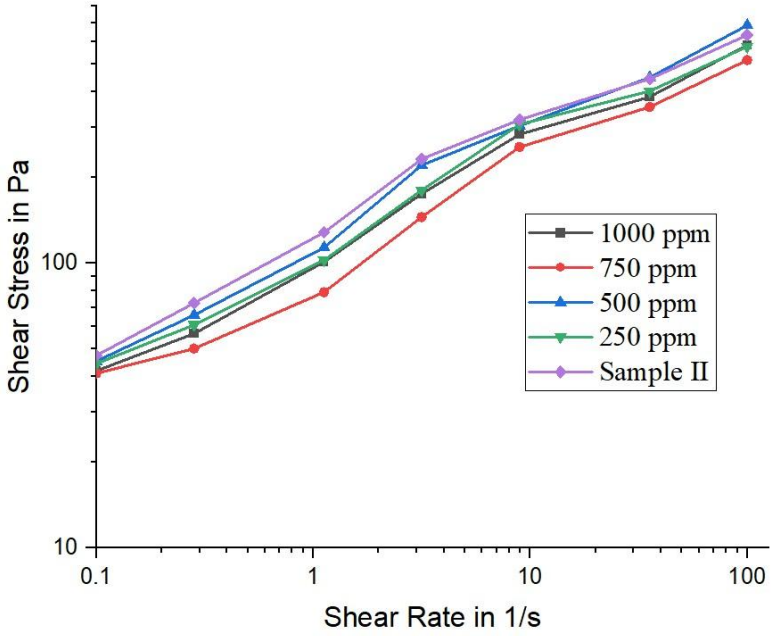


Fig. 6 Rheology of Crude Oil Sample II treated with PPDs at different concentrations

Fig. 6 shows the rheological properties of Sample II crude oil as a function of different concentrations of PPD. Similar to Sample I, untreated Sample II had the highest shear stress indicating a significant level of wax crystal aggregation and structural rigidity. At all shear rates, shear stress is decreased with an addition of PPD. At a concentration of 750 ppm, the largest drop is observed. This demonstrates the best possible performance of PPDs in breaking down wax crystal networks and maximizing flowability. A 1000 ppm level reduces shear stress with much less significant magnitude, thus implying the law of diminishing returns or crystallization at higher concentration levels. This shows that at optimal dosing levels, PPD significantly inhibit flow resistance in waxy crude oils.

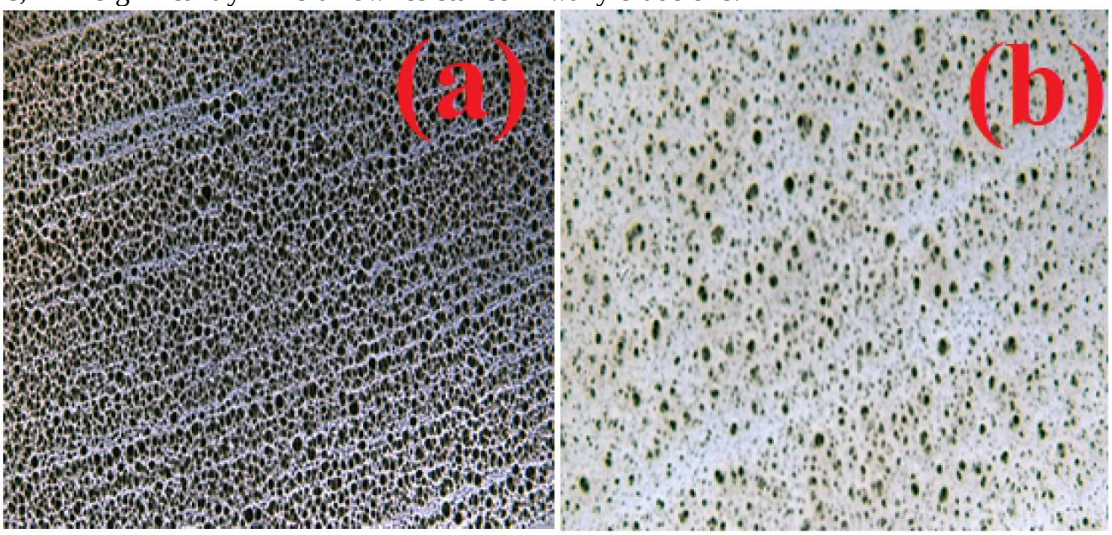


Fig. 7 Microscopic properties of Crude Oil Sample I at various temperatures a) at below pour point b) at 40 °C

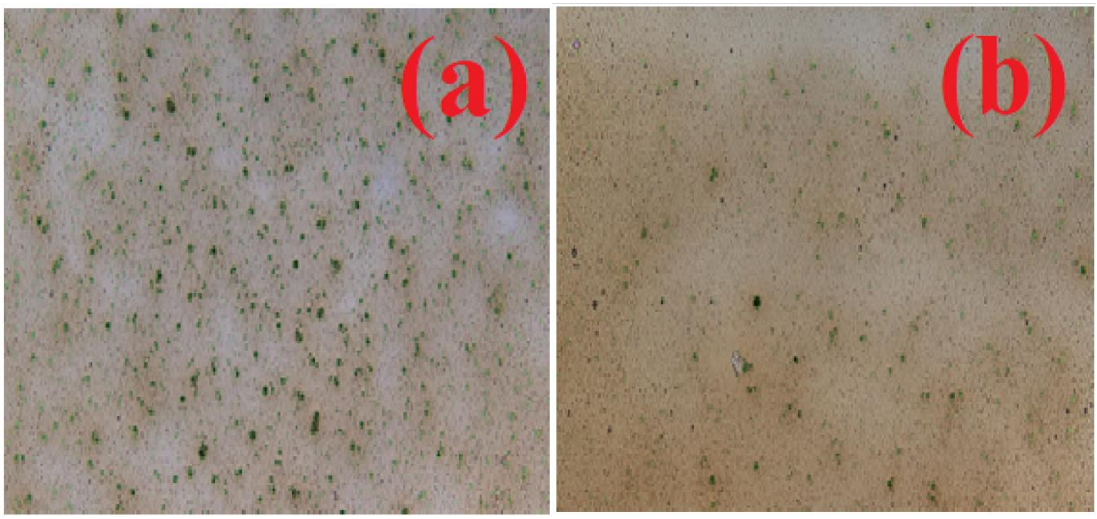


Fig. 8 Microscopic properties of Crude Oil Sample I treated with a) commercial PPD b) at synthesized nanocomposite PPD

Waxy crude oils form crystalline structures at lower temperatures due to decreased solubility of n-paraffins, resulting in gel-like, high-viscosity materials. The crystals interlock into dense 3D networks that trap fluid and hence increase viscosity. Polymeric flow improvers modify the wax crystal morphology by reducing the size of the crystals and changing shapes from needle-like to spherulitic structures. As a result, aggregation is prevented and the crystal network is weakened, thereby dramatically reducing pour points, viscosity, and yield stress. The effectiveness of these additives depends on oil composition and concentration, but with optimal values realized at intermediate concentrations. Overuse can increase crystal nucleation, while underuse fails to disrupt the wax structure effectively. Fig. 7 shows the microscopic image of crude oil at different temperatures and Fig. 8 shows the microscopic images of crude oil with nanocomposite PPD.

## 4. Conclusion

Crude oil samples were collected from Oil India Limited, Assam, and evaluated using a synthesized nanocomposite pour point depressant (PPD) to ensure the fluidity properties. The crude oil samples were subjected to pour point, rheological, and microscopic analysis to evaluate the effectiveness of these additives as flow improvers. The crude oil samples were waxy, medium-heavy characteristics with an API gravity of 26.8 and 26.5. Before tests, the raw oils were heated to melt the waxes and the water content (1.5–2%) was separated and taken out through centrifugation. The pour points of the raw oils ranged between 32–36 °C, necessitating treatment to ensure stable and uninterrupted flow with minimal pressure loss caused by wax deposition. This study indicates that the polymeric flow improvers cause a change in the precipitation temperature of waxes. Since the effectiveness of crude oil treatment depends on its asphaltene and wax content, the treatment concentrations should be optimized within the range of 250–1000 ppm. Overdose would create crystallization conditions, eliminating the advantage. Rheological studies showed that crude oils exhibited non-Newtonian flow at low temperatures and shearing conditions. Viscosity decreased with increase of shear rate, stabilizing at higher rates. Below the pour point, wax accumulation led to increases in viscosity. Adding flow improver caused significant lowering of pour points and the ability to flow smoothly using less energy, hence a more economical process. Microscopic studies showed that crystallization morphologies were dependent on temperature, cooling rate, and additive concentration. With cooling, the wax particle size increased, leading to greater agglomeration. However, the addition of additives transformed the wax crystals from plate-like structures to spherulitic forms, reducing interlocking and improving flow. This study emphasizes the need for additives that are meant to lower pour points and deter wax deposition, thus enhancing the waxy crude oil flow.

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Conflict of interest
The authors declare that they have no competing interests, financial or non-financial, that are directly or indirectly related to the work submitted for publication.
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Abbreviations
API - American Petroleum Institute
ASTM - American Society for Testing and Materials
PPD - Pour Point Depressant
WAT - Wax Appearance Temperature
GO - Graphene Oxide
PMMA-GO - Poly(methyl methacrylate)-Graphene Oxide
MMA - Methyl Methacrylate
SARA - Saturates, Aromatics, Resins, and Asphaltenes
IP - Institute of Petroleum
RPM - Revolutions Per Minute
$KMnO_4$ - Potassium Permanganate
$NaNO_3$ - Sodium Nitrate
$H_2SO_4$ - Sulfuric Acid
$H_2O_2$ - Hydrogen Peroxide
HCl - Hydrochloric Acid
NaCl - Sodium Chloride
UOP - Universal Oil Products
V/V - Volume/Volume
W/W - Weight/Weight
ASTM - American Society for Testing and Materials
BOP - Benzoyl Peroxide
CFD - Computational Fluid Dynamics
FESEM - Field Emission Scanning Electron Microscopy
FTIR - Fourier Transform Infrared Spectroscopy
MCR - Modular Compact Rheometer
MMA - Methyl Methacrylate
PMMA - Polymethyl Methacrylate
PPD - Pour Point Depressant

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