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DEVELOPMENT OF POUR POINT DEPRESSANT USING AFRICAN PEAR SEED OIL (*DACRYODES EDULIS*) AND DESERT DATE SEED OIL (*BALANITES AEGYPTIACA*) AS GREEN ALTERNATIVES

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Abstract

This study investigates the potential development of biodegradable pour point depressants (PPDs) using natural oils from African pear seeds (Dacryodes edulis) and desert date seeds (Balanites aegyptiaca) as eco-friendly options to improve the flow properties of waxy crude oils. Nigerian crude oil is known for being high quality, but it often encounters serious flow assurance issues due to wax buildup, especially in cold weather. Current chemical inhibitors are effective but come with high operational costs and environmental risks. This research investigates the possibility of modifying these natural oils, which are readily available and usually considered waste, to create green-effective PPDs. The study builds on prior evidence that these seeds have a high oil content rich in fatty acids like oleic, linoleic, palmitic, and stearic acids, which may work well as wax inhibitors. By using n-hexane as a solvent in the extraction and modification process, this work aims to increase the economic value of these underused crops, encourage their cultivation, and widen their use in the oil industry. The findings of this study are expected to offer a sustainable and effective solution to reduce wax buildup, ultimately enhancing the efficiency of crude oil transport while addressing environmental issues linked to traditional additives.

Keywords: *Pour point depressants, Wax deposition, Renewable resources, Flow Assurance Natural Additives.*

Introduction

Pour point depressants (PPDs) are essential for improving the flow of mineral oils, especially those from petroleum, at lower temperatures. These additives, usually made of polymers, work well in oils with waxy or paraffinic bases. The pour point is the lowest temperature at which an oil can flow under gravity, which is measured in laboratory tests. By weakening the initial gel structure and improving wax crystal formation, pour point depressants help tackle important challenges related to the transportation and production of crude oil.

Nigeria has a unique kind of crude oil distinguished by its higher API gravity and quality, especially its low sulfur content. However, like many other paraffinic crude oils, Nigerian crudes are characterized by paraffinic moderate and even high wax content [1]. The buildup of wax during transportation and production is a major flow assurance issue for the oil industry [2]. These problems worsen in cold weather, where paraffin formation can block pipelines and production lines, reducing efficiency.

Transporting crude oil effectively is vital. It must be moved from extraction sites through different processing stages to refineries and storage facilities while keeping its flow properties intact. Throughout this process, crude oil undergoes various physical and chemical changes that can cause flow assurance issues, including wax and paraffin buildup. Using chemical additives like PPDs is crucial to ensure crude oil can be transported efficiently by lowering the Wax Deposition Temperature (WAT) i.e. the

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temperature where wax crystals first form during cooling [3, 4]. As the temperature drops, crude oil thickens, leading to a situation where it cannot flow or be pumped once it falls below its pour point temperature [5].

Pour point temperature control is a very important aspect in maintaining crude oil flow properties. Typically, the removal of wax in the pipeline can be carried out by heating the wax, blending it with lighter oils, heating with steam, and employing mechanical removal methods [6]. But the use of chemical additives has prevailed over the other methods in recent times because of their convenience and lower costs, especially as flow improvers (FIs) and pour point depressants (PPDs) [7]. The presence of wax in the oil can not only increase the oil viscosity but can also cause the oil to form a gel that will have flow characteristics that are unfavorable [8]. While PPDs and flow improvers can improve flow properties and reduce expensive remediation needs, the chemical inhibitors currently in use often come with high operational costs and environmental risks if spills happen, due to their non-biodegradable nature.

Wax deposition continues to be a significant challenge in the oil industry, affecting productivity and operational costs [9, 10]. While traditional methods, like increasing the temperature of the wax, can help reduce fluid viscosity, the chemicals used as additives such as xylene, triethanolamine, and toluene, usually bring their own challenges [6, 11]. Exploring biodegradable and green-effective alternatives offers a promising solution for managing wax deposition while addressing environmental concerns linked to current chemical inhibitors. This study seeks to investigate the potential of modifying natural African pear seed oil (*Dacryodes edulis*) and desert date seed oil (*Balanites aegyptiaca*) as green, biodegradable additives to develop PPDs for waxy crude oils.

Materials and Methods

For this study, we used various materials, including natural African pear seed oil (*Dacryodes edulis*) and Desert date seed oil (*Balanites aegyptiaca*) as potential low-cost pour point depressants (PPDs) for waxy crude oil. We chose these oils due to their availability and reported oil content [12, 13, 14. 15]. The non-polar solvent n-hexane was used to modify the natural oils for the PPD formulation.

Preparation of Pour Point Depressants

The oils from the seeds of African pear and Desert date were extracted using hexane as a solvent to get the best results. The procedure included the following steps:

i. Seed Preparation: Seeds of *Dacryodes edulis* and *Balanites aegyptiaca* were collected and cleaned thoroughly to remove any foreign materials.

ii. Oil Extraction: The cleaned seeds were air-dried and ground into a fine powder.

About 250 grams of the seed powder were then mixed with 100 mL of n-hexane in a Soxhlet extractor. The extraction process, shown in Fig. 1, took 6 to 8 hours to ensure complete oil extraction. The resulting oil was collected, and the n-hexane was evaporated under reduced pressure to obtain pure oil.

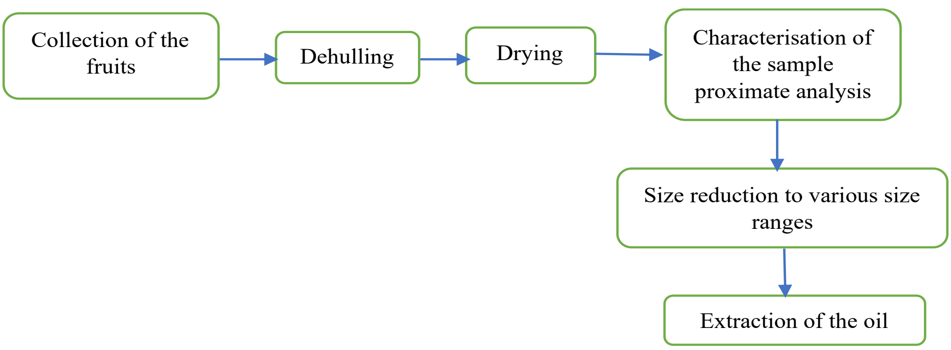

Fig. 1. Flowchart for Extraction Process of Oils from Seeds

Characterization of Oils

The composition of fatty acids and other substances in the extracted oils was analysed using Gas Chromatography-Mass Spectrometry (GC-MS) (Table 1 and Table 2). The samples were treated with a BF_3-methanol solution to convert them into fatty acid methyl esters (FAMEs) before analysis. The following parameters were evaluated:

i. Oil Yield: This was determined by comparing the mass of the oil obtained to the mass of the seed used.
ii. Fatty Acid Profile: This involved measuring important fatty acids, including oleic, linoleic, palmitic, and stearic acids.

Table 1. Substance composition of African pear seed

Substance	Percentage (%)
Oleic	18-60
Lineoleic	15-29
Palmitic	30-62
Stearic	1.3-5.5
Moisture	37-54
Crude protein	12-20
Fats	19-38
Fibres	18

Table 2. Substance composition of Desert date seed

Substance	Value
Colour	Pale yellow
Odour	Pleasant
Density (g/cm^3)	0.8
Specific gravity	0.92
Saponification value (%)	173
Peroxide value	9.4
Acid value	0.93
Free fatty acid	0.47
Moisture content	9.8
Oil content	45.2
Texture	Viscous

Pour Point Depression Testing

The pour point of waxy crude oil was determined using the ASTM D97-12 method. This method identifies the lowest temperature at which the sample can flow. The pour point was recorded as 30°C above the temperature where the sample stops flowing. The wax content significantly affects this temperature. Waxes can form a crystalline structure, leading to solidification when the sample cools. This creates challenges in pipelines and manifolds during production shutdowns. Pour point values are expressed as multiples of 30°C. To find the pour point, we add 30°C to the observed stopping temperature.

The procedure starts with pouring the sample into a test jar and sealing it with a cork. A thermometer was inserted without shaking the sample. Next, the pour point machine was activated, allowing the sample to cool. After this, the assembly was placed in the pour point depressant analysis machine as shown in Fig. 2. Regular checks were conducted to see if the sample pours. When it stops flowing upon tilting, the sample was tilted horizontally for five seconds. If it still does not flow, the temperature is recorded.

The performance of the developed PPDs was evaluated by looking at their effect on the pour point of a waxy crude oil sample. The crude oil was mixed with different concentrations of the modified oils and tested under controlled cooling conditions.

The effectiveness of the PPD was determined using the Pour Point Reduction (PPR) equation (Eq. 1) to assess the performance of the pour point depressants:

$$\text{Pour Point Reduction (PPR)} = P_{oil} - P_{blended} \quad (1)$$

where: P_{oil} = Initial pour point of the crude oil (°C); $P_{blended}$ = Pour point of the crude oil blended with PPD (°C)

Fig. 2. Pour point depressant analysis (machine)

Determination of Wax content of the crude oil sample

The process of determining wax content in crude oil included several steps. First, the sieve was washed with xylene and dried in an oven for about one hour. Its initial weight was recorded afterward. Next, 100 ml of the wax crude sample was measured and weighed in a measuring cylinder. The sample was poured into the sieve, and acetone was washed over it while heating on a mechanical mesh. This allowed the crude oil to pass through the 45-micron sieve, leaving the wax behind. After drying the wax at room temperature for about 20 minutes, the final weight of the sieve was recorded. The wax content was calculated using Eq. 2.

$$\%\ \text{wax} = \frac{W_2 - W_1}{\text{Vol. of sample}} \quad (2)$$

Fig. 3. Wax content determination Apparatus

The Wax Deposition Temperature (WDT) was measured using a cooling bath and visual observation method, as shown in Fig. 3. A sample of the waxy crude oil was cooled in a glass container at a controlled rate, and the formation of wax crystals was observed and recorded.

Determination of the Crude Oil Kinematic and Dynamic Viscosities

The determination of crude kinematic viscosity was done using a U-tube viscometer (Fig. 4).

The process involved pouring the crude oil into the reservoir arm until it reached meniscus A. The sample was drawn through the working arm above meniscus C and then sealed with a cork. The timer started when the crude oil reached meniscus C and stopped when it flowed to meniscus B, recording the elapsed time. Subsequently, the kinematic viscosity was calculated using Eq. 3:

$$V = CT \quad (3)$$

where: V = kinematic viscosity; C = calibration coefficient; T = time taken to flow from C to B.

Also, the dynamic viscosity was determined using the equation:

$$\underset{\sim}{U} = \rho V \quad (4)$$

where: $\underset{\sim}{U}$ = dynamic viscosity; ρ = density of crude oil.

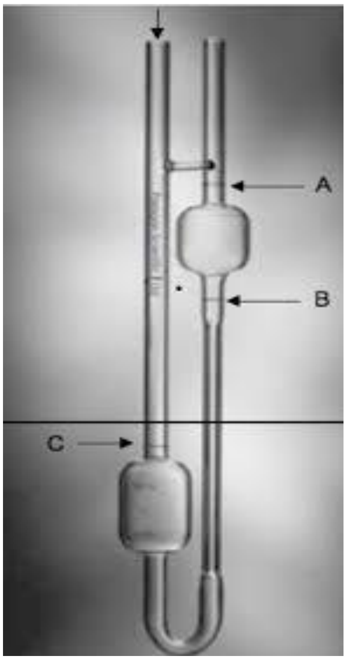

Fig. 4. Schematic of U-tube viscometer

Determination of Crude oil specific gravity and API gravity

The specific gravity and API gravity of the crude oil was using the density bottle method. The process began by measuring the crude oil's temperature. Next, an empty density bottle is weighed, then it is weighed again after filling with the crude oil. This procedure was repeated with fresh water to obtain a second weight. These measurements allowed us to calculate the relative density using the following equation.

$$\text{Relative density} = \frac{mass\ of\ Oil}{mass\ of\ fresh\ water} \quad (5)$$

Statistical Analysis

All viscosity measurements were done in triplicate to ensure reliable results. Results are shown as Mean ± Standard Deviation (SD), calculated using Equation 6:

$$\sigma = \sqrt{\frac{\sum(x_i-\bar{x})^2}{n-1}} \quad (6)$$

Where x_i is each measurement, $\bar{x}$ is the mean, and n is the number of samples (3). The error bars represent one standard deviation from the mean. They show the accuracy of the measurements and the consistency of the polymer samples. Differences between samples were deemed significant when the error bars did not overlap, indicating a strong level of confidence in the observed viscosity trend. Also, statistical significance was determined using a one-way ANOVA, with $p < 0.05$ considered significant.

Results and Discussion

Analysis of the Crude Oil Samples' Viscosity

The viscosities of the crude oil samples were examined. The results showed significant differences due to the addition of pour point depressants. Table 3 illustrates that the undoped wax crude oil had the highest kinematic and dynamic viscosities, measured at 8.97 mm²/s and 7.34 cp, respectively.

Table 3. Sample Viscosities

Parameters	Undoped wax crude oil	APSO	DDSO	EVA
Kinematic Viscosity (mm^2/s)	8.97	8.19	6.25	4.43
Dynamic Viscosity (mPa.s)	7.34	6.64	5.03	3.55

A significant drop in viscosity occurred with the addition of the additives. APSO (African Pear Seed Oil) and DDSO (Desert Date Seed Oil) further lowered the viscosity. EVA (Ethylene Vinyl Acetate), known as a standard pour point depressant, performed better than the others and showed the largest reduction in viscosity. This pattern shows that wax presence greatly influences the viscosity of crude oil, as shown in the graphic in Fig. 5. The viscosity measurements revealed a clear difference among the four crude oil samples and between Kinematic Viscosity (KV) and Dynamic Viscosity (DV). KV was consistently higher than DV, which aligns with the relationship to density (KV = DV/density). The analysis with 0.5 standard deviation error bars confirmed that this difference is statistically significant.

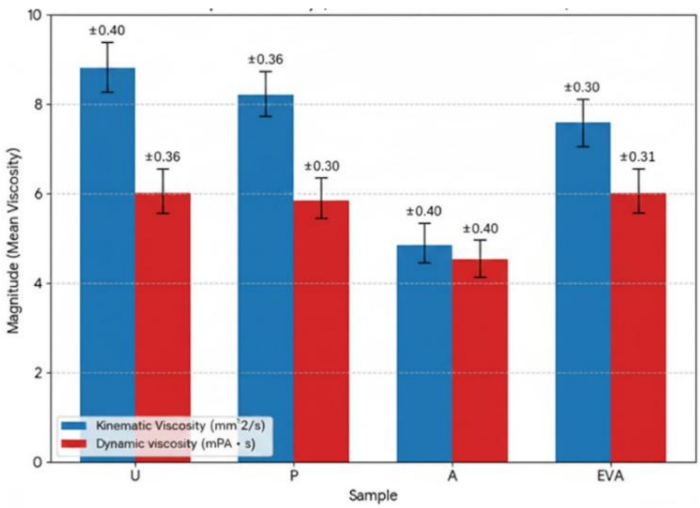

Fig. 5. The graph of the crude oil samples' Kinematic and Dynamic Viscosities

In Fig. 5, the samples showed a clear trend in mean viscosity: U > P > A > EVA. Sample U had the highest values, around 9.0 units for KV and 7.5 units for DV. In contrast, Sample EVA had the lowest, with approximately 4.5 units for KV and 3.5 units for DV. The small overlap in error bars between adjacent samples indicates that the viscosity differences are significant. This confirms the viscosity ranking and suggests that changes in chemical composition or physical state impact flow characteristics. The error analysis offers important insight into material stability. For Sample U, the mean KV of 9.0 ±

0.40 mm²/s shows the highest internal friction. The relatively tight error bars and low standard deviation across all samples indicate that the mixing process for the EVA and Additive A blends was very consistent.

The most significant observation is that KV is always higher than DV in all four samples. Since $v = \frac{\eta}{\rho}$, indicating that all tested samples have a density (ρ) of less than 1.0 g/cm³, approximately 0.83 to 0.85 g/cm³. This aligns with typical polyolefin-based materials or light synthetic oils.

Analysis of the Samples' Specific Gravity and API Gravity

The effect of wax on the specific gravity and API gravity of the crude oil samples was also examined. The results are summarized in Table 4.

Table 4. Samples Specific Gravity and API Gravity

Parameter	U	P	A	EVA
Weight of Empty Density Bottle (g)	21.5	21.5	21.5	21.5
Weight of Density Bottle + Oil (g)	44.7	44.5	44.3	44.2
Weight of Density Bottle + Water (g)	50.2	50.2	50.2	50.2
Oil only (g)	23.2	23.0	22.8	22.7
Water only (g)	28.7	28.7	28.7	28.7
Relative Density of Samples (g/ml)	0.808	0.801	0.794	0.791
Specific Gravity of Samples	0.818	0.811	0.804	0.801
API Gravity of Samples (^{0}API)	41.41	42.89	44.4	45.17

As shown in Table 4, the undoped wax crude oil had specific and API gravities of 0.818 and 41.41, respectively. The trend in Fig. 6 shows that all additives positively affected the properties of the waxy crude oil. They have the potential to improve production. The variability in measurements indicated by the error bars, along with the changes after additive treatments, is evident.

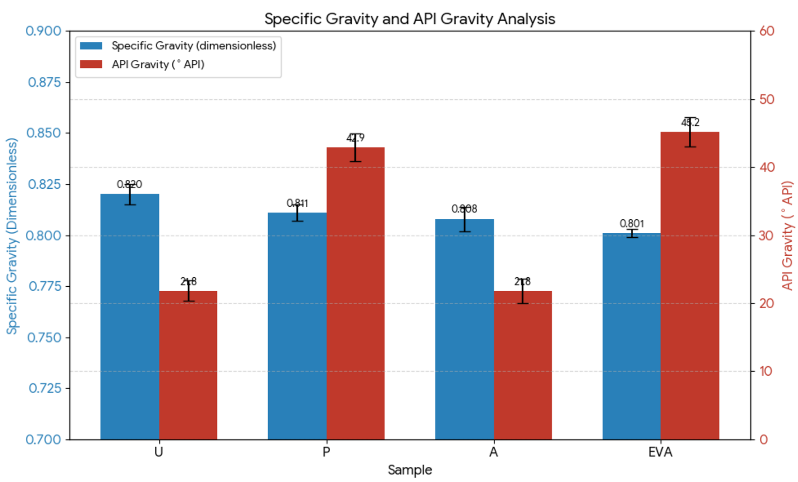

Fig. 6 Chart of the sample's specific gravity and API gravity

Fig. 6 shows that API gravity measurements (red bars) vary much more than specific gravity measurements among all samples. This means API gravity is less precise and more affected by sample differences, especially for untreated crude (U) and DDSO-treated crude (A). In comparison, the error bars for specific gravity (blue bars), which show one standard deviation from three trials, are almost invisible for APSO-treated (P) and EVA-treated samples. This indicates that specific gravity is very consistent, even when the composition changes. The base sample (U) had an average API gravity of 21.8° and a specific gravity of 0.820. Adding the APSO additive to sample P resulted in a significant change ($p < 0.05$) to 42.89° API, turning the material from medium-weight to light-weight. The specific gravity of the EVA sample dropped slightly to 0.801, but this small change, along with the low variability, suggests a consistent lightening effect and less intermolecular packing. Overall, the higher API gravity and lower specific gravity indicate that the chemical changes improved the material's fluid properties.

Analysis of the Crude Oil Samples' Wax Content

The study measured the wax content of the crude oil samples to determine how well the reactants could lower wax levels. This result is shown in Table 5.

Table 5. Results of wax content and pour point depressants

	Wax Content (%)			Pour Point (^{0}C)		
	P	A	EVA	P	A	EVA
Initial	8.66	8.66	8.66	12	12	12
1 ml	2.16	1.04	0.06	9	6	-9
2 ml	2.23	1.21	0.16	9	6	-18
3 ml	2.99	1.55	0.20	12	9	-21

The initial measurement recorded a wax content of 8.66% in the undoped crude oil. After adding 1 ml of APSO, the wax content fell to 2.16%. Adding more DDSO and EVA reduced the wax levels further to 1.04% and 0.06%, respectively. However, when the dosage increased to 2 ml, the trend reversed. The wax contents then rose to 2.23%, 1.21%, and 0.16% for APSO, DDSO, and EVA, respectively. Increasing the dosage to 3 ml again resulted in higher wax content. These results indicate that the best dosage for effective dewaxing is 1 ml, as shown in Fig. 7.

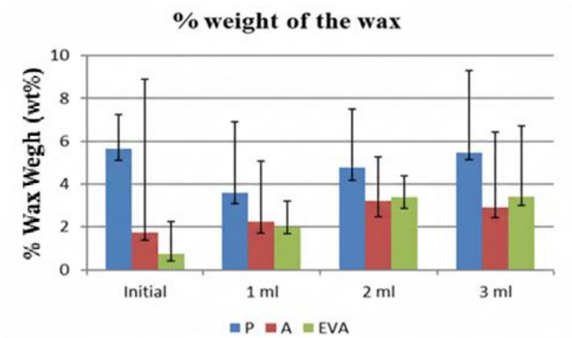

Fig. 7. Chart of the sample's wax content

Results in Fig. 7 show how increasing the additive concentration affects the residual wax content in three different chemical treatments (P, A, and EVA, respectively). The untreated sample (initial) had the highest baseline variability. The P additive showed an initial mean wax content of about 5.6%, with a significant standard deviation. Using the additives usually lowered the mean wax content, with the 1ml concentration being the most effective for all three chemicals. Specifically, the 1ml concentration of P led to a clear mean reduction to 3.5%, while A and EVA reached mean values of 2.2% and 2.0%, respectively, even though their initial baselines were much lower. A key statistical point is the absence of a dose-related improvement. Increasing the concentration from 1ml to 3ml did not further reduce the wax content. Additionally, at the optimal 1ml concentration, the performance difference between A (2.2%) and EVA (2.0%) is likely not statistically significant because of the broad measurement variability, as shown by the overlapping error bars. These findings suggest that the performance of the additives does not increase linearly with dosage above 1ml and highlight the similar effectiveness of A and EVA at lower concentrations.

Analysis of the Crude Oil Samples Pour Point

The pour point characteristics of the crude oil samples were tested. The results showed improvements when pour point depressants were added, as shown in Fig. 8. The initial pour point of the undoped wax crude oil was measured at 12°C. After adding 1 ml of each pour point depressant, the values dropped significantly to 9°C for APSO, 6°C for DDSO, and -9°C for EVA. Increasing the amount to 2 ml resulted in pour points of 9°C, 6°C, and -18°C, respectively. Adding 3 ml caused the pour points to rise again to 12°C for APSO and 6°C for DDSO, while EVA reached 21°C.

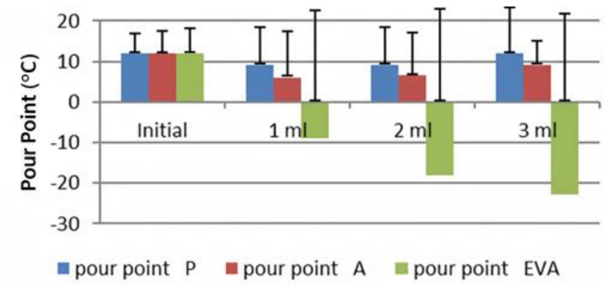

Fig. 8. The graph of the samples' Pour point

The results in Fig. 8 show how effective pour point depressants (PPD) are with three different additives: P, A, and EVA. The untreated oil had a high initial pour point of about 12.5. All early measurements were quite similar. Additives P and A had only slight to moderate PPD activity. Additive P had the least effect, keeping the pour point around 10 at all concentrations. In contrast, the EVA additive performed the best, with a clear effect based on concentration. The average pour point dropped from the baseline to about -8.0 at 1 ml, -18.0 at 2 ml, and hit its lowest point at 3 ml, around -22.5. Statistical analysis shows this drop of over 35 units with EVA is significant compared to the untreated oil and the other additives. However, the higher standard deviation in the EVA measurements at larger concentrations shows that while EVA is the most potent PPD, its performance exhibits greater variability or lower precision at its optimal dosage.

Conclusions

In this study, the evaluation of natural pour point depressants (PPDs) from African pear seed oil (*Dacryodes edulis*) and Desert date seed oil (*Balanites aegyptiaca*) was seen to be a promising substitute for traditional synthetic additives in managing wax precipitation in crude oil systems. The study findings reveal that these bio-based materials, when treated with n-hexane, significantly lower the Wax Deposition Temperature (WAT). The examination of wax crystal shapes through microscopy indicates that these natural PPDs successfully disrupt the gel structures linked to paraffinic oils. This disruption enhances the flow properties of waxy crude oil. Furthermore, the analysis of fatty acid profiles backs the idea that these plant-based materials have qualities that help prevent wax crystallization. This not only tackles challenges in the oil industry but also provides an eco-friendly alternative to traditional chemical additives, which lowers potential environmental risks linked to synthetic options.

CRediT author statement

W. C. Okologume conceptualized the study, supervised the research, analyzed the results, prepared the final manuscript, and handled the submission; L. A. Yusuf conducted the laboratory analysis and prepared the preliminary report.

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