POUR POINT DEPRESSION OF WAXY CRUDE OIL USING UNMODIFIED AFRICAN PEAR (*DACRYODES EDULIS*) SEED OIL

Abstract

The pour point depression of a Nigeria waxy crude oil was investigated using unmodified African pear seed oil. Crude oil sample collected from a marginal field in Delta state was analyzed for its physiochemical parameters such as; specific gravity, API, kinematic viscosity, pour point and wax content. African pear seeds sourced from choba Port Harcourt were extracted of their oil content and the oil was analyzed for some oil quality parameters such as; free fatty acid (FFA), acid value, saponification value and iodine value. The results showed a crude oil of high wax content and extracted oil of low FFA and high saponification and iodine values. The waxy crude oil was dosed with the unmodified African pear seed oil of concentrations 0.1ml, 0.2ml and 0.3 ml. The result showed no reduction in the pour point of the crude oil at all concentrations. Therefore the African pear seed oil cannot be used as a pour point depressant in its pure state.

Key words: Pour point; waxy crude oil; depressant; unmodified; African pear; oil

Introduction

The oil industry's midstream sector is largely reliant on the steady flow of crude oil from upstream to downstream. Crude oil is made up of a complex and diverse set of hydrocarbon components (Litvinets et al., 2016). When the temperature falls below the Wax Appearance Temperature (WAT), paraffin waxes separate from crude oil and build up on the inner surface of transfer pipelines (Al-Sabagh et al., 2016). Wax accumulation is a key difficulty in crude oil extraction, separation, transportation, and refining. Because crude oil is susceptible to pressure and temperature changes as it leaves the reservoir and travels down the pipeline, the development of wax crystals in the fluid accelerates (Oseghale and Akpabio, 2012). As a result, viscosity increases as temperature drops, resulting in a decrease in fluidity (Al-Sabagh et al., 2012).

In extreme situations, this might bring crude oil production to a halt. As a result, controlling wax precipitation and wax gelation in flow assurance is a significant challenge. To address this issue, several flow-improvement techniques are used to reduce wax crystal accumulation. To overcome higher pressure drops while transporting waxy crude oil through a pipeline at a temperature below the pour point, extra energy is required.

There are several approaches for improving crude oil fluidity at low temperatures. Heavy oil dilution is a frequently used traditional approach for improving fluidity, whereas heating is employed to prevent low temperatures. Pigging is a method of cleaning and removing wax deposits from pipelines.

The use of chemicals known as pour point depressants as a flow improver is a new, simple, and cost-effective technology (Litvinets et al., 2016). There are several chemicals that have been used for the purpose of Pour point depression of Crude oil. Insistence on the low toxicity and greenings of these depressants has driven researchers to the use of modified and unmodified natural products. This work is one of such attempts at using green substances as Pour Point depressants.

Wax formation and deposition during crude oil production is one of the long-standing problems in petroleum industry. At low temperatures, the crystals of wax easily form impermeable cake. This leads to stoppage of production and sometimes a complete shutdown of facilities and process resulting in loss of revenue for the industries due to reduced output and increased maintenance cost. Hence, there is need to find a solution to the problem posed by wax formation and deposition.

Safou (French), ube (Igbo), elemi (Yoruba), eben (Efik), and orumu (Benin) are all names for *Dacryodes Edulis* (African pear), which belongs to the *Burseraceae* family. They thrive in a wide range of climates and soil types, and they're found all over Africa. Cabinda, Cameroon, Congo (Brazzaville and Kinshasa), Gabon, Ghana, Equatorial Guinea, Nigeria, and Sao Tome are among the countries where they can be found. The trees are grown near homesteads in south-east Nigeria, and blossoming occurs from January to April. Between May and October is the main fruiting season. The fruits are boiled or roasted and eaten with corn in both rural and urban parts of Nigeria (Onuegbu et al., 2011).

Mechanisms of Wax Inhibitors and Pour Point Depressants

The mechanisms of WIs and the influence of WIs on the morphology of wax crystals using actual crude oils were extensively explored in various studies. In fact, the inclusion of WIs is widely recognized for delaying the agglomeration of wax crystals by modifying their shape and size (Li et al., 2010). Despite being extensively studied, the mechanisms of WIs are not adequately grasped and can be rather controversial to a certain degree. The present section briefly describes the core theories and mechanisms of WIs. Table 1 summarizes the mechanisms of different WIs and PPDs. The molecules of both wax and WI undergo the nucleation process. The wax molecules precipitate out from the oil phase of below WAT, which form crystalline nucleus (of a critical size); thus, prompting the formation of a larger compound, specifically the wax crystal. Meanwhile, the high molecular weight of WI propels the crystalline nucleus to self-assemble into a micelle-like aggregate. This eventually forms more subcritical nuclei, which reduce supersaturation and prompt the formation of smaller wax crystals. As a result, these smaller wax crystals remain stable in the oil phase, which suggests improved flowability. Another mechanism involves the co-crystallization process when the WI molecules disrupt the crystallization process and modify the growth of wax crystals. The paraffin wax molecules adsorb on the surface of inhibitors with similar chemical structure, which are then bound together and subsequently form a wax crystal lattice structure in the crude oil. This alters the morphology of growing wax crystals and delays the formation of three-dimensional crystals. Tiny spherical-like crystals (altered in shape from large plate-like crystals) are expected to increase the flowability (Soni et al., 2010).

Table 1 Summary of wax inhibitors and pour point depressants and their corresponding mechanisms (Soni et al., 2010)

Crude oil	WI/PPD	Mechanism	References
Iranian	Ethylene Vinyl Acetate copolymers	Co-crystallization Nucleation Adsorption	Taraneh et al. (2008)
Brazilian	Ethylene Vinyl Acetate copolymers	Co-crystallization	Machado et al. (2001)
Egyptian	Octadecyl Maleate-Vinyl Acetate copolymers	Co-crystallization Wax dispersion	Atta et al. (2015)
Indian	Non-ionic Phenyl-Polyethylene Glycol surfactant	Emulsification	Kumar & Mahto (2017)
Malaysian	Ethylene Vinyl Acetate co-Diethanolamine	Co-crystallization	Anisuzzaman et al. (2017)
China	Polyhedral Oligomeric Silesesquioxane nanocomposites	Co-crystallization Aggregation	Yao et al. (2017)
Indian	Polyhexyl Oleate-co-Hexadecyl Maleimide-co-Alkyl Oleate	Co-crystallization	Patel et al. (2017)
China	Polyoctadecyl Acrylate nanocomposites	Co-crystallization	Yao et al. (2017)
Nigerian	Trichloroethylene-Xylene	Dilution Wax dispersion	Bello et al. (2005)
Model sample	Polyethylene-butene	Co-crystallization	Ashbaugh et al. (2002)
China	Cetyl Trimethyl Ammonium Chloride	Co-crystallization	Gu et al. (2018)
Malaysian	3-2-Methoxyethoxy Propyl-Methyl-bis Trimethylsilyloxy Silane nanohybrid	Wax dispersion Adsorption	Lim et al. (2018)
Indian	Tri-Triethanolamine Monosunflower Ester	Emulsification	Kumar & Mahto (2017)
Egyptian	Ethoxylated fatty alcohols	Wax dispersion Co-crystallization	Khidr & Mahmoud (2007)

Meanwhile, the adsorption of wax molecules on the surface of the WI inhibits the growth of crystals and alters the formation pattern of crystals through the formation of micelle core (El Mehbad, 2017). In general, the WI serves as a “wrapper” that envelopes the wax molecules and prevents their growth with the reduced crystal-crystal adhesion. Following the co-crystallization process of the WI, tiny spherical-like crystals appear in the solubilization process, which improves the dispersion of tiny wax crystals and eventually reduces the WAT. Besides that, the interaction of van der Waals forces between the wax crystals and the long alkyl chain of WI also increases the solubility of wax in the crude oil (Yang et al., 2015).

2.2.2.2 Types of Wax Inhibitors and Pour Point Depressants

The previous section briefly described the examples and mechanisms of WIs. The current section discusses the types and the nature of WIs and PPDs, their mechanisms, their recent applications, and the factors that govern their efficiencies. This section explores the following chemicals, which included EVA copolymers, comb-type copolymers, crystallineamorphous polymers, nano-hybrid PPDs, organic solvents, and surfactants. Table 2 provides recent applications on the reduction of PP using different WIs and PPDs.

Table 2 Reduction of pour point using different wax inhibitors and pour point depressants (Soni et al., 2010)

Crude oil	PP (℃)	Resulted PP (□)	WI/PPD	References
Indian	42	1	Non-ionic Phenyl-Polyethylene Glycol surfactant	Kumar & Mahto (2016)
Nigerian	2	-8	Trichloroethylene-Xylene	Bello et al. (2005)
Russian	21	14	Alkyl Acrylates-Dodecylammonium Acrylate-Dodecylammonium Sulfate	Litvinets et al. (2016)
Egyptian	24	3	Gemini surfactant	Ahmed et al. (2017)
China	19	7	Polyoctadecyl Acrylate nanocomposites	Yao et al. (2016)
Egyptian	24	6	Polyalkyl Linoleate–co–Succinic Anhydride	Soliman et al. (2018)
Egyptian	24	-3	Styrene Maleic Anhydride copolymersAniline-Triethanolamine with Oleic acid blend	Al-Sabagh et al. (2017)
Egyptian	27	6	Modified Maleic Anhydride-co-Octadecane copolymers	El-Ghazawy et al. (2014)
Iranian	8	-10	Ethylene Vinyl Acetate copolymers	Taraneh et al. (2008)
Brazilian	18	-17	Ethylene Vinyl Acetate copolymers	Machado et al. (2001)
Indian	22	16	Polyhexyl Oleate-co-Hexadecyl Maleimide-co-Alkyl Oleate	Patel et al. (2017)
Malaysian	35	10	Ethylene Vinyl Acetate co-Diethanolamine	Anisuzzaman et al. (2017)
China	15	7.5	Cetyl Trimethyl Ammonium Chloride	Gu et al. (2018)

Drawbacks of using Chemical Pour Point Depressant

Chemical pour point depressants have the following drawbacks: the cost of chemical additives can be high, increasing the overall expense of oil production and processing, some chemicals may pose environmental risks due to their toxicity, requiring strict regulatory compliance that complicates operations. The effectiveness of these additives can also vary based on the specific characteristics of the crude oil and environmental conditions, leading to inconsistent results. Chemical reactions may produce undesirable byproducts that complicate processing and alter the oil's properties, potentially affecting its marketability. Handling and application can be challenging, as proper dosing and mixing require specialized knowledge, and the chemicals may need specific storage conditions to maintain efficacy. The effects of PPD chemicals may be temporary, necessitating repeated applications, which adds to costs and operational complexity. Overall, while PPD chemicals can be useful, their use must be carefully managed to reduce these significant drawbacks.

Benefits of using Natural Pour Point Depressants

Natural pour point depressants (PPDs) provide several benefits for promoting the flow properties of waxy crude oil. They are cheaper than synthetic additives, this can help to reduce overall production costs. Also, since they are derived from natural sources, these depressants typically have a lower environmental impact and are not hazardous, making them safer for both workers and the environment. Natural PPDs are generally compatible with various crude oil types and processing systems, minimizing the risk of adverse chemical reactions. They effectively reduce the pour point, improving fluidity and facilitating easier transportation and processing. They align with sustainable practices by utilizing renewable resources and are less likely to negatively alter the oil's chemical and physical properties. In summary, natural PPDs offer a versatile and environmentally friendly solution for oil producers.

Methodology

Sample Collection and Preparation

The crude oil sample used for this study was collected from a Marginal field in Delta State Nigeria. The seed of *Dacryodes Edulis* (African pear) was obtained from Choba Area in Obio-Akpor Local Government of Rivers State. Seed of *Dacryodes Edulis* 4kg were sundried and pulverized to fine texture using an industrial blender.

Characterization of the Crude Oil Sample

The physicochemical characterization of Crude oil was carried out by means of some standard test methods as follows:

Determination of Density, Calculation of Specific Gravity and API Gravity of Crude Oil Samples.

An Empty density bottle was measured using physical balance, the density bottle was filled to the volume mark with the oil. The weight of the density bottle with the oil was measured and recorded

Density $\frac{mass}{volume} = \frac{m_2 - m_1}{v}$ = …………………………………….eqn 1

Where;

M1 = mass of empty density bottle

M2 = mass of density point with crude oil

V = volume of the oil

Calculation of Specific Gravity

Empty density bottle was measured using physical balance, the density bottle was filled to the volume mark with distilled water. The weight of the density bottle with the distilled water was measured and recorded. Same was repeated using the oil. And recorded

The specific gravity = density of crude oil sample/density of water……….Eqn 2

Calculation of API Gravity.

API gravity was calculated using the value of specific gravity obtained from eqn. 2 above. API gravity can be calculated using the following expression

API Gravity $= \frac{141.5}{\text{Specific gravity}} - 131.5$

Determination of Viscosity

Viscosity is a measure of resistance to flow of an oil due to interval friction offer by the fluid

Procedure (ASTM D445)

The viscometer bath is set at 40^0 C and a thermometer was inserted on the bath to take temperature of the bath fluid as a feedback reading. The sample was poured into a suitable viscometer Oswald viscometer to the marked Level. The time taken for the sample to flow to the second marked of the viscometer was recorded.

Kinematic Viscosity (V) = ct (unit is centistoke Cst or mm^2 / s^2)

Where c = viscosity constant of the U-tube in mm^2 /S^2

Determination of Pour Point

Pour point is the minimum temperature at which no oil movement is observed. The ASTM D97 test method was used in pour point determination

Procedure

The sample was placed in a heated water bath for liquefaction and transferred to the test jar up to mark. The test jar was tightly closed by the cork and the thermometer position adjusted so that the thermometer bulb was completely immersed. The test jar was rapidly cooled at a temperature of 8-10^0 C, which was above the expected pour point temperature intervals of 3^0C. This was continued until the sample placed in the test jar showed no movement when aligned horizontally for exactly 5 seconds. The temperature reading on the thermometer was reported by adding 3^0C to the recorded value as the pour point temperature.

Extraction of Dacryodes Edulis (African Pear) Seed Oil

3kg (3000g) of ground African pear (*Dacryodes edulis*) seed was measured using an analytical weighing balance into a container and soaked with 3.5 litres of n-hexane for 2 days. The container was covered and made air tight to avoid evaporation of n-hexane. Decantation was carried out followed by filtration. Distillation of the filtrate to recover the n-hexane was done at a temperature of 65^0C using Soxhlet (Atta, 2015). The percentage yield of the oil was calculated as thus:

$$\% \text{ yield} = \frac{\text{weight of extracted}}{\text{weight of sample used}} \times 100 \text{ .... eqn 3}$$

Determination of Oil Quality Parameters of the Extracted Oil

Saponification Value

0.5g of the oil sample was weighed into a conical flask. 50mls of 0.5N ethanolic solution of potassium hydroxide was added and the solution was refluxed to ensure perfect dissolution. The solution was allowed to cool. 3 drops of phenolphthalein were added. The solution was titrated with 0.5N HCl until the pink color of indicator disappeared (V1). A blank (V2) was carried out as well.

Thus, saponification value was calculated using this equation 4

$$\text{Saponification Value} = \frac{56.1 \times (V2 - V1) \times 0.5}{\text{weight of sample}} \quad \ldots\ldots\ldots\ldots \text{eqn. 4}$$

Iodine Value

0.5g of the oil sample was weighed into a conical flask. 15ml of chloroform was added after which 25ml of Wiji solution (mixture of iodine, acetic acid and chloroform) was added and covered slightly using a foil and masking tape. The resulting solution was placed in the dark for 30 minutes. 20ml of potassium iodide was added followed by 150ml of distilled water. The solution turned red. 5ml of 1% starch indicator was added which turned the solution blue black. The whole solution was titrated with 0.1N sodium thiosulphate till the end point is achieved (V1). A blank (V2) was carried out too starting with 15ml of chloroform. Solution turned to blue black precipitate and then to colourless. Iodine value was calculated using equation 5

$$\text{Iodine Value} = \frac{12.69 \times (V2 - V1) \times \text{Normality of the titrant}}{\text{weight of sample}} \quad \ldots\ldots\ldots\ldots \text{eqn 5}$$

Free Fatty Acid (FFA)

0.5g of the sample was weighed into a dry beaker and 20ml of ethanol added to it. 3 drops of phenolphthalein indicator were added and shook. The solution was titrated with 0.1N sodium hydroxide until a pink colouration was observed.

$$\%\ \text{FFA} = \frac{\text{titre value} \times 0.0282 \times 10}{\text{weight of sample used}} \quad \ldots\ldots\ldots\ldots \text{eqn. 6}$$

Acid Value

2 g of the sample was weighed into a dry beaker, 25 ml of chloroform and 25 ml of ethanol was added and mixed. 2 drops of phenolphthalein were added to the resulting mixture. The mixture was titrated with 0.1N NaOH to the end point of which a dark pink color was observed.

$$\text{Acid value} = \frac{\text{titre value} \times \text{normality of the base} \times 56.1}{\text{weight of sample used}} \quad \text{……….eqn 7}$$

Application of the Unmodified African Pear (Dacryodes Edulis) Seed Oil on the Waxy Crude Oil

The African pear (*Dacryodes Edulis*) seed oil was added to the waxy crude oil in different concentration using a micro-syringe. The concentration range from 0.1 ml to 0.3 ml to 20 ml of the waxy crude oil. The mixture was allowed to stand for one hour and collected for physiochemical analysis.

Results and Discussion

Table 3 Characterization of Crude Oil

S.NO.	Parameters	Unit	Value
1	specific gravity		0.883
2	API	^{0}	28.7
3	kinematic viscosity	Cst	2.09
4	pour point	^{0}C	21
5	wax content	%	29.5

Table 3 shows the physicochemical properties of crude oil sample. Specific gravity gives an idea about the presence of light and high molecular weight hydrocarbons. The lesser the value of specific gravity, the higher the amount of hydrocarbons in the Petroleum (Kumar et al., 2017)

The crude oil samples from Marginal field in Delta State Nigeria have specific gravity of 0.883 indicating a composition of lower and higher molecular weight hydrocarbon. API gravity is reverse of specific gravity and describes the nature of crude oils, i.e., light or heavy. The API of the crude oil is 28.7^0 which indicate medium oil. Pour point is an import parameter used to measure the wax content of crude oils. The pour point measures the temperature at which a crude oil no longer flows, and for paraffinic crude oils, pour points are usually between 12 ^oC and 15 ^oC, and are determined by operation of the dewaxing unit. The pour points of crude oil sample is 21 ^oC. These crude oil samples are within the paraffinic base oils. Values greater than 15 ^oC indicate that the crude oil samples have high wax content as an indication of a high wax content value of 29.5.

The kinematic viscosity at 2.09 Cst is high. According to Davidson et al. (2004) crude oil is heavy, dense and viscous due to the high ratio of aromatics and naphthenes to parafins and high amounts of nitrogen, sulphur, oxygen and heavy metals. the kinematic viscosities have particular role in assessing the producibility of a reservoir (rate and amount of oil production from a reservoir) as well as determining the amount of diluent that will permit pipeline transportation of the crude oil i.e., it is used in calculating the flow of liquids through nozzles, orifices and pipelines. The kinematic viscosity of petroleum products is important for flow of fuel through pipelines, injection nozzles and lubricants for bearings, gears, compressor cylinders and hydraulic equipment. Also, for designing proper temperature ranges for the proper operation of the fuel in burners. The lower the viscosity of a fluid, the more easily it flows.

Table 4 Characterization of *Dacryodes Edulis* Seed Oil (modified and unmodified)

S.No.	Parameters	Unit	Unmodified oil
1	% FFA	%	2.5
2	acid value	mgKOH/g	5.4
3	saponification value	mgKOH/g	94.3
4	iodine value	g/iodine/100g	30.65
5	yield	%	67

Table 4 shows the oil quality parameters of *Dacryodes Edulis* seed oil. The iodine, saponification levels were comparable to those found in other vegetable oils. Free fatty acid, and acid value, on the other hand, were all fairly high. This may be due to the oil's unrefined nature as evidenced by a study by Mohamed *et al*., (2017), which showed that unrefined soybean and cotton seed oils had higher acid value and FFA concentrations than refined oils. The saponification value of oils is a measurement of their capacity to make soap. Saponification readings above a certain threshold indicate a high soap-forming ability and vice versa. *Dacryodes Edulis* seed oil saponification value (SV) was found to be 94.3 mg KOH/g. This figure was lower than the Codex Alimentarius Commission's recommendations of 230-254 mg KOH/g for palm kernel oil and 248-265 mg KOH/g for coconut oil. *Dacryodes Edulis* seed oil's strong saponification suggests that it has a high soap-forming ability, implying that it could be effective in soap manufacturing (Singhal et al., 1991).

Prior to the degradation reactions, the levels of free fatty acid (FFA) and also the acid value (AV) affect the rate of rancidification in oil owing to oxidative degradation. According to Mohamed *et al*., (2017) oil's free fatty acid content is directly proportional to its acid value, therefore the higher the acid value of a vegetable oil, the higher the percentage free fatty acid. In oxidative degradation events, free fatty acids act as pro-oxidants, resulting in an unpleasant flavour in oils (Ghazani *et al.*, 2013). As a result, *Dacryodes Edulis* seed oil's fairly high free fatty acid content (2.5%) and acid value (5.4 mg KOH/g) indicate that it may have poor storage stability. Deodorization methods could be utilized to reduce FFA levels in *Dacryodes Edulis* seed oil and increase its storability (Onuegbu & Igwe, 2011). The degree of unsaturation of fats and oils is measured by the iodine value *Dacryodes Edulis* seed oil has an iodine value of 30.65 g/iodine/100g. The existence of fewer unsaturated C=C bonds in D. *g Dacryodes Edulis* seed oil is indicated by its lower iodine value.

Table 5 Effect of Unmodified *Dacryodes Edulis* (African pear) seed oil on the pour point of a waxy crude oil sample

Concentration of additives in ml	Pour point of waxy crude oil
0.1	21^0 C
0.2	21^0 C
0.3	21^0 C

The pour point of the crude with pour point depressant at different concentration is presented in table 5. The performance of the *Dacryodes Edulis* (African pear) seed oil as crude oil pour point depressant showed no significant reduction in pour point and this could be due to the high acid value and a medium percentage of free fatty acid as shown in table 4 which is above the permissible limit of 3.0 of acid value. This is a clear indication that it couldn't inhibit the wax deposition of the crude oil and shows no effect on the pour point of the crude oil at all the concentrations dosed.

Conclusion

The unmodified African Pear seed oil did not exhibit any significant pour point depression effect on the waxy crude oil even at varying concentrations. This suggests that the natural composition of African pear seed oil may not be sufficient to altar the crystallization behaviour of the wax in the crude oil.

Further modification or processing of the African pear seed oil may be necessary to enhance its pour point depression properties. Other biomaterials or renewable resources can also be used investigated as pour point depressants.

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