Iron Oxide and Graphene Oxide nanocomposite with Polymethyl Methacrylate as innovative Pour Point Depressant for Waxy Crude Oil of North-East India

Gaurav Himanta Khaklari[1], Prasenjit Talukdar[2*]

[1]Department of Petroleum Technology, Dibrugarh University, Dibrugarh-786004, Assam, India
[2*]Department of Petroleum Engineering, DUIET, Dibrugarh University, Dibrugarh-786004, Assam, India

***Corresponding Author:** Dr. Prasenjit Talukdar
*E-Mail: prasenjit_duiet@dibru.ac.in

Abstract
This paper focuses on the transportation challenges associated with waxy crude oil found in the North-Eastern region of India, particularly in Assam. The high paraffin and wax content in the crude oil leads to issues such as high pour point and viscosity, which can obstruct the flow of oil and cause disruptions in the transportation process. In this study, a composite material consisting of Graphene Oxide (GO) and Iron Oxide (Fe_3O_4) was synthesized and evaluated in crude oil rheology test. This study includes the characterization of the waxy crude oil sample e.g., specific gravity, API gravity, pour point, water content, wax content, FTIR analysis and SARA distribution. The dispersion stability of the rGO-Fe_3O_4 composite material was tested in polar (water) and nonpolar (xylene) solvents. The composite material showed high hydrophobicity and remained insoluble in water, while it dissolved in xylene. The rheological test for the crude oil and Pour Point depressant (PPD) such as viscosity, shear stress, storage modulus, loss modulus etc., were also performed. The crude oil sample's pour point was effectively reduced by 18°C with the application of a 500-ppm dosage. The findings of this research contribute to a better understanding of the challenges associated with transporting waxy crude oil and offer insights into the potential use of nanocomposite PPDs (NPPD) for improving the flow properties of the oil.

Keywords: Flow Improver, Pour Point Depressants, Rheological Properties, Wax Deposition, Nanocomposite, North East India

1. Introduction

North-East India has substantial reserves of crude oil, with most of the production concentrated in the Upper Assam Basin of India [1]. The basin contributes a significant portion of India's domestic oil production. The crude oil found in Assam is known for its high viscosity and heavy nature. It is characterized by a mixture of high paraffinic and wax content, and exhibits challenging flow behavior at low temperatures. The elevated wax content contributes to a high pour point, causing the crude to behave as a non-Newtonian fluid below this temperature threshold. Crystallization of wax occurs during cooling, leading to the formation of either a gel or a partial gel and therefore specialized refining processes is required to obtain valuable petroleum products. Several public and private sector companies are involved in oil exploration and production in Assam. The Oil and Natural Gas Corporation (ONGC) Limited and Oil India Limited (OIL) are the primary players in the region.

These companies employ advanced drilling techniques and technologies to extract crude oil from the reservoirs [2].

The waxy crude oil extracted from the heavy oil reservoirs in Assam poses significant challenges during winter, particularly when the temperature drops below the Wax Appearance Temperature (WAT). As temperatures drop, the solubility of wax in crude oil decreases, prompting the formation of wax crystals. This phenomenon intensifies as the quantity, size, and aggregation of these crystals increase. Beginning as small rod particles at higher temperatures, the crystals undergo a transformation, gradually evolving into complex aggregates with distinct fractal characteristics [3]. This leads to the precipitation of waxy components on the pipeline's inner walls, causing restrictions in smooth flow [4]. Sun et al. (2000) conducted research on how changes in seasonal temperatures affect the behavior of crude oil, noting that temperature reduction near the pipeline's internal boundary wall induces crystallization more than at the axis [5][6][7]. Additional decrease in temperature may lead to the development of gel on the inner surface, causing a shift in the rheological properties of the crude oil from being Newtonian to Non-Newtonian, thereby increasing the energy required for transportation. Transporting waxy crude oil during winter months is costly and challenging, necessitating pre-treatment to improve flowability. Changes in parameters like API gravity, viscosity, and rheological properties are crucial for easier transportation. Employing oil-soluble surfactants or polymeric additives prior to pumping into pipelines aids in preventing wax deposition, thereby tackling the long-standing challenge of oil gelation encountered by the Exploration and Production (E&P) sector in pipeline transportation systems over the years.

Different strategies are implemented to control wax deposition when transporting such crude oil through pipelines [8][9][10]. These include insulating pipelines and applying external heat sources which can help keep the temperature of the crude oil above the Wax Appearance Temperature (WAT) to avoid wax

deposition [11], pipeline pigging [12], proper pipeline design [13], including insulation and flow control mechanisms, all these can minimize the risk of wax deposition. Regular inspections, cleaning, and monitoring of pipelines are crucial to identify and address potential wax deposition issues promptly [14]. One of the most effective methods is the use of specialized chemicals known as pour point depressants or wax inhibitors which can be added to the crude oil to modify its wax crystallization behavior [15]. These additives assist in decreasing the pour point and diminishing the chances of wax deposition. This proactive method aids in averting significant blockages and guarantees the secure and effective conveyance of crude oil.
In this work, a nanocomposite pour point depressant comprising Graphene Oxide and Iron Oxide was created, and its effect on the rheological properties of waxy crude oil was investigated by examining its impact on the morphology of wax crystals and the rheology of Assam crude oil. Finally, a complete analysis of the rheology of waxy crude oil was conducted to determine its compatibility for pipeline transportation of waxy crude oil.

1.1 Challenges

Transporting heavy waxy crude oil through pipelines poses numerous hurdles owing to its distinctive characteristics. A notable obstacle arises from its elevated viscosity and tendency to solidify when exposed to lower temperatures. As the oil moves through pipelines, especially in colder climates or during transportation over long distances, it can solidify, leading to flow restrictions, increased pumping pressures, and potential pipeline blockages. Moreover, the presence of wax deposits along the pipeline walls can worsen these issues, requiring frequent maintenance and cleaning operations to ensure uninterrupted flow. Additionally, the high viscosity of heavy waxy crude oil increases frictional losses and energy consumption during transportation, impacting operational efficiency and economics. In order to overcome these difficulties, creative approaches such as employing sophisticated heating mechanisms, incorporating chemical enhancements, or designing pipelines with specialized features might be necessary to ensure the continuous flow of heavy waxy crude oil and enhance the efficiency of pipeline operations.

1.2 Motivation

The global reliance on crude oil underscores the importance of addressing its transportation challenges, particularly its high pour point in cold climates. Conventional pour point depressants (PPDs) have limitations, prompting exploration into innovative solutions. Nanoparticles offer unique properties that can enhance PPD performance, especially when incorporated into nanocomposite formulations.

1.3 Objectives

The research aims to develop and evaluate nanocomposite PPD, focusing on their efficacy, and stability. Through the implementation of these innovative strategies, the authors aim to enhance the effectiveness, safety, and environmental friendliness of crude oil transportation, thereby offering advantages to both industry participants and the ecosystem.

2. Literature review

Esterification is a chemical reaction where an alcohol (in this case, 1-docosanol) reacts with a carboxylic acid group present in the PEAA polymer chain. The PTSA catalyst facilitates this reaction by increasing the rate of ester formation without being consumed itself. As a result of this catalyzed esterification process, ester linkages are formed between the PEAA polymer and the 1-docosanol molecule, by using this process Atta et al. (2011) synthesized an effective additive that demonstrated substantial reductions in pour point and viscosity when added to crude oil samples. Notably, increasing the size of the alkyl group in the copolymer composition enhanced its performance. The findings suggest that the prepared PEAA copolymeric additives hold potential as pour point depressants and flow improvers for crude oils, particularly at concentrations of 5000ppm. However, discrepancies between pour point reduction and rheological results highlight the complexity of wax particle behavior, warranting further investigation to optimize additive performance for diverse crude oil compositions [16].

A study found that the EVAc/CG compositions exhibited superior performance compared to pure EVAc when used as PPDs for waxy crude oil. In 2020, Marenov et al. introduced a new method aimed at improving the effectiveness of ethylene-vinyl acetate copolymer (EVAc) as pour point depressants (PPDs) for waxy crude oil. This improvement was achieved by incorporating crude gossypol (CG), which was isolated from refined cottonseed oil soapstock, into the EVAc polymer. The process involved synthesizing compositions of EVAc and CG through joint milling, where both materials were mixed together and processed. The resulting EVAc/CG compositions were then evaluated for their performance as pour point depressants. The blends exhibited improved flow characteristics, which can be attributed to the incorporation of extra nonpolar and polar groups into the composite structure. These groups, different from wax crystals, contribute to the understanding of the mechanism behind the enhanced performance [17].

Xie et al. (2020) examined how applying both electrical field treatment and Ethylene-Vinyl Acetate Copolymer (EVA) treatment impacts the flow characteristics of waxy crude oil, with the goal of mitigating flow assurance issues during pipeline transportation. Findings suggest that employing both treatments together leads to more pronounced decreases in viscosity, yield stress, and thixotropy compared to applying each treatment separately. However, while the combined treatment outperforms singular treatments under low shear conditions, the enhancement in flowability resulting from the combined treatment is not as significant as the total improvement achieved by applying the electric field and EVA treatments separately. Microscopic analysis reveals broader size distribution of wax particles with the combined treatment, contributing to enhanced flowability. Both shearing and reheating deteriorate treatment performance, with the electric field's viscosity-reducing effect diminishing completely after prolonged shearing or reheating above the Wax Disappearance Temperature (WDT), while the chemical performance may be partially restored after reheating above the WDT. Overall, while the combined treatment shows promise, its robustness against shearing and reheating remains limited compared to individual treatments, particularly under low shear conditions [18].

Ridzuan et al. (2020) analyzed the effectiveness of Pour Point Depressant (PPD) and nanoparticle, sodium cloisite Na+, in treating Malaysian crude oil to mitigate wax-related issues in pipelines. Results demonstrated that both PPD and the PPD/nanoparticle blend significantly reduced viscosity, with the blend showing an 8% reduction compared to 4% with PPD alone. Furthermore, the blend exhibited a higher Performance Improvement Efficiency (PIE) of 88.27% at a cold finger temperature of 15°C. This indicates that the addition of nanoparticles enhances the dispersion of wax crystals, resulting in softer deposits and improved flowability. The study suggests that the blend of PPD and nanoparticles offers a promising solution for reducing wax deposition and viscosity in crude oil pipelines, potentially lowering production costs and improving operational efficiency in the oil industry [19].

Studies by Yanhu et al. (2020) presents crucial findings on the thermal interaction between buried warm-oil pipelines and permafrost in Northeastern China, focusing on the China-Russia Crude Oil Pipeline (CRCOP). Field observations and numerical simulations were conducted to assess the impact of pipeline heat loss on permafrost thawing and propose a novel design integrating seasonal air-cooled embankments (SACE) and pipe insulation to mitigate these effects. Results indicate that without insulation, rapid permafrost thawing occurs along the pipeline's right-of-way (ROW), with substantial soil temperature depth (STD) increases. However, with the proposed design, STD reductions of up to 50% are achievable, significantly slowing permafrost thawing rates. Additionally, the SACE effectively manages heat transfer, preventing extensive permafrost degradation for at least two decades. Overall, these findings underscore the importance of innovative design approaches to control ground thermal regimes in cold regions, ensuring the long-term stability of pipeline systems and mitigating environmental impacts [6].

Jia et al. (2022) investigated the potential of utilizing carbon nanomaterials as pour point depressants (PPDs) in the oil industry. Three types of carbon-based hybrid nano-PPDs were synthesized by modifying a basic poly-a-olefins-acrylate high-carbon ester PPD with graphene oxide (GO), carbon nanospheres (Cna), and oxidized carbon nanotubes (OCNTs) through a solvothermal method. Characterization analyses confirmed successful polymerization of the basic PPD onto the carbon nanomaterials. Evaluation of the nano-PPDs using simulated oil revealed significant improvements in pour point reduction and viscosity. Particularly, the PPD modified with 1% OCNTs demonstrated the most effective pour point reduction, surpassing the basic PPD by 5°C. The study elucidates the mechanisms underlying the enhanced performance of carbon-based nano-PPDs, attributing it to the unique structures and characteristics of the carbon nanomaterials, which facilitate efficient wax crystal morphology modification and nucleation. This research expands the application scope of carbon nanomaterials and provides innovative insights for the development of efficient PPDs in the oil industry [20].

In addressing the challenge of pipeline blockage from waxy deposits in the Shengli oilfield, Yuan et al. (2023) developed eight novel polyacrylic acid pour point depressants (PPDs) tailored for oil field in Shandong province, China. These PPDs, synthesized through copolymerization of maleic anhydride and ene monomers featuring diverse polar and aromatic pendant chains, were characterized using techniques like FTIR, NMR, GPC, and POM, revealing promising results. The research showcased the efficacy of each PPD variant in significantly reducing
the pour point of the crude oil featuring elongated alkyl side chains and aromatic units, showing remarkable performance, lowering pour points by up to 12°C. Additionally, the study found that the introduction of these depressants changed the wax crystallization pattern in the crude oil, resulting in the formation of larger and more densely packed wax crystal clusters, thus enhancing the flowability of the crude oil. These discoveries highlight the promising potential of modified maleic anhydride co-polymers as efficient pour point depressants for waxy crude oil not only in the Shengli oilfield but also in other locations [21].

Elkatory et al. (2023) tackled the significant challenge of paraffin buildup in oil pipelines during transportation and start-up procedures at low temperatures. They explored the effectiveness of pour point depressants (PPDs) in altering paraffin crystallization and enhancing the flow of waxy crude oil. Comb-type copolymers, synthesized via free radical polymerization of maleic anhydride with benzyl oleate followed by amidation with stearyl amine, were evaluated for their capability to decrease the pour point of crude oil by as much as 3°C at a concentration of 2000 ppm. Structural characterization methods like Fourier Transform Infrared and X-ray diffraction were employed, along with examinations of potential interactions between PPDs and waxes using techniques such as differential scanning calorimetry and microscopy. The copolymer, Poly(benzyl oleate-co-distearyl amine), exhibited promising outcomes in hindering paraffin crystallization, reducing crystal size, and preventing the formation of crystal platelets, indicating its potential as an effective modifier for enhancing crude oil flowability [22].

Pal et al. (2022) explored the utilization of shikakai fruit extract (SE) as a bio-based additive to enhance the flow characteristics of waxy crude oil (WCO) and decrease pumping expenses during pipeline transportation. SE was produced using a solvent extraction technique and characterized using various analytical techniques. Addition of 1,000 ppm of SE to WCO resulted in a notable decrease in pour point by 12°C and a significant reduction in viscosity, offering an alternative to the application of heat during oil flow. Microscopic investigations indicated that crude oil treated with SE showed reduced wax particle size and more even dispersion, inhibiting the formation of three-dimensional networks and facilitating easier flow. Wax deposition was substantially reduced after SE addition, ensuring smoother pipeline transportation. The biodegradable nature of SE suggests its potential as a cost-effective solution for improving WCO flowability without requiring special treatment before refining [23].

Alpandi et al. (2022) tested the effectiveness of plant-derived inhibitors, such as Jatropha seed oil (JSO), crude palm oil, and crude palm kernel oil (CPKO), in inhibiting wax deposition and improving rheological properties of Malaysian waxy crude oil. Results showed that JSO, CPKO, and crude palm oil significantly reduced wax deposition and lowered the viscosity of the crude oil compared to synthetic chemical inhibitors. Specifically, 5% JSO and 1% CPKO were identified as the most effective viscosity-reducing agents at 60°C below the wax appearance temperature. These findings suggest that natural plant-based additives offer a promising solution for reducing wax deposition waste and minimizing environmental contamination compared to synthetic alternatives. Further research is recommended to explore the application of these additives with various types of waxy crude oils and under dynamic flow conditions, as well as to conduct comprehensive characterization of crude oil properties for future studies [24].

In tackling the challenges of heavy oil demulsification in the Liaohe Oilfield, Wei et al. (2021) synthesized FB series fluorinated polyether demulsifiers from trifluoromethyl phenol and formaldehyde, confirming their synthesis through FTIR spectra analysis. These demulsifiers effectively reduced the surface tension of oil-water interfaces and disrupted natural polar substances, facilitating water removal. By conducting water removal experiments using Liaohe Oilfield heavy oil emulsions, the optimal demulsification temperature and dosage of FB series demulsifiers were determined. Results indicated that at 100 mg/L dosage and 60°C temperature, FB 4 demulsifier achieved a remarkable water removal percentage of 90.33%, surpassing existing demulsifiers used in the Liaohe Oilfield. This research signifies the potential of FB series demulsifiers in enhancing demulsification efficiency amid evolving crude oil emulsion complexities [25][26].

Ning et al. (2022) studied the efficacy of ethylene vinyl acetate (EVA) and EVA/SiO_2 nanohybrids in inhibiting wax deposition and improving the fluidity of Shengli waxy oil. Findings obtained through techniques such as differential scanning calorimetry, thermal X-ray diffraction, and polarized optical microscopy indicate that nanohybrids improve the effectiveness of EVA in lowering the wax appearance temperature and altering the morphology of wax crystals. EVA/SiO_2 nanohybrids demonstrate superior performance in decreasing viscosity, inflection point, and yield stress of the crude oil compared to EVA alone. Optimal dosages of EVA and SiO_2 nanoparticles are identified for maximizing flow improvement. Controlled stress tests further demonstrate the benefits of nanohybrids in maintaining crude oil flow. This study provides valuable insights into the mechanisms underlying the flow improvement of crude oil by nanohybrid materials, offering practical guidelines for the development of effective flow improvers in the oil industry [27].

Sharma et al. (2019) explored the application and development of novel polymer nanocomposites, specifically poly(2-ethylhexyl acrylate)−graphene oxide [P(2-EHA)−GO], as potential pour point depressants (PPDs) for waxy crude oil. Through in situ free-radical polymerization, nanocomposites with various concentrations of GO were synthesized and characterized. Results demonstrated that the optimized concentration of 1 wt % GO in the nanocomposite effectively improved the flow characteristics of the crude oil, leading to a notable reduction in pour point and apparent viscosity. Microscopy studies revealed the mechanism behind this improvement, attributing it to the nucleation and cocrystallization mechanism facilitated by GO, which helps disperse wax crystals in the crude oil. Long-term stability tests further supported the effectiveness of P(2-EHA)−GO nanocomposites in maintaining flow performance over a 30-day period. Overall, this study underscores the potential of graphene-based polymeric nanocomposites as effective PPDs, offering significant benefits for flow assurance in the petroleum industry.

Cashew Nut Shell Liquid (CNSL) extracted from waste cashew nut shells was investigated as a natural flow enhancer for transporting heavy crude oil through pipelines by Pandian et al. (2021). The CNSL was thoroughly characterized, and its effectiveness as a flow improver was evaluated through pour-point and rheology measurements, as well as optical micro-imaging analysis. Results showed a significant enhancement in flow properties, particularly at an optimum dosage of 2000 ppm of CNSL concentration, 40 °C temperature, and 1000 rpm shear rate. Taguchi statistical method aided in optimizing the process variables, with concentration found to contribute the most significantly to flow improvement. The reduction in viscosity and wax precipitation temperature, along with microscopic observations confirming wax structure distortion, highlighted CNSL's efficacy as a moderate pour-point depressant, viscosity reducer, inhibitor of crystal growth, and modifying agent, CNSL demonstrated significant potential as a bio-based enhancer of flow. Its cost-effectiveness and environmental friendliness make it a promising alternative to conventional additives, effectively tackling major challenges in the transportation of heavy crude oil [28].

Elkatory et al. (2022) presented a novel approach to address the challenge of wax deposition in subsea pipelines transporting waxy petroleum crude oil (WPCO) by utilizing free fatty acids derived from waste generated during sunflower oil refining. By esterifying and copolymerizing these fatty acids with maleic anhydride, comb-shaped poly fatty esters (PFES) were synthesized and characterized. The efficacy of PFES as a pour point depressant (PPD) for WPCO was assessed, revealing a significant reduction in pour point from 24 to 3°C with a dosage of 3000 ppm. Structural analyses indicated that PFES hindered wax formation and modified the crystallization of paraffinic waxes in WPCO. This innovative approach demonstrates the potential of utilizing agro-industrial waste as a green, efficient, and cost-effective solution for improving the flow properties of WPCO, thereby enhancing its transportation and storage capabilities.

3. Materials and methods

3.1 Materials

All chemicals n-hexane, n-heptane, toluene, methanol, sulfuric acid (H_2SO_2), potassium permanganate, graphite, hydrogen peroxide (H_2O_2), nitrogen, benzotriazol-1-yloxytris(dimethylamino) phosphonium hexafluorophosphate (BOP), methyl methacrylate (MMA) was obtained from local vendors. For the current investigation the crude oil sample were provided by Oil India Limited (OIL) of India's North-Eastern oil fields. Iron Oxide and Graphene Oxide were synthesized in the laboratory using various chemicals.

3.2 Methods: Characterization for crude oil

3.2.1 Specific Gravity

Density or specific gravity was determined by utilizing pycnometer which is a fairly precise method. Pycnometers are well-defined-volume glass or metal containers used for determining the density or specific gravity of liquids or solids. A capillary tube and a tiny vial with a tight-fitting stopper are often included. Excess liquid can be let out through the capillary tube while air bubbles can't get in. The following formula can be used to calculate the specific gravity of waxy crude oil sample.

Specific Gravity = (M2 - M1) / (M3 - M1)

Given,

Weigh of the empty and dry pycnometer is 'M1'

The pycnometer containing half filled with the waxy crude sample 'M2'.

The pycnometer containing half water and half waxy crude oil sample 'M3'.

3.2.2 API Gravity:

It is employed to determine the crude oil's quality. The specific gravity values at 15.6°C were used to estimate the API gravity values for the crude oil sample. When calculating API gravity from specific gravity (S.G.) at 60 ^{0}F temperature, the following formula is used:

API Gravity = (141.5/S.G.) – 131.5

3.2.3 Pour Point:

The temperature at which a crude oil begins to gel as a result of wax crystallisation is known as the pour point. The ASTM D97-06.11 pour point test method was used to evaluate the temperatures of the crude oil samples. The pour point was assessed utilizing the ASTM pour point apparatus, comprising a test jar, bath, jacket, and thermometer. The procedure involved heating the sample to 48°C, allowing it to cool gradually, and then monitoring it every 3°C until flow ceased when the test jar was tilted horizontally.

3.2.4 Water Content:

Knowing the amount of water and sediment in crude oil is crucial since they can lead to corrosion and other issues during the refining of crude oil. The centrifuge method was used to determine the bottom sediments and water (ASTM D 96-58 T). In this approach, a centrifuge tube containing 25 ml of sample and 25 ml of toluene was completely mixed before spinning at the required speed for 10 minutes inside the centrifuge setup. Following this, two layers were form, and the amount of water was determined using the calibration at the centrifuge tube's bottom.

3.2.5 WAX Content:

Oil sample is collected in a beaker, and n-pentane is added while stirring for 30 minutes at a ratio of 1:20 (weight per unit volume). Freeze the liquid overnight after adding acetone. After that, Whatman filter paper 934 was used to filter solid organic wax in a Buchner funnel while it was under vacuum. The wax is then filtered away using hot hexane.

3.2.6 SARA Distribution:

The SARA distribution of crude oil refers to the relative composition of different hydrocarbon fractions within the oil. SARA stands for Saturates, Aromatics, Resins, and Asphaltenes. These fractions represent different types of hydrocarbons present in the crude oil and can provide insights into its properties and behaviour.

Large heterogeneous molecules with condensed aromatic centres are known as asphaltenes. They combine to produce colloidal-sized particles and have a significant impact on the wax crystallisation as well as the viscosity of the oil medium. The approach is based on the accepted test procedure ASTM D2007-93. Asphaltenes and insoluble resins are separated in the first stage using n-hexane precipitation. Asphaltenes that have precipitated are removed once the mixture is cooled. To separate the filtered sample (maltene fraction) into saturated chemicals, aromatic compounds, and polar resins, chromatographic column was used. The solid asphaltenes were dried and then weighed after being cleaned with n-heptane [29].

3.3 Synthesis of Graphene Oxide and Iron Oxide composite

The modified Hummers method is used for the preparation of graphene oxide (GO). Prepare a solution of concentrated sulfuric acid (H_2SO_2 around 100ml) by adding it to a beaker or flask. Place the container in an ice bath to keep it cool throughout the procedure. Add a known amount of graphite powder (typically around 1 gram) to the cooled sulfuric acid solution. Stir the mixture vigorously using a stirrer or a magnetic stir bar. Slowly add potassium permanganate ($KMnO_4$ around 3.0g) powder to the acid and graphite mixture while stirring continuously. The molar ratio of $KMnO_4$ to graphite can vary but is typically in the range of 2:1 to 3:1. Continue stirring the mixture for several hours (3-4 hours) at a temperature below 20°C. The ice bath should be maintained during this time to control the reaction temperature. After the desired stirring time, slowly add hydrogen peroxide (H_2O_2) solution to the mixture while maintaining stirring. This step helps in terminating the reaction and decomposing excess permanganate. Continue stirring the mixture for another 1-2 hours. The colour of the mixture will change from dark brown to yellowish-brown or light brown, indicating the formation of graphene oxide. After the reaction is complete, transfer the mixture to a centrifuge tube and centrifuge it at high speed (around 10,000 rpm) for 10-15 minutes to separate the solid precipitate. Carefully remove the supernatant liquid, which contains the by-products and unreacted chemicals, and dispose of it properly following appropriate waste disposal procedures. Wash the obtained solid precipitate with distilled water several times to remove residual acid and impurities. Centrifuge the mixture after each wash and discard the supernatant. Finally, dry the graphene oxide obtained by using an oven at a low temperature (below 100°C). The resulting graphene oxide can be further characterized and used for various applications [30].

Reduced GO was prepared by ultrasonically dispersing 1.0g GO in 300 mL of water for 1 hour in a nitrogen atmosphere. At 80°C, 10 g of ascorbic acid was added while stirring for 20 minutes. The dark material was obtained after filtering and washing with ethanol, then dried at 80°C in vacuum for 10 hours [31].

Iron oxide nanoparticles was prepared using the co-precipitation method and to prepare a composite of reduced graphene oxide (rGO) and iron oxide nanoparticles simple mixing and deposition method was used: Prepare a solution of iron salts by dissolving the desired amount of iron(II) chloride or iron(III) chloride in deionized water. The concentration of the iron salt can vary based on the desired nanoparticle size and concentration. Sonicate the reduced graphene oxide (around 500mg) dispersion for a specific period of time to exfoliate the graphene oxide sheets and enhance their dispersion (typically around 2 hour). Place the iron salt solution in a glass beaker and start stirring it using a magnetic stirrer or a stir bar. Then add the sonicated graphene oxide dispersion into the mixture. Add the base solution (ammonium hydroxide) dropwise to the iron salt solution while continuously stirring. The base will react with the iron salt, resulting in the precipitation of iron hydroxide. Continue stirring the mixture for a certain period of time (typically around 1-2 hours) to ensure complete precipitation and formation of iron hydroxide nanoparticles. After the desired stirring time, adjust the pH of the mixture to around 7 by adding acid (hydrochloric acid) dropwise. This step helps in the conversion of iron hydroxide to iron oxide nanoparticles. Stir the mixture to ensure proper mixing and dispersion of the nanoparticles within the graphene oxide matrix. Centrifuge the composite mixture to separate any unbound iron oxide nanoparticles. Adjust the centrifugation parameters (speed and time) based on the particle size and density. Carefully remove the supernatant liquid after centrifugation, leaving behind the composite material at the bottom of the container. Wash the composite material with deionized water to remove any residual salts or impurities. Centrifugation can be employed to separate the composite from the washing solution. Finally, dry the composite material by using an oven at a low temperature (below 100°C). The resulting graphene oxide-iron oxide composite is produced and ready for further use [32].

3.4 Synthesis of poly(methyl methacrylate)-(graphene-oxide)-(iron-oxide) (PMMA-GO-Fe3O4) nano-composite

Varying weights of Graphene Oxide and Iron Oxide composite were added under sonication to a 100 ml round flask containing dry toluene (15 ml) and maintained for 30 min at 5 °C under a nitrogen atmosphere. Subsequently, calculated weights of methyl methacrylate (MMA) diluted in toluene (40 wt %) were added to the dispersed the nanocomposite mixture, corresponding to different concentrations of 0.1%, 0.3%, 0.5%, and 1.0% relative to PMMA. The resulting mixture was sonicated for an additional 5 min at 5 °C under a nitrogen flow. At 75 °C, benzotriazol-1-yloxytris(dimethylamino)-phosphonium hexafluorophosphate (BOP) dissolved in toluene was injected under stirring and maintained for 15 min, followed by a temperature reduction to 56 °C for an additional 5 h to initiate MMA polymerization. Next, the reaction mixture was mixed with methanol to cause the precipitation of the PMMA-GO-Fe_3O_4 nanohybrid products. These products were then dissolved in toluene, poured into petri dishes, and dried at 60 °C to produce PMMA-GO- Fe_3O_4 films. The resulting polymer nanohybrids showed different nanocomposite contents, ranging from 89% to 78%, corresponding to PMMA-(0.1%, 0.3%, 0.5%, and 1%) GO- Fe_3O_4, respectively.

4 Result and discussion

4.1 Characterization of waxy Crude Oil

Table 1 Physico-Chemical properties of crude oil sample

S. No	Parameters	Observed
1	Specific Gravity (15°C)	0.90411 gm/ml^2
2	API Gravity	25.21
3	Pour Point (°C)	30^{o}C
4	Water Content (%)	0.7%
5	WAX (°C)	7.8
6	Saturates (%)	65.66
7	Aromatics (%)	20.4
8	Resins (%)	11.12

Table 1 shows characterization results of the crude oil sample [33]. Specific gravity is a measure of the density of a substance relative to the density of water. The specific gravity of crude oil can vary significantly depending on its composition and the type of oil. It is typically expressed as a dimensionless value. The American Petroleum Institute (API) gravity is a measurement used to determine the density of petroleum liquids, including crude oil. It is an inverse measure of the density, meaning that higher API gravity values indicate lighter and less dense oils, while lower API gravity values indicate heavier and more dense oils. The pour point of crude oil refers to the lowest temperature at which the oil will flow under specific conditions.

It is an important property to consider as it indicates the oil's ability to flow and be pumped at low temperatures. Crude oil typically contains some amount of water, which can vary depending on factors such as the oil's source, production methods, and storage conditions. The water content in crude oil is generally expressed as a percentage by volume or as a ratio of water to oil. Wax is more commonly found in heavier crude oils, particularly those with a high paraffin content. SARA analysis involves fractionating crude oil based on the solubility characteristics of these different fractions. The analysis provides valuable information about the composition, properties, and behavior of the crude oil, which is crucial for refining, processing, and product formulation.

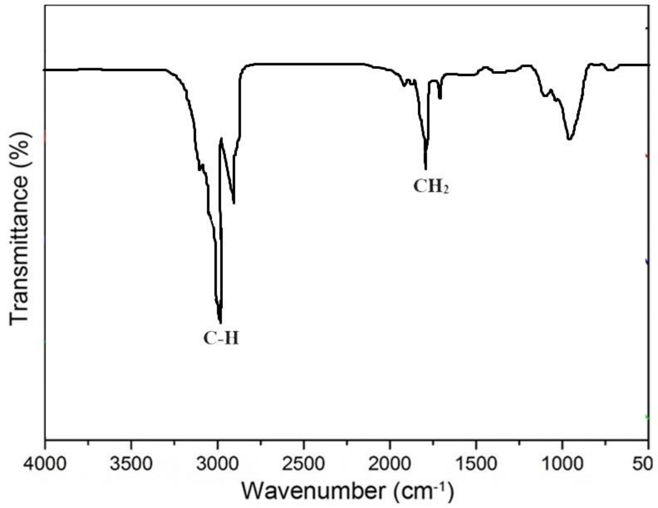

Figure 1 FTIR analysis of crude oil sample

FTIR (Fourier Transform Infrared) analysis is a spectroscopic technique used to detect and examine the chemical composition of a sample through its infrared absorption. and transmission properties. It provides information about the functional groups and molecular bonds present in the sample [34]. In figure 1, the wavenumber 2953.25 cm^{-1} is commonly associated with the elongation vibration of the C-H (carbon-hydrogen) bonds in aliphatic (saturated) hydrocarbons. The FTIR band at 2918.76 cm^{-1} and 2850.08 cm^{-1} is also associated with the stretching vibration of the C-H (carbon-hydrogen) bonds in hydrocarbon compounds. This particular band is often attributed to the symmetric stretching of the methylene groups in alkanes. The band at 1460.09 cm^{-1} is typically associated with the scissoring or bending vibration of the CH_2 groups, whereas the band at 1376.88 cm-1 corresponds to the rocking or wagging vibration of the CH_2 groups. The band observed at 634.28 cm-1 might indicate the bending vibration of the C-H bonds in aromatic compounds like aromatic hydrocarbons or polycyclic aromatic hydrocarbons (PAHs). Aromatic compounds are commonly found in crude oil, and this band could indicate their presence. The band at 604.94 cm^{-1} could be associated with the bending vibration of C-H bonds in alkenes or alkynes. These functional groups may be present in waxy crude oil, which can contain unsaturated hydrocarbons.

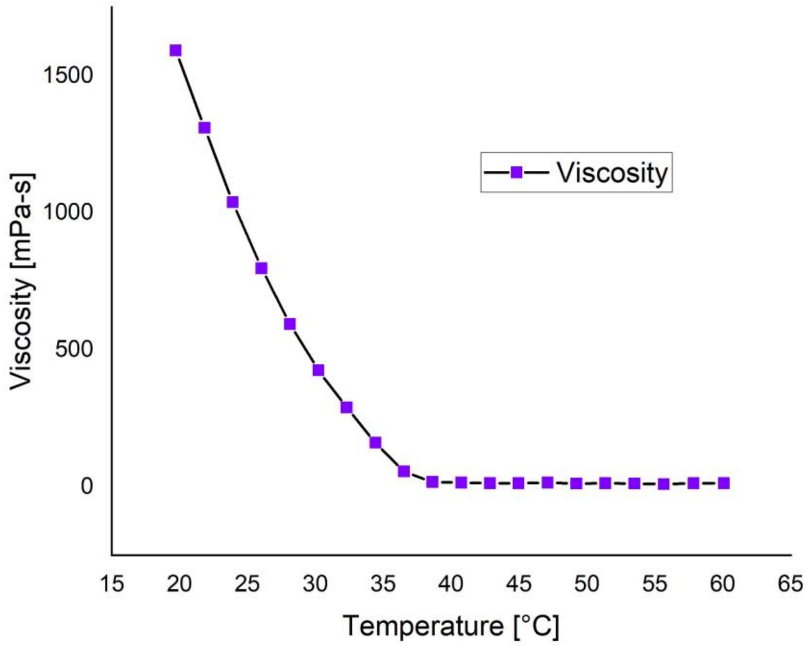

Figure 2 Viscosity vs Temperature for raw crude oil

In the graph (Figure 2), as temperature increases from left to right, the viscosity of crude oil decreases. The rate of viscosity decrease may vary at different temperature ranges. At lower temperatures, the viscosity tends to decrease more slowly, while at higher temperatures, the viscosity reduction becomes more significant [35].

4.2 Dispersion stability of rGO-Fe_3O_4 in water and xylene

Figure 3 Dispersion of additive in water and xylene

To test their dispersion stability in polar and nonpolar solvents, the same-quality rGO-Fe_3O_4 composites were introduced to water and xylene. The results are presented in Fig 3. The rGO-Fe_3O_4 composite material was not soluble in water, demonstrating its extraordinarily potent hydrophobicity. While the composite material was soluble in xylene and there was no precipitation when it was dissolved in xylene.

4.3 SEM Image of the composite material

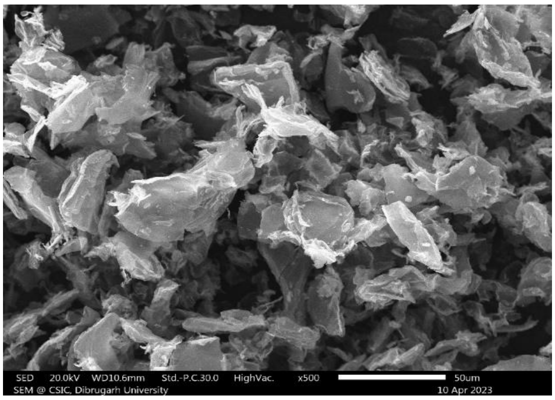

Figure 4 SEM image of rGO and Fe_3O_4 composite

In the SEM image (figure 4), there is a mixture of reduced graphene oxide and iron oxide. The graphene oxide sheets may serve as matrix or support for the iron oxide particles, leading to a heterogeneous distribution across the sample surface. The iron oxide particles can be observed attached to or dispersed on the graphene sheets. The SEM image reveal the surface roughness and contours of the composite material. The graphene sheets exhibit folds, wrinkles, or overlapping layers, while the iron oxide particles contribute to variations in height and surface texture [36].

4.4 Evaluation of Pour-Point Depressants for viscosity reduction

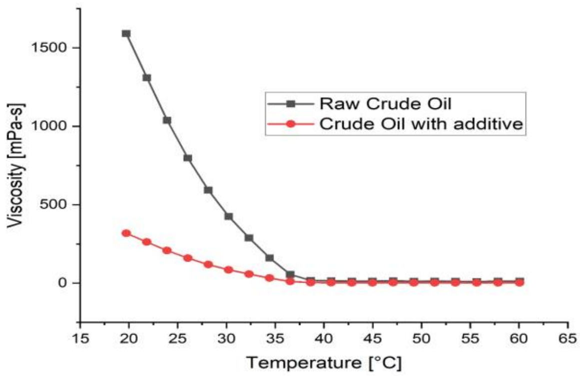

Figure 5 Viscosity vs Temperature of crude oil with and without additive

In 10 mL of xylene the flow improver i.e., rGO-Fe_3O_4 with mass of 0.005 g was dissolved, then ultrasonic dispersion was performed for 10 min. Subsequently, 10 grams of heavy oil were mixed with 1 milliliter of the solution under a constant temperature of 65°C for 1 hour. The selection of 65°C was based on the necessity for complete melting of paraffin, as a pour-point depressant is effective only under such conditions [37]. The pour points and viscosities were measured and calculated based on the rates of pour-point depression and viscosity reduction provided by the pour-point depressant. Before the additive was added, the heavy oil's tested pour-point temperature was 30°C. At a shear rate of 50 s-1, the viscosity was 4,578 mPa s with temperature 20°C for the raw crude. After the addition of the flow improver, it can be seen that the viscosity curve decreases to a certain extent. At a shear rate of 50 s-1, the viscosity was 3,319 mPa s with temperature 20°C for the crude after the addition of pour point depressant.

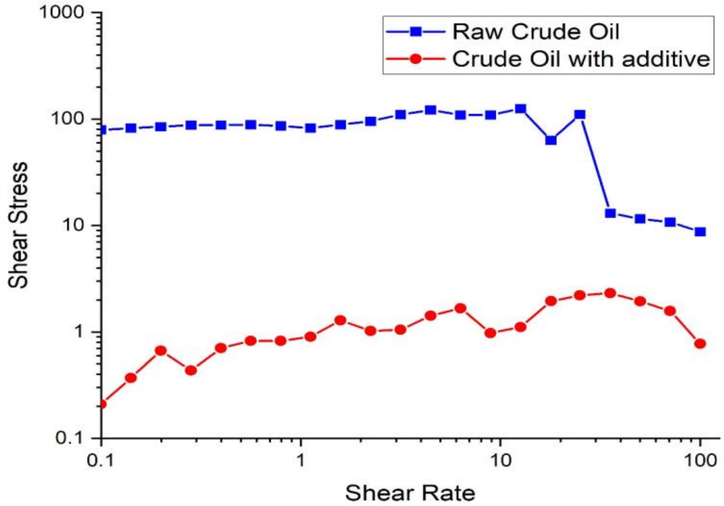

Figure 6 Shear stress vs Shear rate of crude oil with and without additive

Here, figure 6 depicts the change in shear stress as a function of the shear rate, which ranges from 0.1 to 100 s-1. The various coloured lines denote various constant temperature curves, which demonstrate that shear stress typically increases with shear rate while decreasing with temperature increase for each curve. This is probably because thermal motion weakens the network between particles.

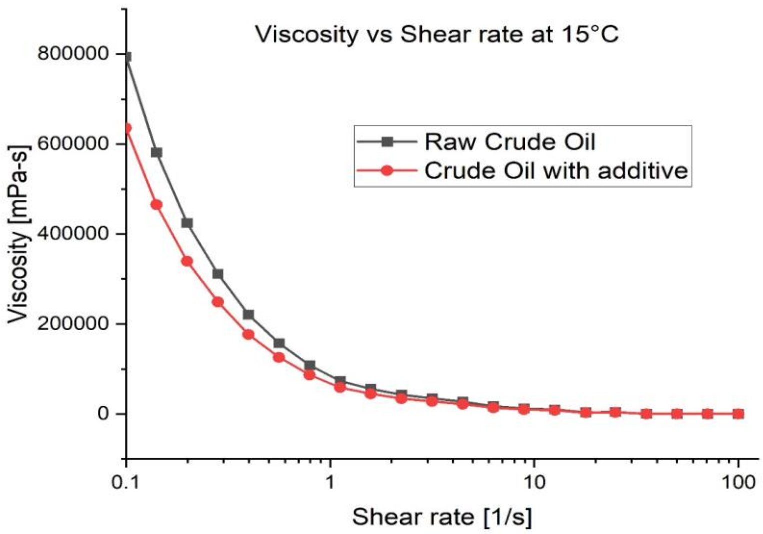

Figure 7 Viscosity vs Shear Rate of Crude Oil with and without additive at 15°C

Following the experimental work done at various shearing rates in sec-1, the viscosity of both the raw crude sample and the crude with additive was initially detected to be 66700 and 43700 mPa-s at a shearing rate of 0.2 sec-1, according to the plotted profile (Figure 7). However, as it can be seen, there is a declining trend of viscosity for crude oil with additive. Additionally, at a high shearing rate of 100 sec-1, the viscosity measurements were recorded as 5178 and 245 mPa-s, indicating that the samples viscosity decreased solely as a result of the additions. By itself, nanocomposite PPD substantially lowers the value of viscosity for the crude sample.

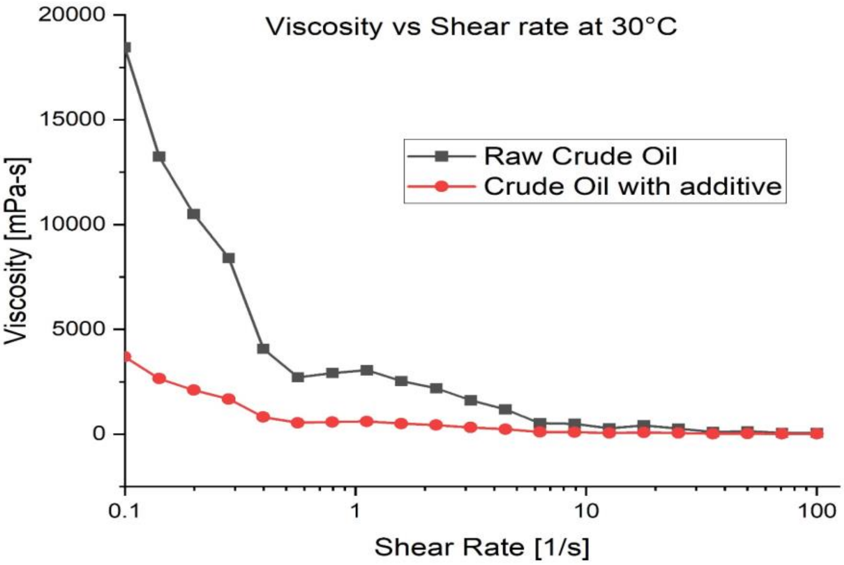

Figure 8 Viscosity vs Shear Rate of Crude Oil with and without additive at 30°C

Figure 8 illustrates the representation of viscosity vs shear rate graph at 30°C and against a shear rate of 0.5 s-1; viscosities of 43700 and 8340 have been observed for raw crude and crude with additive; this data clearly demonstrates a decreasing rate trend with the application of additive.

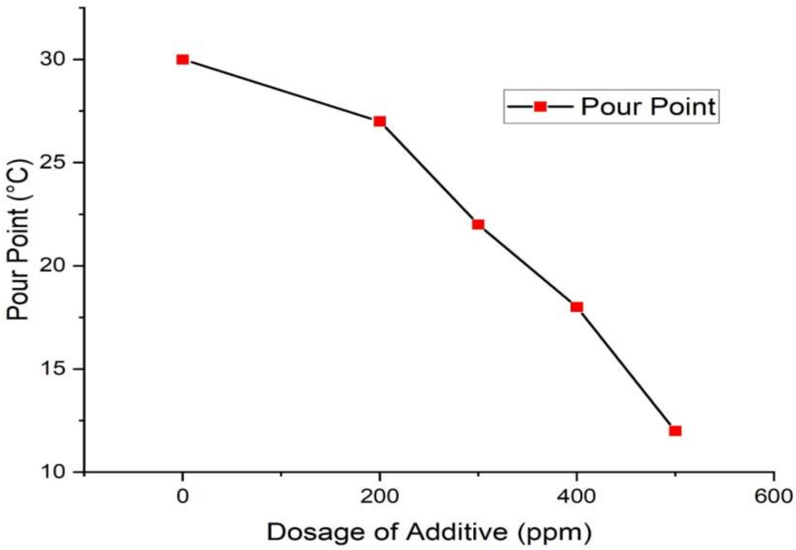

Figure 9 Pour Point with respect to different dosage of PPD

The viscosity of crude oil can be decreased by adding a small amount of the chemical pour point depressant. The pour point value in the crude oil sample was successfully decreased from 30°C to 27°C at a dosage of 500 ppm (fig.9). These events are possible as a result of the nano-particles ability to absorb the wax crystal content of crude oil and prevent the formation of deposits of hardened wax that would otherwise obstruct crude oil movement.

5 Evaluation Storage Modules and Loss Modules

The viscoelastic behaviour of materials is described by the storage modulus and loss modulus. The storage modulus (G') reflects a material's elastic or solid-like reaction. It measures how much energy can be held in a material when stress or strain is applied. A stiffer or more elastic material with better deformation resistance and better shape recovery is one with a greater storage modulus. The loss modulus (G'') describes a material's viscous or liquid-like reaction. It measures the amount of energy lost during deformation as heat. A more viscous or energy-dissipating material with a higher loss modulus may experience more internal friction and heat loss.

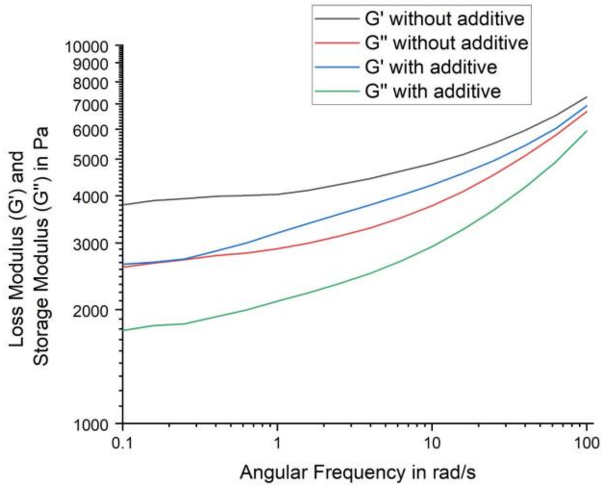

Figure 10 Storage and Loss Modules vs Angular Frequency

Figure 10 illustrates the alterations in viscoelastic characteristics concerning angular frequency variations, comparing crude oil with and without additives. At a low frequency of 0.1 (rad/s), the raw crude exhibits a storage modulus of approximately 3785 (Pa) and a loss modulus of about 2590 (Pa). As the angular frequency escalates, both moduli increase. For instance, at 100 (rad/s), the storage modulus reaches 6921 (Pa) and the loss modulus hits 5935 (Pa). This disparity between storage and loss moduli remains consistent across varying temperatures for waxy crude oil, suggesting the dominance of elasticity over viscosity, thus characterizing the crude as a viscoelastic solid. Upon treatment with additives, the moduli values decline to varying extents. For instance, the storage modulus reduces to 2638 (Pa) and the loss modulus to 1760 (Pa), with similar trends observed at higher frequencies [38] [39].

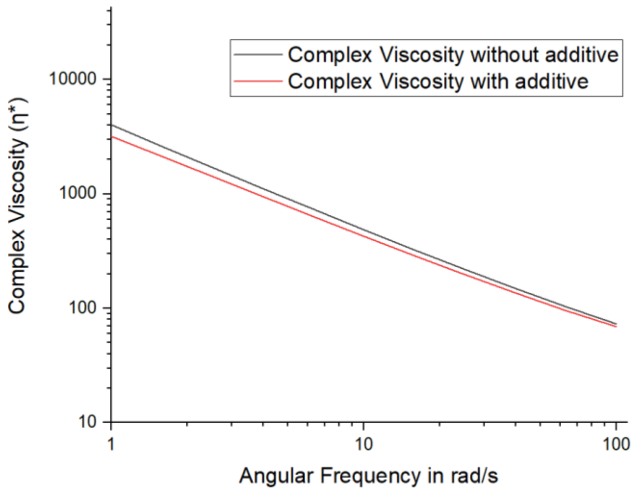

Figure 11 Complex Viscosity (η*) with and without additive

The complex viscosity (η*) was calculated using the formula:
η* = Storage Modulus (G') / Angular Frequency (ω)

At a low angular frequency of 0.1 (rad/s), the raw crude oil sample exhibits a complex viscosity of approximately 37853 (Pa), and with the increase in angular frequency, the complex viscosity diminishes. At 100 (rad/s), the complex viscosity is 72 (Pa) for raw crude oil. The complex viscosity is around 26386 (Pa) for the crude oil with additive at 0.1 rad/s and as the angular frequency increases the complex viscosity also decrease. At 100 (rad/s), the complex viscosity around 69 (Pa). It can be seen that the difference between the values of complex viscosity decreases as the frequency increases.

6 Conclusion:

In this study, the author has synthesised a composite based in graphene oxide and iron oxide to test its efficiency to enhance the viscosity of heavy waxy crude oil from North East India. To create a solution, the nanocomposite material that was created was dissolved in the organic solvent xylene. Based on the test outcomes, it appears that the nanocomposite material could potentially lower the viscosity of heavy oil by more than 40% regardless of its pour point. Additionally, the outcomes demonstrated that it can lower their pour points to some extent, demonstrating a positive application effect. Upon treatment of the crude oil sample with the additives (the nanocomposite PPD), there is a reduction in both storage and loss moduli across various frequencies. The storage modulus declines to 2638 Pa, while the loss modulus decreases to 1760 Pa. At higher frequencies, the values increase slightly, with the storage modulus becoming 6921 Pa and the loss modulus reaching 5935 Pa. Comparing the complex viscosities of the untreated crude oil and the crude oil with the additive reveals that incorporating the nanocomposite PPD results in a reduction in complex viscosity at both low and high angular frequencies. These results indicate that the incorporation of the nanocomposite pour point depressant leads to changes in the viscoelastic properties of the crude oil, resulting in lower storage and loss moduli compared to the raw crude. The additives seem to have an effect on the material's elasticity and viscosity, potentially contributing to the improvement in flow properties observed in the previous experiments. Considering the gradual decline in traditional oil and gas reservoirs, the composite material holds considerable importance for the advancement of unconventional oil and gas sources, like heavy waxy oil.

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Conflicts of interest

The authors have no conflicts of interest to declare.

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