

## Cashew Nut Shell Liquid-Ethylene Glycol Derivatives as Pour Point Depressants for Waxy Crude Oil

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### Abstract

The addition of pour point depressants is widely adopted as a mitigative strategy for wax control during the production and transportation of waxy crude oils. Pour point depressants derived from plant-based natural products have the potential to meet the three-fold criteria of efficiency, affordability, and eco-sustainability. Natural cashew nut shell liquid was extracted from *Anacardium occidentale* shells – an agro-waste – and derivatized using ethylene glycol. The derivative was evaluated as a pour point depressant and low-temperature flow improver for waxy crude oil. The pour point of oil doped with additive was reduced by -15°C. The effect of additives on the morphology and microstructure of wax crystals in oil was studied by cross-polarized microscopy. The micrographs, processed and analyzed with Image J software, showed changes in wax microscopic properties as the size, aspect ratio, and boundary fractal dimension of wax crystals in doped oil were reduced, while crystal circularity and solidity increased, signifying the formation of smaller, rounder crystals with smoother, even surface. The flow properties of the waxy crude oil, measured using a coaxial cylinder rotational viscometer, showed 47.8% reduction in oil shear stress and viscosity. The improvement in oil flowability was related to the changes in wax microscopic properties.

**Keywords:** Cashew nut shell liquid; Pour point depressant; Flow improver; Wax crystal morphology; Waxy crude oil; Ethylene glycol.

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## 1. Introduction

Precipitation and deposition of wax often impede the production and transportation of paraffinic crude oil. Wax formation and the ensuing flow assurance problems can be severe and difficult to alleviate, especially in deep-water developments [1-3]. Long-chain alkanes, which constitute a large proportion of paraffinic oils, are sensitive to temperature change. Decreasing temperature, coupled with the loss of light end hydrocarbons, destabilize these high molecular weight normal alkanes (wax), resulting in their precipitation from the oil [4-6]. Wax precipitation commences by the formation of stable clusters of paraffin molecules in the nucleation stage, followed by crystal growth and agglomeration as new paraffin molecules are attached to the nuclei [7-8]. Waxes form an interconnected network of crystals in the oil, which increases oil viscosity, reduces pumpability, and increases the risk of oil gelation in the pipe if the flow is temporarily stopped [9]. Wax formation has serious cost implications arising from additional energy cost in pumping oil as it becomes more viscous, expenses incurred on repairs/replacement of damaged production equipment, reduction in oil output, and reduction in capacity utilization [10-12]. Chemical addition is often employed as a cost-effective mitigative strategy for the control of wax formation [13-14]. These chemical additives variously described as pour point depressants/wax inhibitors/ wax crystal modifiers/wax dispersants interfere with the wax formation process, thus helping the oil retain its flowability at low temperatures [1,14]. Most pour point depressants investigated in literature are synthetic polymers [15]. However, due

to the high cost and deleterious environmental impact of many of the traditional pour point depressants, there has been growing interest in the application of natural products and their derivatives as inexpensive, eco-friendly pour point depressants [16]. Pour point depression and wax deposit inhibition have been reported using raw palm oil and palm kernel oil as well as castor, jatropha, and rubber seed oils [16-18]. Derivatives of olive, canola, and soybean oil have also been applied as crude oil pour point depressants [19-21]. Also, natural cashew nut shell liquid and derivatives of distilled (technical) cashew nut shell liquid have been used for pour point depression and flow improvement of waxy crude oils [23-24]. Cashew nut shell liquid (CNSL) is particularly attractive for this application because it is a cheap source of alkylphenols, and unlike most plant seed oils, is not a food-source [25]. CNSL is a mixture of four alkyl/alkenyl phenols: anacardic acid (6-pentadecyl salicylic acid) cardanol (3-pentadecyl phenol), cardol (5-pentadecyl resorcinol) and methylcardol (2-methyl-5-pentadecyl resorcinol) [25-28]. Its potential as an oil additive is evident in its unique chemical composition. Depending on the method of extraction, cashew nut shell is classified as either natural or technical. Natural CNSL is extracted cold or with low-boiling solvents and contains mainly anacardic acid, whereas technical cashew nut shell liquid is extracted by pyrolysis of the shell and contains mainly cardanol [26].

This paper reports the preparation and evaluation of low-cost additives for pour point depression of waxy crude oil by esterification of natural CNSL with ethylene glycol. The effect of additives on wax crystal morphology and microstructure was studied by cross-polarized microscopy. Changes in the flow properties of oil on addition of the CNSL derivative was evaluated by viscometric analysis.

## 2. Experimental

### 2.1. Materials

Ethylene glycol (BDH chemicals); methanol, n-heptane, petroleum benzene, dichloromethane, toluene, and xylene (Sigma-Aldrich); Niger Delta waxy crude oil (donated by Shell Petroleum Development Company, Port Harcourt)

### 2.2. Crude oil characterization

Specific gravity and API gravity (15.5°C) of the crude oil samples were determined by the hydrometer method (ASTM D 1298-12b). Standard test method for kinematic viscosity of transparent and opaque liquids and calculation of dynamic viscosity (ASTM D455-12) was adopted for determination of kinematic viscosity at 40°C and 100°C using a Stanhope-Seta KV-8 Viscometer bath.

ASTM D 4006-11 was adopted for the determination of crude oil water content by Dean-Stark distillation. 50mL of xylene was mixed with 50mL of crude oil. The mixture was heated for 1hr and water collected in a Dean-Stark trap.

The wax content of crude oil was determined by the acetone method (UOP 46-64) with slight modification by eliminating the oil clarification step. 5g of crude oil was completely dissolved in 50mL of toluene, after which the solvent was evaporated. The residue was dissolved in 100ml of 3:1 mixture of petroleum benzene and acetone, stored in deep freeze (-20°C) for 2hours, and then filtered with a pre-weighed filter paper and dried. The weight of dried wax precipitate was taken, and wax content was calculated.

The wax appearance temperature was determined according to ASTM D5772- Standard test method for cloud point of petroleum products (linear cooling rate method) using an Olympus BX51 cross-polarized microscope. The detailed procedure is provided in 2.5.2.

Pour point was determined according to the Standard Test Method for Pour Point of Crude Oils (Procedure A) (ASTM D5853-17) using a Stanhope-Seta cloud & pour point refrigerator (93531-7). The sample was warmed to 60°C in a water bath so as to dissolve any pre-existing wax and then inserted in the cooling jacket of the pour point equipment and monitored for flow every 3°C decrease in temperature.

3g of crude oil sample was refluxed in 90mL of heptane for 1hr to determine the asphaltene content (heptane insolubles). The solution was allowed to cool, and then vacuum-filtered using a 0.45µm Millipore® filter paper. The weight of asphaltene precipitate was taken, and asphaltene content was calculated.

Asphaltene component was first removed using heptane. The saturates, aromatics, and resin components of deasphalted crude oil (maltenes) were separated in order of increasing polarity on a silica gel column by elution with petroleum benzene, toluene, and 3:1 dichloromethane: methanol mixture, respectively.

### **2.3. Extraction of CNSL**

Cashew nut shell liquid was extracted from macerated cashew nut shell using acetone as described in literature [22]. The solvent was recovered from the extract in vacuo using a rotary evaporator. Natural CNSL contains 65–90 % anacardic acid [26–30]

### **2.4. Derivatization of CNSL**

Cashew nut shell liquid (50mL) in a three-necked flask was mixed with 8mL of ethylene glycol. The reaction was catalyzed with sulfamic acid. The mixture was heated at 110–120°C for 45mins with constant stirring and temperature monitoring. Reaction continued till the end of collection of water of esterification. The product was labeled CN<sub>1</sub>EG. A similar reaction was carried out with 100mL of CNSL and 8mL of ethylene glycol. The product was labeled CN<sub>2</sub>EG. The spectra of each derivative and the precursors were recorded with a Fourier Transform Infrared Spectrophotometer (FTIR).

### **2.5. Performance evaluation of CNSL derivative**

#### **2.5.1. Pour point**

The crude oil sample was warmed to 60°C in a water bath in order to dissolve any wax that may already exist in the oil. Using a Hamilton Co. micro-syringe, oil in pour point test jars were doped with the additive at 500ppm, 1000ppm, 2000ppm, 4000ppm, and 5000ppm, respectively. Test jar containing doped oil was re-corked, a thermometer inserted, and then placed in the cooling jacket of Stanhope-Seta cloud & pour point refrigerator. Pour point was determined according to ASTM D 5853-17.

#### **2.5.2. Cross-polarized microscopy**

Optical microscopy study of wax formation in the crude oil was carried out using an Olympus BX51 cross-polarized microscope. 5 drops of crude oil sample in quartz sample holder was placed in the sample compartment of the Linkam THMS 600 thermal stage. The process was controlled by Linksys32 software system via T95-linkpad system controller. The temperature program was automated, involving an initial heating cycle at a rate of 5°C/min to a maximum temperature of 60°C, a hold-time of 1min, followed by the cooling cycle at a rate of 1°C/min to a minimum temperature of 10°C. The stage was cooled with liquid nitrogen (powered by LNP95 liquid nitrogen pump) and water. Micrographs of the oil were captured as the oil-cooled. For samples containing the additives, the oil was first warmed to 60°C before doping and analysis. The micrographs were processed and analyzed using Image J software to determine the microscopic properties of the wax crystals. Wax crystals were characterized in terms of size (crystal area), aspect ratio, circularity, solidity, and boundary fractal dimension. The boundary fractal dimension was determined by the box counting method.

#### **2.5.3. Viscometric analysis**

The viscometric analysis was carried out using a coaxial cylinder rotational viscometer (Ofite 900). The oil sample was warmed to 60°C in a water bath, doped with additive, and transferred to the viscometer cup. The shear stress and viscosity of the oil were measured at shear rates between 10-100rpm (corresponding to shear rates of 17.02/s–170.23/s) and temperature intervals of 5°C from 50°C–10°C.

### 3. Results and discussion

#### 3.1. Crude oil characterization

The physico-chemical properties of the crude oil are presented in Table 1. It shows that it is a light oil with moderate wax content. The SARA composition indicates an abundance of saturates and low asphaltene and resin content (Table 2). The predominance of saturates correlates with its high API gravity and predisposes the oil to wax instability.

Table 1. Physico-chemical properties of crude oil

Property	Crude oil	Property	Crude oil
Specific gravity (15.5°C)	0.839	WAT (°C)	26.1
API gravity	37.2	Pour point (°C)	12
Kinematic viscosity (40°C) cSt	6.94	Wax content (%)	8.9
Kinematic viscosity (100°C) cSt	1.43	Water content (%)	0.025

Table 2. SARA Composition of crude oil, molecular group type

Saturates (%)	Aromatics (%)	Resins (%)	Asphaltenes (%)
63.7	31.8	3	0.05

#### 3.2. Derivatization of CNSL

The FTIR spectra of the derivatives, ethylene glycol, and natural cashew nut shell liquid are shown in Fig.1.

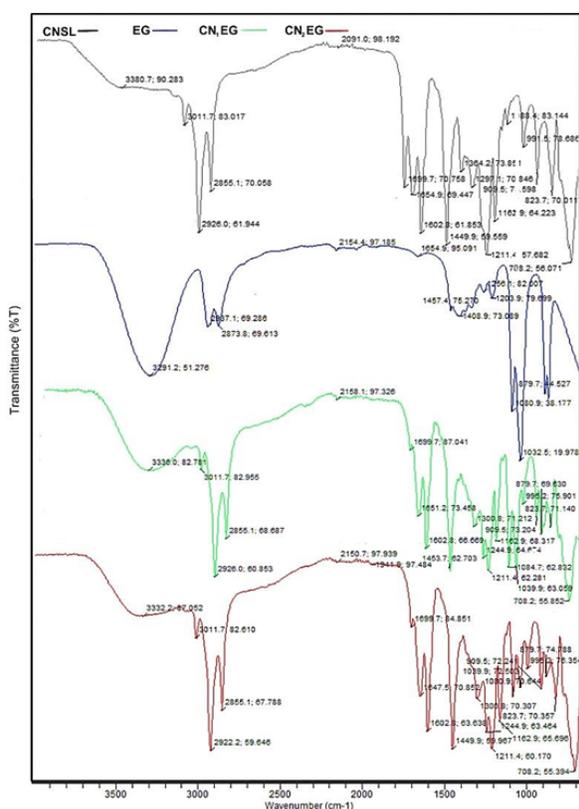


Figure 1. FTIR spectra of CNSL-ethylene glycol derivatives

ethylene glycol are progressively diminished with increasing the mole ratio of increases from 19.9% in EG to 63.06% in CN<sub>1</sub>EG (monoester) and 72.5% in CN<sub>2</sub>EG (diester), indicating that the alcohol groups CNSL, as they are used in ester bond formation with carboxylic acid groups of CNSL. The intense bands at 1602.8cm<sup>-1</sup> in natural CNSL, CN<sub>1</sub>EG, and CN<sub>2</sub>EG are due to aromatic C = C vibration of the benzene ring.

The broad bands at 3332.2cm<sup>-1</sup> and 3336.0cm<sup>-1</sup> in the spectra of CN<sub>1</sub>EG and CN<sub>2</sub>EG are due to O – H vibrations of the phenol group of CNSL. The band at 3011.7 cm<sup>-1</sup> is the = C – H stretching vibration of the alkene from the 15-carbon aliphatic chain of CNSL, confirmed by = C – H deformation (bending) vibrations at 708.2 cm<sup>-1</sup>. Similarly, the vibrations at 2926.0 cm<sup>-1</sup> – 2922.2cm<sup>-1</sup> and at 2855.1 cm<sup>-1</sup> are due to C – H vibrations of methylene and methyl groups of the alkyl chain. Deformation vibrations of methyl C – H occur at 1453.7 cm<sup>-1</sup> – 1449.9cm<sup>-1</sup>. The broad band at 3291.2 cm<sup>-1</sup> in the spectrum of ethylene glycol (EG) is due to O – H vibration of the alcohol, confirmed by C – O vibration of primary alcohol at 1032.5 cm<sup>-1</sup>. The intense band at 1669.7cm<sup>-1</sup> in natural CNSL corresponds to C=O vibrations of the carboxylic acid group (attached to aromatic). The diminution of this band and increase in its transmittance value in the spectra of CN<sub>1</sub>EG and CN<sub>2</sub>EG coincides with the appearance of new bands at 1244.9 cm<sup>-1</sup>, which correspond to C–O vibration of ester. As a corollary, the transmittance value of the C–O band of ethylene glycol (1039.9cm<sup>-1</sup>) sequentially of

### 3.3. Evaluation of CNSL Derivatives

#### 3.3.1. Effect on pour point

The effect of CN<sub>1</sub>EG and CN<sub>2</sub>EG on the pour point of crude oil is shown in Table 3. Pour point depressant activity of the additives is dosage dependent increasing up to an optimal dosage beyond which pour point depression efficiency declines. CN<sub>1</sub>EG and CN<sub>2</sub>EG reduced the crude oil pour point by -15°C and -9°C respectively at the same concentration indicating greater pour point depression efficiency of the monoester. This is attributable to the compositional and structural differences of the derivatives. The progressive increase in oil pour point at treat rates above the optimal dosage suggests that the presence of the excess amount of additive molecules in the oil is detrimental to the pour point depression mechanism. For its greater pour point depression efficiency, CN<sub>1</sub>EG was selected for further evaluation by optical microscopy and viscometry.

Table 3. Effect of additive dosage on pour point

Dosage(ppm)		0	500	1000	2000	4000	5000
Pour point (°C)	CN <sub>1</sub> EG	12	0	-3	0	3	3
	CN <sub>2</sub> EG	12	3	3	6	6	9

#### 3.3.2. Effect on wax crystal morphology

The optical micrographs of neat crude oil (without additive) and crude oil doped with 1000ppm of CN<sub>1</sub>EG at 20°C and 10°C are shown in Fig. 2.

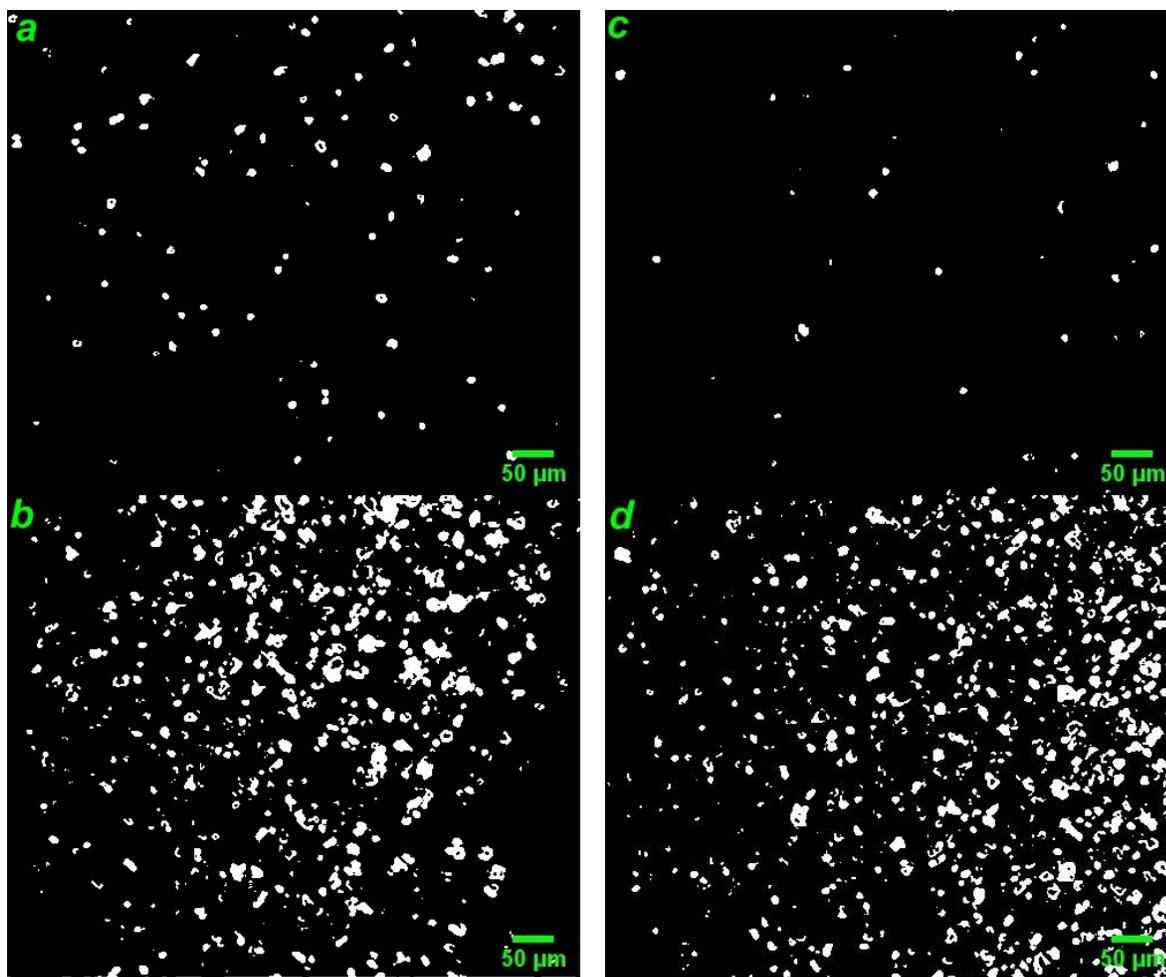


Figure 2. Cross polarized micrograph of neat crude oil at 20°C (a); 10°C (b); and crude oil doped with CN<sub>1</sub>EG at 20°C (c); 10°C (d)

As the temperature of the crude oil decreases further below the wax appearance temperature, the size and quantity of crystallized wax increase rapidly. This worsens the flowability of the oil. Visual inspection shows that wax crystals in the micrographs of doped oil are smaller in size relative to the neat oil. The microscopic properties of wax crystals, computed with image analysis software, enable objective, quantitative comparison of the effect of the addition of pour point depressant on crystal morphology and micro-structure (Table 4).

Table 4. Microscopic properties of neat and doped crude oil

Temp.	Sample	Microscopic parameters			
		Aspect ratio	Solidity	Circularity	Boundary fractal dimension
20°C	Neat oil	1.41	0.88	0.90	0.89
	Doped oil	1.38	0.89	0.93	0.67
10°C	Neat oil	1.72	0.85	0.83	1.40
	Doped oil	1.60	0.86	0.85	1.41

Aspect ratio and circularity are morphology descriptors, while solidity and boundary fractal dimension provide a description of wax microstructure. Increasing aspect ratio (major axis/minor axis of wax crystal) indicates elongation of wax crystals and rod-like crystal shape, where an aspect ratio of 1 signifies a perfectly round crystal. Circularity, which is dependent on the crystal perimeter, may be used as a measure of the degree of regularity in the shape of the wax crystal. Circularity value tending towards the maximum value of 1.0 denotes a perfect circle and implies higher regularity of crystals. Crystal solidity is the ratio of the area of the crystal to its convex hull area. It provides a measure of the evenness of the crystal surface. Decreasing solidity value indicates that the surface of the wax structures are more ruffled and uneven, while increasing boundary fractal dimension indicates increasing roughness of wax crystal edges. Cooling of oil results in increasing crystal aspect ratio and boundary fractal dimension and decreasing solidity and circularity (Table 4), meaning that wax structures become elongated and rod-like in shape, exhibiting greater irregularity, uneven surfaces, and rough edges at lower temperatures.

The crystal size distribution presented in Fig. 3 shows the increase in wax crystal size due to crystal growth as the oil cools. These changes in the morphology and microstructure of wax as the oil cools are detrimental to oil flowability because larger, longer and rougher crystals in oil tend to overlap with each other and interlock more effectively as the roughened surfaces provide crystal anchor points [31]. The oil eventually gels as the network of interlocked wax becomes larger and stronger with decreasing temperature.

Addition of the CNSL derivative results in a reduction in aspect ratio and boundary fractal dimension and an increase in crystal circularity and solidity, which indicates that the crystal shape is rounder and more regular, and the surfaces of the wax structures are more even. This is buttressed by the size, aspect ratio, and solidity distributions of wax crystals shown in Figs.3-5. The slight increase in boundary fractal dimension of doped oil relative to neat oil at 10°C is due to the formation of a much higher number of smaller crystals in doped oil (Table 4; Fig.2).

It is observed from Fig.3 that the size of wax crystals in doped oil was reduced, resulting in an increasing proportion of smaller crystals, mainly in the 10-30 $\mu\text{m}^2$  range, and fewer large-sized crystals (<100 $\mu\text{m}^2$ ). Similarly, the aspect ratio distribution shifts to the left, favoring perfectly round crystals at the expense of rod-like crystals (Fig.4). The solidity distribution at 20°C shows that crystals of low solidity ( $\geq 0.55$ ) do not exist in the doped oil; rather, there is an increase in the proportion of high solidity crystals, especially in the 0.9-1.0 range. The increasing proportion of low solidity crystals is expected at 10°C as the crystals become more ruffled. However, in doped oil, the distribution shifts to the right towards high solidity (0.8-1.0) as the proportion of even-surface crystals increases (Fig. 5)

These observations explain the reduction in pour point of crude oil upon addition of CN<sub>1</sub>EG as the smaller, rounder, and smoother wax crystals in the doped oil cannot overlap and interlock

effectively, inevitably forming weaker wax networks. Hence the doped oil can still pour at much lower temperatures than the neat oil.

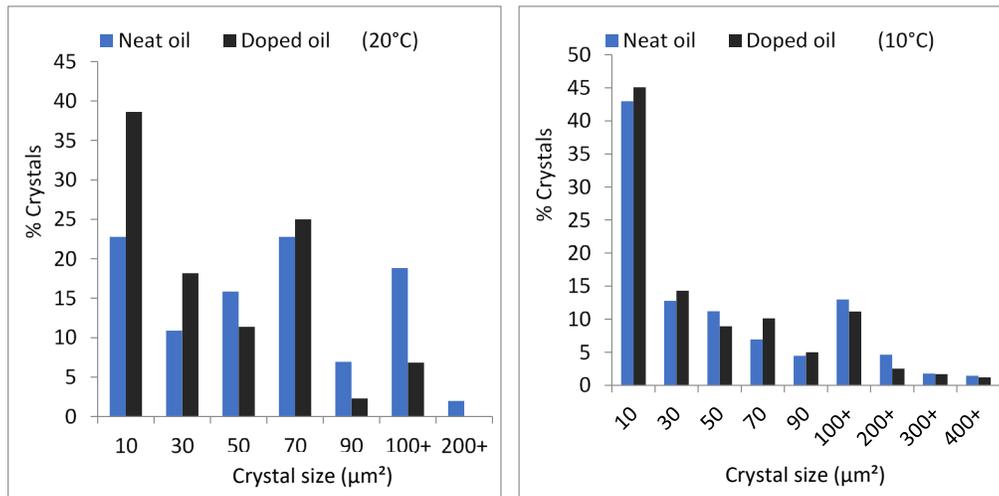


Figure 3. Effect of additive on the wax crystal size distribution

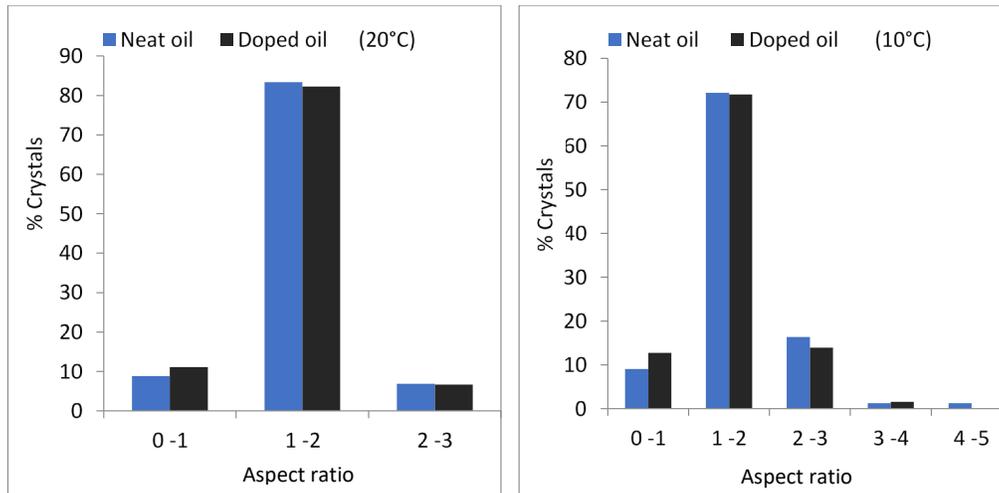


Figure 4. Effect of additive on wax crystal aspect ratio distribution

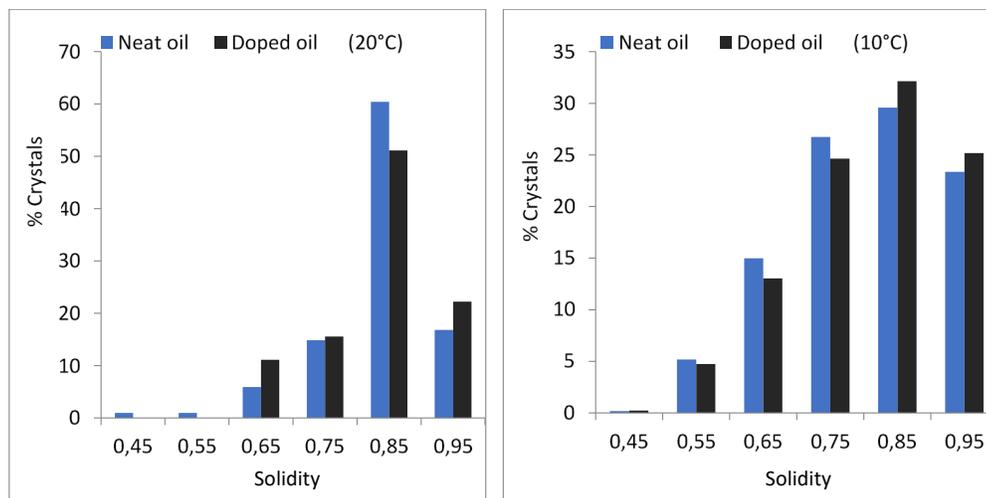


Figure 5. Effect of additive on wax crystal solidity distribution

The microscopic data suggest that the additive molecules interact with waxes in crude oil in a manner that hinders the wax agglomeration process. It is speculated that the additive molecules may adsorb onto incipient wax crystals via interactions between paraffins and the aliphatic chains of CNSL. The high polarity of the phenol, hydroxyl, and ester groups of the additive molecule may induce electrostatic repulsion between small wax particles onto which the additive is adsorbed. Such a mechanism will improve the dispersion of small wax crystals and hinder their agglomeration into larger more complex wax structures.

The foregoing suggests that the decline in pour point depression efficiency above the optimal dosage is likely due to the presence of large number of free (unadsorbed) additive molecules in the oil at these dosages. These excess additive molecules may act as bridges linking the wax crystals that were originally dispersed in the oil.

### 3.3.3. Effect on flow properties

The effect of the CNSL derivative on the shear stress and viscosity of crude oil at temperatures above and below the oil WAT is shown in Figs. 6–8. At 50°C, the oil temperature is far above its WAT, and waxes still exist in solution in the oil. The shear stress and viscosity of the doped oil are slightly higher than the neat oil. This suggests that in the absence of crystallized wax, which the additive molecules interact with, the presence of additive has a slight viscofying effect on the oil. As the temperature of the oil drops to 20°C and wax crystallizes, the shear stress and viscosity of the doped oil is reduced relative to the neat oil. The degree of reduction in the values of oil flow properties is dependent on shear rate, showing an increase at higher shear rates. Shear stress and viscosity reduction, taken as the average across the experimental shear rate, was 4.41% and 4.03%, respectively.

At 10°C, large amount of wax has precipitated out of the oil (Fig. 2). Consequently, the shear stress of neat oil increases rapidly with increasing shear rate due to the high resistance of oil to shear (Fig. 6). The average viscosity of the neat oil increases fourfold from its value at 20°C (Fig. 7). On doping with the additive, oil shear stress and viscosity were reduced by 47.8%. The viscosity-temperature profile of the oil at the constant shear rate (51/s) also shows that the presence of additive impedes the rapid increases in viscosity that occurs from the inflection point of the curve (Fig. 8). The inflection point indicates the onset of wax crystallization in the oil and worsening oil flowability as temperature decreases further below WAT. The viscosity of neat crude oil increases by approximately 16.4cP (centipoise) for every degree decrease in temperature compared to 6.5cP in doped oil.

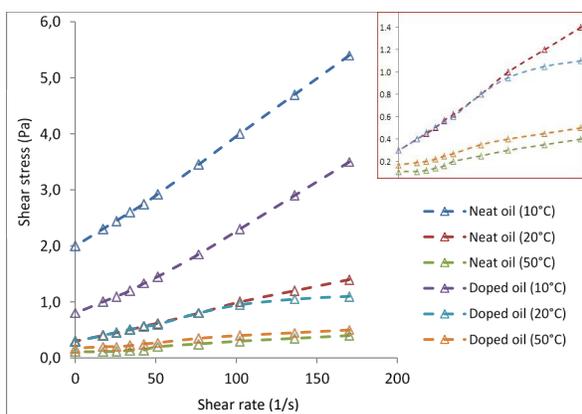


Figure 6. Effect of shear rate on shear stress of neat and doped oil

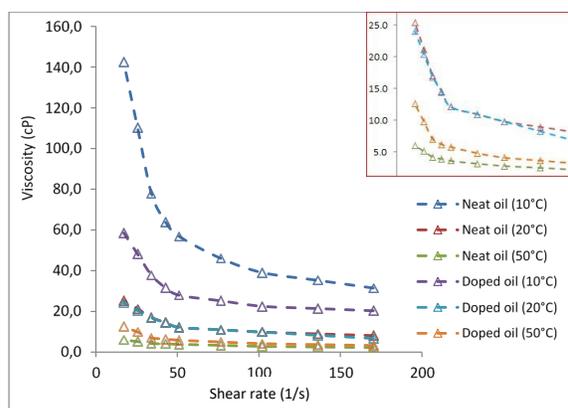


Figure 7. Effect of shear rate on viscosity of neat and doped oil

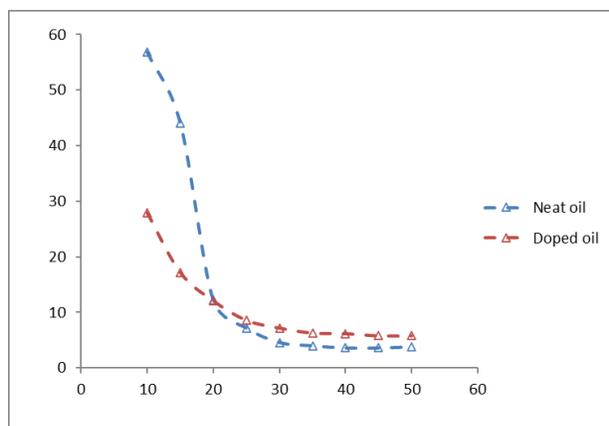


Figure 8. Effect of temperature on viscosity of neat and doped oil

The improvements in flow properties of the oil are attributable to the changes in morphology and microstructure of wax crystals in doped oil. Smaller, round crystals with smooth surfaces likely create less internal friction within the flowing oil and offer less resistance to shear due to inability to network effectively. The aromatic group and hydrocarbon chain of the additive molecule may also solubilize waxes in oil, especially under shear, thereby increasing oil flowability.

#### 4. Conclusions

Natural cashew nut shell liquid was extracted, esterified with ethylene glycol, and evaluated as pour point depressant and flow improver for waxy crude oil. The addition of the CNSL-ethylene glycol ester to waxy crude resulted in depression in oil pour point and modification of wax crystal morphology and microstructure. In the presence of the additive, the size, aspect ratio, and boundary fractal dimension of wax crystals were reduced while the circularity and solidity increased as smaller, rounder, and more regular crystals with smoother, even surfaces were formed. Changes in the microscopic properties of the crude oil are due to wax-additive molecule interactions, which are mainly via adsorption and dispersion, to the effect that the additive hinders the normal wax agglomeration process. The depression in pour point and improvement in low-temperature flow properties of the waxy oil originates from the changes in wax crystal morphology and microstructure, which weakens the wax network, reduces the internal friction within the oil, and lowers the resistance of the oil to shear. The CNSL derivatives are promising low-cost solutions to wax problems in production, storage, and long-distance pipeline transport of waxy crude oil.

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