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Study of linseed oil, its biodiesel and xylene as flow improver for Nigerian waxy crude oils

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ABSTRACT

In this study, the physicochemical properties of linseed and linseed biofuel were studied and their effect on the flow properties of Nigerian waxy crude oil at different volume fractions was investigated. The Nigerian wax crude oil was characterized for its specific gravity, API gravity, wax content, pour point and cloud point. The properties of the linseed oil and linseed biodiesel investigated include: density, specific gravity, viscosity, pour point, cloud point and flash point. Upon esterification of the free fatty acid (FFA) contained in the linseed oil reduced from 3.5 to 0.75 and the viscosity of the seed oil reduces from 2169 to 362 mm^2/s. The linseed oil and its biodiesel exhibited similar trend of reduced viscosity at higher shear rate, however, linseed biodiesel demonstrated the lowest viscosities, cloud points and pour points owing to reduced fatty contents. The effect of the linseed oil and its biodiesel on the pour point and cloud point of the waxy crude oil was determined; and the performance compared to that of xylene. A rapid drop in the pour point and cloud point was observed with the wax crude pre-heated with 0.1 v/v linseed oil, its biodiesel and xylene.

1. Introduction

Waxy crude oil production and its transportation to the processing facilities face several challenges in the oil and gas industry (Kumar et al., 2018; Fadairo et al., 2019; Soliman et al., 2018). Highly viscous crudes are thicker, more resistant to flow, and include higher quantities of asphaltenes and resins, whereas standard crude oils have API gravities greater than 20 API and contain fewer asphaltenes and resins. Heavy crude oils are difficult to transport because of its density and viscosity. Crude oil with a lot of wax has a high pour point and behaves differently from Newtonian fluid viscosity below the cloud point. Generally, waxy crude structure shifts over three stages; aggregation, precipitation/deposition and gelation (Elganidi et al., 2020). Consequentially, such phase-transitional behavior alters the rheological behavior of waxy crude from Newtonian to a more complicated non-Newtonian behavior resulting in an instant change in the viscosity of the waxy crude (Deka et al., 2018; Soliman et al., 2018; Fadairo et al., 2019; Bern et al., 1980; Elganidi et al., 2020). Deposition of paraffin wax in production flow lines generally results in decreased internal pipe diameter, reduced flow rate, and eventually stopped-flow (Elganidi et al., 2020; Fadairo et al., 2019; Soliman et al., 2018). The reduced flow ability of waxy crude is considered to be one of the costly complications which contributes to high downtime and priced treatment strategies (Fadairo et al., 2019). The severity of the deposition of paraffin depends primarily on the percentage of paraffin wax present in the crude oil, the roughness of the vessel, the cloud point, the pouring point, the rate of gas evolution, the depth of the well, the ambient temperature, the operating temperatures, the drop in pressure, the shear history and manufacturing practices (Singh et al., 2004; Singh et al., 2004; Singhal et al., 1991; Fadairo et al., 2019).

The oil industries invest heavily into prevention and removal process of wax deposition. There are various methods for improving flowability of heavy crude by reducing viscosity, including crude oil dilution with lighter crudes, alcohols, surfactant addition, and pipe line heating, among others (Singh et al., 2000).

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The viscosity of the crude reduces dramatically as the temperature rises, indicating a shift in rheological behavior (Fadairo et al., 2019; Soliman et al., 2018). Chanda et al. (1998) evaluated the effect of asphaltenes in the rheological behavior of Indian waxy crude. They made Polybehenyl acrylate (PBA) with various molecular weights and tested its performance as a flow improver and a pour point depressant. Another option for increasing crude oil flowability is to use surfactants and water to build stable O/W emulsions with crude -oils. When used appropriately, surfactant addition aids emulsification. However, they have a number of disadvantages, including the fact that surfactants are difficult to come by, complicated to manufacture, and expensive. The use of Na_2CO_3 to emulsify heavy crude in water has been utilized to increase flow (Zhao et al., 2013). Adding more Na_2CO_3 to oil results in an increase in crude-water interfacial tension, despite the fact that it is less expensive.

The inclusion of light petroleum fraction is one of the other modes of transportation. Pre-treatment of waxy crude by a combination of the aliphatic solvents' such as kerosene or diesel with an aromatic solvent such as xylene or toluene have been observed to improve the flow behavior of waxy crude and increase the efficiency of wax removal, likewise pre-treatment with surfactant which has demonstrated great performance especially in dispersion of wax crystal (Akinyemi et al., 2016). Their demerits are in the initial cost of addition and later their extraction and toxic nature. Another remediation method in the pipeline industry involves the use of polymeric additives in small amount to depress pour and gelation points and enhance the rheological properties of waxy crude in low temperature environment (Elbanna et al., 2017; Aiyejina et al., 2011; Than et al., 2017). It has been reported that the performance of polymeric additives as flow improver is a function of the extent of similarities between the polymer and waxy component structures. Therefore, establishing these similarities is a major concern, coupled with limitations due to cost or how much crude oil the additive can handle per time and its environmental friendliness. Furthermore, study on the use of non-edible plant seed oils as pre-treatments known as flow improver was reported by Akinyemi and Ayoola (2016). These natural chemicals obtained from plant seeds showed good potentials as flow improvers and pour point depressants however, their high unsaturated fatty acid contents could pose a serious challenge during cold flow. Recently, experimental results of Fadairo et al. (2018) gave insight into the potentials of low pour and cloud points seed oil biodiesel as flow improver for Nigerian waxy crude. The study in this research involves characterization of crude oil sample from the Niger Delta oilfield Nigeria to determine its wax content and the extraction/esterification of linseed oil as pour point and cloud point depressant of Nigerian waxy crude. Linseed oil has a comparatively low cloud and pour point (Table 1) (Demirbas, 2009). Hence, we have chosen linseed oil over other seed oils. Linseed comes from a flax crop which is a cool season herbaceous annual plant crop with short taproot system. It belongs to the genus of Linum. It is a Mediterranean crop that has now spread across the world today (Otabor et al., 2019).

2. Materials and method

In this study the following materials were used; linseed oil, benzene, acetone, methanol, crude oil, phenolphthalein indicator, 0.5M hydrochloric acid, potassium hydroxide (KOH) pellets, sodium methanoxide and magnesium sulphate. All chemicals are of analytical grade and supplied by Equilab Nigerian limited, Nigeria. The crude oil was obtained from an indigenous oil operator in the Niger Delta area of Nigeria (Name and location withheld due to proprietary reasons). Linseed oil was procured from FIIRO (Federal Institute of Industrial Research) a research institute that uses mechanical press to extract oils from seeds. The fatty acid composition analysis involves the determination of the triglycrides present in the oil achieved by classifying the free fatty acids present in the oil, this was conducted using the Agilent Technologies 7890A Gas chromatograph system USA. The gas chromatograph is a very sensitive apparatus consisting of a flame ionization detector (FID) and a capillary column.

2.1. Crude oil characterization and rheological analysis

The wax content of the virgin crude was analyzed at the Petroleum Training Institute, Effurun, Nigeria (PTI). The method is as given: a modified Universal Oil Products (UOP) 46–64 method was used to measure the amount of wax present in the virgin crude oil. Before the characterization, the crude oil was heated to remove all water content dispersed in the oil as emulsion. The presence of water in the oil could lead to ambiguous readings during the characterization and determination of the crude's other characteristic properties.

2 g crude oil was mixed with 40 ml n-pentane and thoroughly agitated for about 30 min 120 mL acetone (acetone/n-pentane ratio 3:1) was added to the mixture and preserved in a deep freezer for 24 h at $-20.15\ ^\circ C$. The solid deposition in the mixture was then separated using a Buchner funnel with a glass microfiber Whatman filter (No. 934AH). Separated solid phase was then dissolved in n-hexane to remove asphaltene. After solvent removal, the product was weighed to obtain wax content. Pour point of crude oil samples was determined using standard ASTM method (ASTM D97-17a). Crude sample in a test tube fitted with a thermometer was preheated to around 60 °C before introducing into a cooling bath of pour point apparatus. At every 3 °C drop in temperature the flow was recorded and the lowest temperature at which the flow stops is noted as the pour point.

Viscosity is an essential physical property of any fluid to describes its flow ability. The crude oil, linseed oil and its biodiesel were characterized for their rheological behavior using an OFITE 800 viscometer equipped with a regulated speed that can be manually controlled. According to standard procedure, the deflection readings for the shear rate of rotor speed (3 -600RPM) were taken at about 42 °C, since the crude already congealed at room temperature.

Table 1
Seed oils and their properties.

Seed Oil	Botanical Name	Cloud Point (°C)	Pour Point (°C)	References
Castor seed oil	*Ricinus communis*	−9	−18	Salimon et al. (2010)
Jatropha seed oil	*Jatropha Curcas*	0	−19	Akintayo (2004)
Rubber seed oil	*Hevea brasiliensis*	4	−6	Abdulkadir and Danbature (2014)
Linseed oil	*Linum usitatissimum*	−14	−24	Demirbas (2009)
Neem seed oil	*Azadirachta indica*	7	−8	(Ayhan, Abdullah and Manzoor 2016)
Karanja seed oil	*Pongamia pinnata*	10	0	(Ayhan, Abdullah and Manzoor., 2016)
Tobacco seed oil	*Nicotiana tabacum*	−7	−17	Ayhan and Manzoor, 2016)

2.2. Bio-diesel conversion of the seed oil

The esterification process was conducted as follows (Abdulkadir and Danbature, 2014): firstly, 200 ml Linseed oil (180 g) was added into 500 ml three-necked round-bottom flask mounted with a reflux condenser that is equipped with a mechanical magnetic stirrer set at 1200 revolution per minute. To start with, 46 ml methanol in ratio 6:1 was added to the flask and heated to 60 °C via a Stuart™ digital hotplate stirrer (model Z761303). The inner temperature of the mixture was monitored using a digital temperature probe. This was followed by addition of 0.5 wt % sodium methanoxide (25 wt % in methanol). The mixture was transferred into a separating funnel after the reaction mixture attained equilibrium at about 2 h. From the segregated layers, the base glycerol part was removed by gravity segregation after settling time of around 2 h, subsequently, residual methanol was removed using a rotary evaporator under reduced pressure of about 25 mbar at room temperature. The remaining Linseed oil methyl ester layer was kept and washed with 1000 ml of deionized water at 60 °C until a resultant pH of 8.18 was attained. The oil was then cooled in a separating funnel at room temperature after the water had been successfully removed. After cooling, the linseed methyl ester was dried with magnesium sulphate.

3. Result and discussion

3.1. Analysis of the crude oil sample

The crude oil sample was characterized for API gravity, pour point, wax content, and cloud point, the results presented in Table 2. The API gravity was measured using ASTM D287 method indicated that the crude oil is 29.8%, this based on the API specifications implies the crude oil sample is within the medium oil classification. However, the crude can be considered highly waxy owing to the high percentage of wax it contained (29.8%) and can be seen congealing even at room temperature. For the crude sample, a very high pour point value was recorded. The high pour point of the oil can be linked to its high wax content, which is a major contributor in wax deposition in the pipeline and lower flowability (Deka et al., 2018). Hence, the major reason why this particular crude could not flow even at room temperature and also the deposition of its wax content in piping.

3.2. Analysis of the linseed oil and its biodiesel

Fig. 1 shows the linseed oil's fatty acid composition, while Table 3 presents some of the basic physicochemical properties of the linseed oil and its biodiesel. The fatty acid analysis indicates that oleic acid, linoleic acid and linolenic acid are the major fatty acids in the biodiesel fatty component. The basic properties of the linseed oil and its biofuel are comparable, as shown in Table 3. The density of linseed oil is higher than that of its biodiesel, which is owing to the linseed oil's fatty acid content, as well as its free and bound glycerine content (Acaroglu, Demirbas, 2007; Benjumea et al., 2008). The linseed oil pour point, which is the minimum temperature below which loses its flow characteristics, demonstrated a drop in measured value from −24 to −27 °C upon esterification. The high monounsaturated content of the linseed oil is the reason for its eventual cold flow properties. Furthermore, important acid values present in the linseed oil and its biodiesel are free glycerol, total glycerol and free fatty acids, and these values are vital to biodiesel's low operating temperature and kinematic viscosity (Acaroglu, Demirbas, 2007). As can be observed, the free fatty acid content (FFA) reduced from 3.50 to 0.75 upon esterification while the seed oil viscosity drops from 2169 to 362 mm^2/s in the linseed oil biodiesel. The reduction in biodiesel's friction-related properties can be attributed to the reduced amount of free fatty component that has been removed during biodiesel conversion. This drop-in pour point indicates that the formulated bio-diesel from linseed oil could serve as a flow improver of waxy crude. The cloud point and pour point values of the linseed biodiesel (−17 and −27 °C) indicates an excellent low temperature operability compare to the raw seed oil owing to the low saturated fatty acid content (Demirbas, 2007).

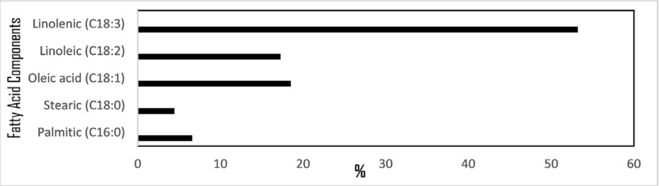

Fig. 1. Fatty acids of Linseed oil.

3.3. Rheological analysis

The viscosity values at different shear rates for the waxy crude were estimated from the deflection readings and plotted against shear rate to understand the trend of viscosity variation with shear rate and were compared for the ordinary linseed oil and the biodiesel, with results presented in Fig. 2. As it can be observed from the rheogram (Fig. 2), the linseed oil biodiesel and waxy crude exhibited reduction in viscosity with increase in shear rate but generally, the viscosity values are fairly constant at higher shear rate values.

The waxy crude demonstrated a shear thinning behavior above its pour point 23 °C which could be due to shift in the structure of the waxy crude over the stages of wax precipitation at temperatures above it pour point that eventually affects its rheology (Elganidi et al., 2020). At temperatures below coagulation of the wax crystal in the crude a no flow condition results. The linseed oil and its biodiesel showed close viscosity values with variation in shear rate but similar trend because the ordinary linseed oil contains less amount of free fatty acid (see Table 3) (Demirbas, 2009). It can be argued that original wax content of waxy crude mostly affects its rheological behavior and it is evident in the differences observed between the waxy crude viscosity-shear rate trend and that of the linseed oil and its biodiesel.

Table 2
Physical properties of crude oil samples.

Parameter	Observed	Method
Specific Gravity (at 15 °C)	0.88	ASTM D1480 -15
API Gravity	29.3	ASTM D287
Pour Point (C)	23	ASTM D97 -17a
Cloud Point (C)	37.9	
Wax Content (%)	29.8	Universal Oil Products (UOP) 46–64 method

Table 3
Physiochemical properties of the additives.

Properties	Linseed oil	Linseed oil Biodiesel	Ordinary Diesel
Density (g/cm^3)	0.89	0.86	0.835
Colour	Brown	Golden Brown	Brown
Specific Gravity	0.065	0.129	0.835
Acid Value (mgKOH/g)	7.01	1.12	–
Peroxide Value	2.50	1.50	–
Saponification Value (mgKOH/g)	17.50	22.50	–
Iodine Value (g/100g)	247.00	339.12	–
Viscosity (mm^2/s)	2169	362	381
Free Fatty Acid (mg/KOH/g)	3.50	0.75	–
pH	5.89	6.69	–
Pour Point (°C)	−24	−27	−16
Cloud Point (°C)	−14	−17	−1
Flash Point (°C)	–	–	145

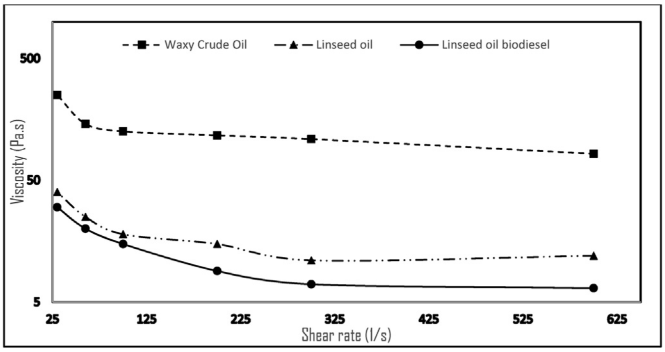

Fig. 2. Rheological behavior of the waxy crude in the presence of bio-additive.

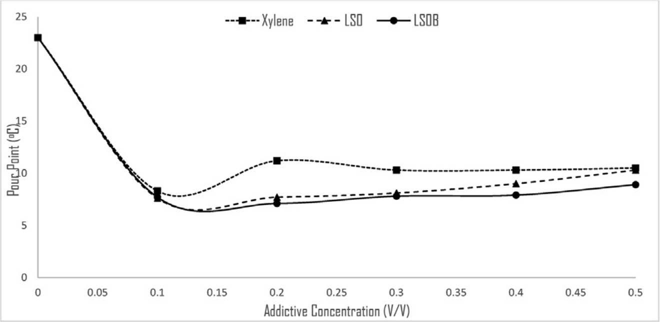

Fig. 4. Effects of different concentrations of bio-additive on pour point of Nigerian waxy crude.

3.4. *Effect of formulated biodiesel on crude oil*

To evaluate the effect of linseed oil and biodiesel on the pour point and cloud point of the waxy crude, the waxy crude was pre-treated with linseed oil and biodiesel at different volume fractions, and then compared with commercial chemical wax dissolver (xylene). These pre-treated samples were then tested for its pour and cloud points with results presented in Figs. 3 and 4 for both pour points and cloud points respectively. From both plots, the cloud points and pour point values were obtained for the raw crude at 37.9 and 23 °C respectively. A rapid drop in both pour point and cloud point was observed with the wax sample pre-treated with 0.1 v/v linseed oil, its biodiesel and xylene. This reduction could be attributed to the interaction between the molecules in the wax structure and that of the esters from the natural plant seed oil and its biodiesel. Consequently, results into reduced ability to form hydrogen bonds with the hydroxyl or carboxyl groups (Elganidi et al., 2020). Generally, samples pre-treated with xylene exhibited the least depression on the pour point and cloud point. As the volume fraction of the additives in the crude sample increases, the pour point and cloud point depression increases gradually and eventually remains fairly unchanged. It has been generally reported that xylene is an effective pour point depressant at low concentration but linseed oil and its biodiesel would achieve better performance at the same concentration on most waxy crude. As explained by Akinyemi and Ayoola (2016), the reduction in pour point and cloud can be attributed to the binding of the mono-saturated molecules present in the linseed oil and its biodiesel with the larger paraffin molecule in the waxy crude preventing them from coagulating and aggregating before been deposited. For additive concentrations above 0.1 v/v, the fairly constant values of both pour point and cloud point can be said to be due to the fact that most of the paraffin molecules have been taken care of at optimum concentration which is 0.1 v/v. Therefore, the extent to pour point and cloud point depression upon addition of additives is a function of the amount of paraffin wax and the molar distribution in the raw crude (Leggieri et al., 2018).

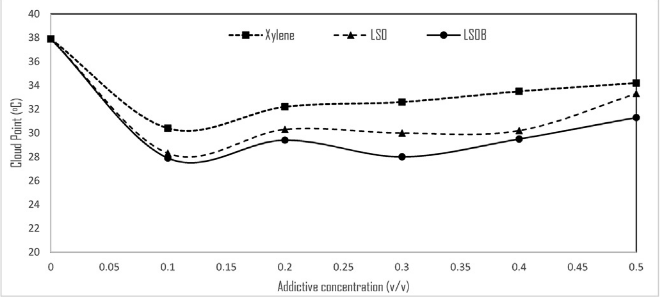

Fig. 3. Effects of different concentrations of bio-additive on cloud point of the Nigerian waxy crude.

4. Statistical analysis of the treatments effectiveness

Statistically, one-sample *t*-Test is generally used to compare a sample mean to a hypothesized value for the population mean to determine whether the two means are significantly different statistically (De Winter 2013). In other words, it helps to ascertain whether the mean of a population is statistically different from a known value. Therefore, in this section, the effectiveness of each of the additives was evaluated statistics-wise using a commercial

Table 4
Statistical evaluation of the effects of several additives on the waxy crude's pour point.

	Additives		
Statistical parameter	***Xylene***	***LSO***	***LSBD***
Test Value (Base case cloud point)	23	23	23
Mean	10.12	8.54	7.88
Std. Deviation	1.08	1.13	0.65
Std. Error Mean	0.48	0.51	0.29
t-value	−26.60	−28.658	−52.05
Mean Difference	−12.88	−14.46	−15.1230
Sig. (2-tailed)	0.000012	0.000009	8.15E-7

Table 5
Statistical evaluation of the effects of several additives on the waxy crude's cloud point.

	Additives		
Statistical parameter	***Xylene***	***LSO***	***LSBD***
Test Value (Base case pour point)	37.9	37.9	37.9
Mean	32.58	30.34	29.30
Std. Deviation	1.45	1.93	1.29
Std. Error Mean	0.65	0.86	0.58
t-value	−8.23	−8.77	−15.81
Mean Difference	−5.32	−7.56	−8.60
Sig. (2-tailed)	0.01	0.000931	0.000121

software (statistical package for social sciences, SPSS). The untreated waxy crude cloud and pour point values were used as the hypothesized mean (known mean or base case). The sample mean for each of the treatment methods' cloud and pour points across all the concentrations was estimated. Then, the performance of the additives (treatment methods) in depressing the cloud and pour points was quantified via some descriptive statistics. In addition, to determine whether there was significant difference in the cloud and pour point of the waxy crude with and without additives, the average cloud and points of all the samples treated with the three (3) additives (xylene, linseed oil (LSO), linseed biodiesel (LSBD)) were compared with the known mean. The null hypothesis is such that there no significant difference between the sample means (the 3 additives) and the hypothesized mean. From the analysis of the results, Tables 4 and 5 for both pour and cloud points, respectively, all the three additives show significant differences between the hypothesized mean and the sample mean which is evident in the significant differences reported. This implies a substantial reduction in the waxy crude pour and cloud points upon doping with the additives. However, both linseed oil and its biodiesel compete well with the commercial wax dissolver (xylene) with added advantage of being biodegradable and environmentally friendly, which also bolsters the earlier results analysis. The descriptive statistics results also indicate linseed biodiesel exhibits better depression power for both pour and cloud points. This is evident in its superior mean and mean difference.

5. Conclusions

The effect of linseed oil and its biodiesel on the cloud point and pour point of Nigerian waxy crude oil was investigated in this present study. Linseed oil and biodiesel used in this study had cloud points of −14 °C and 17 °C and pour points off −24 °C and −27 °C respectively. The linseed and its biodiesel remarkably depressed the cloud point and pour point of the Nigerian waxy crude and hence, offer alternative solvent for wax dissolving. The cloud point and the pour point of the biodiesel had a close relationship with the that of the linseed oil. It was observed that a pre–treatment of the wax crude with between 0.1 and 0.15 v/v concentration of the linseed and its biodiesel as additives significantly decreased the cloud point and pour point of the waxy crude thereby enhancing its rheology.

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