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Evaluation of silane-based surfactant nanohybrid as flow improver in a Malaysian light crude oil

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Abstract. Paraffin deposition in the crude oil production pipeline has been an alarming problem to the flow assurance community. This phenomenon causes a tremendous amount of material loss in the production and substantial resources are expended to resolve these flow assurance problems—which included the chemical treatment. This study examined an agricultural non-ionic silane-based surfactant and its blends (with silica nanoparticles) as a flow improver using Malaysian light crude oil (42.4°API). In particular, this study performed the following experimental measurements: wax appearance temperature, pour point, viscosity, and FTIR spectroscopic analysis. The result showed that the surfactant-nanoparticles blend affected the viscosity (significant reduction by approximately 67 %) within certain temperature range and were able to depress both pour point (to 4°C) and wax appearance temperature (15.6°C). It was also revealed that the most potent blend consisted 400 ppm of silane-based surfactant and 200 ppm of SiO_2 nanoparticles. The study also evaluated the underlying mechanisms for the variation of viscosity through FTIR spectroscopic analysis.

1. Introduction

The precipitation and deposition of waxes in the pipelines during the transportation of waxy crude oils contribute to several issues such as the flowability reduction, excessive pumping cost, and wax gel formation, which adversely affect the steady offshore oil production [1-2]. When the temperature of crude oil hits below the wax appearance temperature (WAT), these paraffin waxes precipitate from the crude oil and subsequently form "house-of-cards" structure—overlapping and interlocking of these wax crystals that develop volume-spanning crystal network [3]. This crystal network entraps the liquid oil in a cage-like structure, which increases the pour point and viscosity of the crude oil; thus, reducing the flow properties of crude oil [4].

Chala *et. al.* [1] recently revealed that the oil producers have started to extend their production to the offshore and deep-water region, which mean the production has explored deeper into the risky ultra-deep-water region. Consequently, this reduces the flow assurance in the subsea region of colder environment (at least 5˚C) with higher distance of offshore oil rigs, which leads to critical wax deposition problem [5]. As of January 2018, there are approximately 600 offshore wells worldwide, of which the North Sea (184 rigs) and the Gulf of Mexico (175 rigs) are the base to most of these offshore rigs with possibly the deepest well of as deep as 3,000 m below sea level [6]. To date, the South China Sea, the Gulf of Mexico, North Sea, and Persian Gulf are amongst the most active offshore well productions. Meanwhile, in Malaysia, the oil wells reportedly contained as high as 20 wt% of wax content and pour point temperature of as high as 36°C [7]. Oil fields, such as Penara, Angsi, and Dulang,

encounter critical wax deposition problem. These oil fields with a water depth of between 60 m and 70 m have surface temperature of 34°C and average seabed temperature of about 25°C at the depth of 61 m [8-9]. The wax deposition in the inner walls of production and transportation pipelines and on the surface of equipment were reportedly the prevalent cause to the flow assurance problem [7, 10-12]. In the worst-case scenario, the accumulation of deposited wax causes a complete blockage in the pipeline.

Addressing the flow assurance issues, chemical method which serves as preventive measure is considered as a significant practical application in the crude oil industry. Various wax inhibitors (WIs), polymeric pour point depressants (PPDs), and flow improvers (FIs) have been applied to enhance the flowability of waxy crude oils [13-15]. The heating method and the addition of light ends or organic solvents downhole and to the flowlines were formerly applied, which effectively remove the deposited waxes. With these methods, the precipitated waxes are dissolved, which eventually ease the transportation of crude oils to the surface. However, these methods are costly and highly dependent on feedstocks. Thus, these methods are only recommended if there are inexpensive and large amount of light ends or solvents available in the oil field.

Ethylene vinyl acetate (EVA) copolymers and comb-type copolymers are the most common types of WIs and PPDs with promising results over the past decade [16]. These WIs and PPDs reduce the pour point and subsequently delay the gelation rate of crude oils—as the temperature decreases, the flowability improves [17]. However, it is a challenging process to decompose these chemicals in the refinery system due to their high molecular weight, long polymer chain, and high thermostability; thus, making them costly and not environmentally friendly [15].

With that, this study considered a cost-effective and environmentally friendly surfactant as a clean flow improver (FI)—non-ionic silane-based surfactant. This surfactant is widely used in the agriculture sector due to its biodegradability, low toxicity, and rapid spreading characteristic. More specifically, this study assessed the surfactant individually and in mixture (with silica nanoparticles, SiO_2-NPs) as potential FIs using light crude oil of 42.4°API. Furthermore, the surfactant blend (nanohybrid) were found to be inadequately explored in the past, hence served as the objective of this study to extend the existing knowledge base on the role of surfactants and surfactant nanohybrids in improving the overall flowability of crude oils. The efficiencies of these FIs were also compared based on several parameters, such as WAT, pour point, and viscosity. This study then explained the possible mechanism of FI using Fourier-transform infrared (FTIR) spectroscopy.

2. Experimental section

2.1. Materials

This study selected a non-ionic polyether modified trisiloxane surfactant with (3-(2-methoxyethoxy)propyl-methyl-bis(trimethylsilyloxy)silane) as the primary surfactant, which was coded as S3 hereinafter. It was screened a potential as WI for the candidate oil [18]. Figure 1 presents the chemical structure of S3.
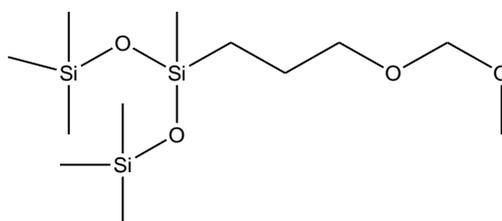

Figure 1 Chemical structure of S3.

Meanwhile, SiO_2-NPs supplied for the synergistic study of the surfactant and NPs have varying particle sizes with a main average particle size of 10–15 nm, which was measured by X-ray diffraction. Besides that, the crude oil sample was generously provided by PETRONAS Penapisan Terengganu. The wax appearance temperature, pour point, viscosity, API gravity, surface tension, wax and asphaltenes

content of the crude oil used are 21.9°C, 13°C, 22 mPa.s, 42.4°, 23 mN/m, 8.22% and 0.65%, respectively.

2.2. Preparation of surfactants, SiO_2 nanofluids, and surfactant blends

This study fixed the initial concentration of S3 and SiO_2 nanofluids at 400 ppm. Subsequently, 0.8 mL (or the equivalent amount) of surfactant was dissolved in 100 mL of deionized water in the preparation of diluted surfactants (400 ppm). The solution was then stirred at 700 rpm for one hour to form a stable homogenous solution. Following that, 40 mg of SiO_2-NPs were weighed and mixed with 100 mL of xylene (which was used as the base fluid) for the preparation of 400 ppm nanofluids. The mixture was later heated up to 80°C and stirred at 700 rpm for one hour, followed by ultra-sonication treatment (Ultrasonic bath FB15051, Fisherbrand) for another 30 minutes at 25°C. This study prepared the surfactant blends (SN3) by mixing 3 wt% of diluted surfactants (400 ppm) and 1 wt% of nanofluids (400 ppm). The weight percentage (wt%) of chemicals was measured based on the weight of crude oil sample used in each experiment [18].

2.3. Measurement of wax appearance temperature

This study measured WAT using differential scanning calorimetry (DSC) (Q1000, TA Instruments) as per the ASTM D2500 standard. The crucible was heated with a constant temperature of 80°C for one minute before it was cooled to −20°C at 5°C/min. When the temperature of crude oil is below WAT, wax crystals are formed, which produce a small amount of heat and therefore a slight increase in the temperature of the sample.

2.4. Measurement of pour point

This study added 3 wt% of diluted surfactants and 1 wt% of nanofluids into 40 mL of crude oil in the pour point designated test jar. The pour point was measured using the digital pour point test apparatus (K46100, Koehler) as per the ASTM D97 method.

2.5. Rheological measurement

This study measured the dynamic viscosity using a programmable rheometer (DV-III, Brookfield) for the rheological measurement. Approximately 10 mL of the sample was introduced in the cup of holder of the viscometer priory cooled (or heated). The viscosity was then measured at various shear rate (35 s^{-1}, 85 s^{-1}, and 135 s^{-1}) and temperature (10°C, 20°C, 30°C, 40°C and 50°C). As suggested by Naiya et al [19], the degree of viscosity reduction (DVR) in this study was evaluated based on Equation (1):

$$DVR\% = \frac{(Nr - Nc)*100}{Nr} \tag{1}$$

Where Nr denotes the reference viscosity (blank crude oil at 35s^{-1}); Nc denotes the viscosity at the corresponding shear rate (of 35s^{-1}) and the temperature (of 10°C).

3. Results and discussion

3.1. Wax appearance temperature and pour point analysis

Principally, WAT is the temperature at which the crystallization first appears in crude oil, while pour point is the temperature at which the gelation of crude oil occurs and losses its flow characteristics. Both WAT and pour point are the key parameters to examine the flowability of crude oil. Table 1 shows the results of measured WAT and pour point for the blank and treated samples, which revealed that WAT slightly increased with the sole use of S3. Meanwhile, the pour point of S3 treated sample was reduced from 13°C to 7°C, with the difference of 6°C. Similar trend was observed with the SN3 treated sample, which was further depressed to the lowest (5°C). Adding to that, SN3 treated sample was also capable to depress WAT with the highest reduction of 2.8°C. These results proved that the addition of SiO_2-NPs

inhibits the wax crystallization, which deems this particular addition to be the most effective in reducing both WAT and pour point.

Table 1. Effect of the flow improvers on the wax appearance temperature and pour point of crude oil samples.

Sample	WAT (°C)	Pour point (°C)	ΔWAT (°C)	Δ Pour point (°C)
Crude oil	21.9	13	-	-
Crude oil + S3	23.3	7	1.4	6
Crude oil + SN3	19.1	5	2.8	8

3.2. Temperature and shear effect on rheological properties of crude oil
Nonetheless, the pour point measurement alone does not describe the flowability of crude oil, which explains the need to perform the rheological studies in establishing a realistic flow behaviour for the tested crude oil. Table 2 reveals the viscosity of blank and treated samples while Figure 2 and Figure 3 show the trend of the viscosity behaviour according to the shear rate (35–135 s^{-1}) and temperature (10–50°C).

**Table 2.** Effect of shear rate and temperature on the viscosity of crude oil samples.

Sample	Shear rate (s^{-1})	Apparent viscosity (mPa.s) at different temperature				
		10°C	20°C	30°C	40°C	50°C
Crude oil	35	64.0	22.0	7.50	4.50	3.00
	85	51.0	20.4	6.40	2.67	2.31
	135	42.0	18.5	5.75	2.30	1.80
Crude oil + S3	35	31.5	21.0	7.10	3.00	3.00
	85	25.8	16.8	5.80	2.40	2.34
	135	23.6	14.2	5.25	2.25	1.80
Crude oil + SN3	35	27.1	16.5	6.50	3.00	3.00
	85	24.2	15.3	5.60	2.25	1.80
	135	22.9	13.5	4.37	1.87	1.50
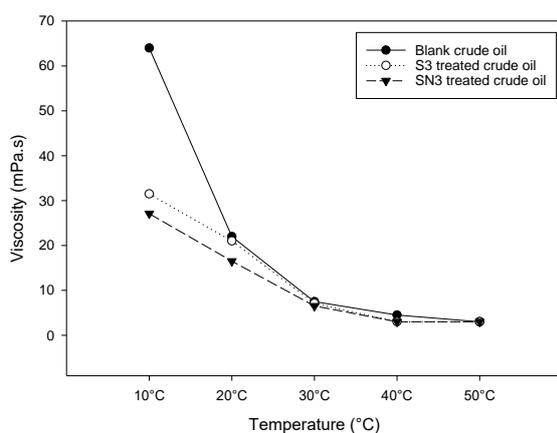
(a) Shear rate 35 s^{-1} (b) Shear rate 85 s^{-1}
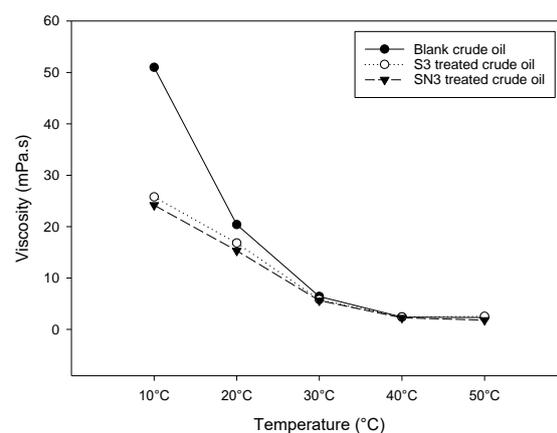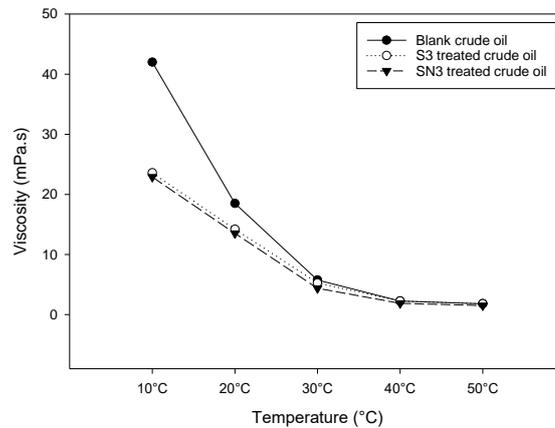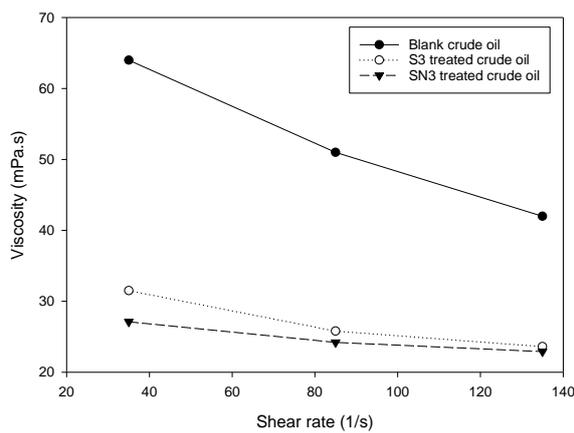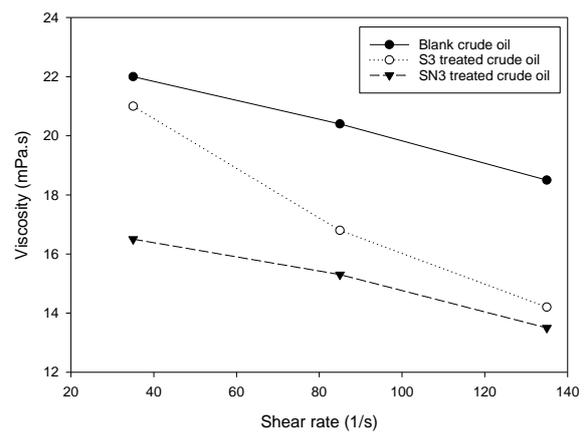
(c) Shear rate 135 s^{-1}

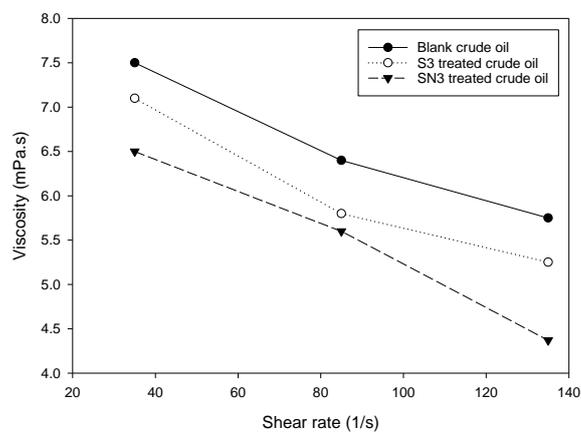**Figure 2.** Viscosity behaviour of crude oil against temperature at: a) Shear rate 35 s^{-1}, b) Shear rate 85 s^{-1}, c) Shear rate 135 s^{-1}.

(a) Temperature 10°C

(b) Temperature 20°C

(c) Temperature 30°C

Figure 3. Viscosity behaviour of crude oil against shear rate at: a) temperature 10°C, b) temperature 20°C, c) temperature 30°C.

Figure 2 shows the viscosity of each crude oil sample was relatively lower at the temperature beyond 40°C, but progressively increased when the temperature approached WAT and pour point, regardless of various shear rate. In other words, when the temperature of crude oil exceeds 40°C, the rheological properties of all tested samples remain rather similar. With the increasing temperature, the components of the crude oil with high molecular weight (such as asphaltenes, resins, and even waxes) are not able to agglomerate and form aggregates, which eventually affects the bonds between these solid particles and subsequently reduces the oil viscosity. Thus, this implies that the temperature predominantly affects the chemical structure and viscosity of these components of crude oil with high molecular weight.

Meanwhile, treated with only S3, the apparent viscosity of blank crude oil experienced reduction from 64 mPa.s to 31.5 mPa.s (DVR of 51%) at 10°C. The addition of surfactant in the crude oil tends to reduce the entanglements of long-chain hydrocarbons, forming a thin film over the wax particles; thus, preventing them from agglomerating to larger wax crystals [20]. The depressing effect of viscosity for SN3 treated sample was also evident. With the inclusion of SiO_2-NPs, the SN3 treated sample demonstrated superior depressing effect of viscosity, which exhibited higher DVR of 58% at the shear rate of 35 s^{-1}. The depression of both WAT and pour point were shown to be in complete agreement with the depressing effect of viscosity for SN3 treated sample. This could be due to their unique surface adsorption effect and small size which favour the dispersion of wax crystals [21].

Apart from that, the results also showed that the viscosity of every sample substantially decreased with the increasing shear rate, particularly when the temperature approached pour point (Figure 3). In fact, this demonstrates non-Newtonian shear thinning behaviour. Specifically, this particular behaviour is due to the energy exerted by shear, in which the dissipated energy in the matrix tends to partially break down the wax crystals at a lower shear rate. However, with flowing of higher dissipated energy (at higher shear rate) that overcomes the yield stress, the viscosity of crude oil decreases with the increasing shear rate until these agglomerates are broken down into basic particles [22]. At this point, the process deforms the crude gel structure and reduces the size of the agglomerates.

3.3. Effects of the concentration of blended surfactant on wax appearance temperature, pour point, and viscosity
Overall, the SN3 treated sample exhibited the most significant (positive) alteration in terms of WAT, pour point, and viscosity in this study. In order to grasp the obtained results, the synergistic effects of the surfactant and NPs in forming emulsion were evaluated. The emulsion, in this case, serves as the stabilizer for wax crystals. As tabulated in Table 3, different concentrations of surfactant and nanoparticles (200 ppm, 400 ppm, and 1000 ppm) in SN3 blend were determined for the evaluation. Among these five different blends, the blend with the lowest concentration of SiO_2-NPs (200 ppm) demonstrated the most notable changes in the reduction of WAT and pour point. Both WAT (15.6°C) and pour point (4°C) were evidently the lowest with the incorporation of 400 ppm of S3 and 200 ppm of SiO_2-NPs. Thus, this particular combination of SN3 is suggested to enhance flowability of crude oil.

Table 3. Influence of five different concentrations of blended surfactant on wax appearance temperature and pour point of crude oil.

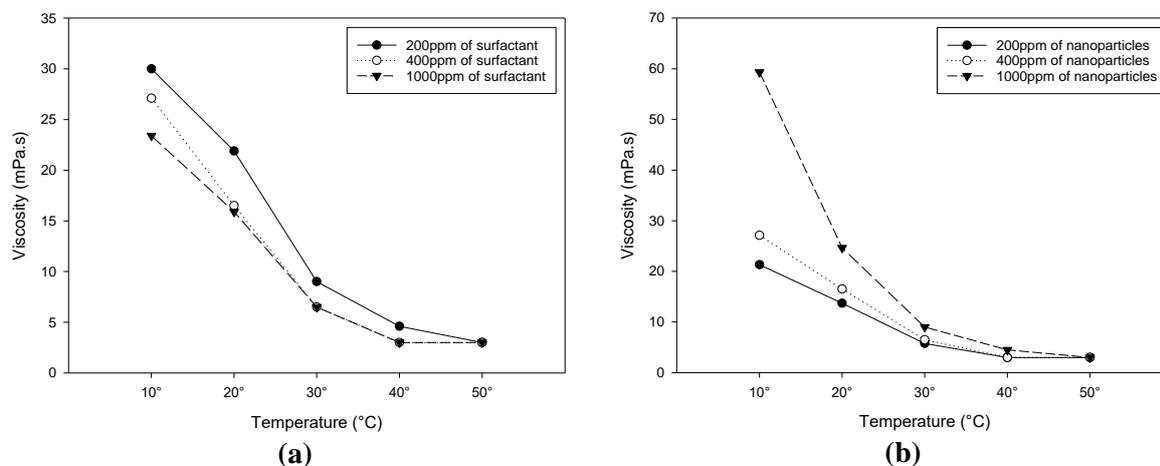| Concentration of SN3 blend (ppm)							
Surfactant	SiO_2-NPs	WAT	Pour point	SiO_2-NPs	Surfactant	WAT	Pour point
200	400	19.7	9	200	400	15.6	4
400	400	19.1	5	400	400	19.1	5
1000	400	18.3	8	1000	400	20.3	9

Figure 4. Effects of the concentration of surfactant (a) and nanoparticles (b) in SN3 blend on the viscosity of crude oil at different temperatures.

As presented in Figure 4 (a), the viscosity was shown to decrease with the increasing concentration of surfactant in SN3. In this case, with the increasing concentration of surfactant, the stability of the emulsion increases, as well. Consequently, the surfactant molecules have higher coverage, which enhances their adsorption to these wax crystals; thus, the reduction in viscosity. Meanwhile, referring to Figure 4 (b), the results of rheological measurement aligned with the results of WAT and pour point reduction across different concentration of NPs. Likewise, the blend with the lowest concentration of NPs (200 ppm) demonstrated the lowest viscosity (21.3 mPa.s), which offered the highest DVR (67%). The SiO_2-NPs, as the additional suspended particles in liquid, reduces the flowability of crude oil which explains the poor performance at high concentration of NPs (1000 ppm) in this study [18,23]. In this case, the increase in the number of silica sites for the wax crystallization is plausible.

These results illustrate different synergistic effects in the blended surfactants, which reaffirm the influence of concentration (surfactant and NPs) in the blend on the wax inhibition. The concentration of FI may also influences the van der Waals interaction within these wax crystals, which predominantly determines the solubility of these waxes in crude oil [24-25]. In general, the optimum concentration of FI is well correlated with the improved of WAT, PP, and viscosity in this study.

3.4. Fourier-transform infrared spectroscopy
This study subsequently performed FTIR spectroscopy to determine the presence of functional groups (that predominantly affect flowability) in these samples. The FTIR spectra of blank crude oil as well as its mixture with different FIs are displayed in Figure 5. The display of characteristic absorption bands at certain dominant peaks, such as the asymmetrical vibration (at 1040 cm[-1]), the stretching vibration (at 1105 cm[-1]), and the C-H group stretching vibration (at 1251 cm[-1]), revealed the presence of Si-O-Si backbone in the molecular structure of S3 treated sample [14] [26-27]. Meanwhile, the peak at 840 cm[-1] represents the Si-C stretching vibration [28]. The characteristics of these bands correspond to the functional group in silane-based surfactant, which are responsible for the variation of flowability of the candidate oil.
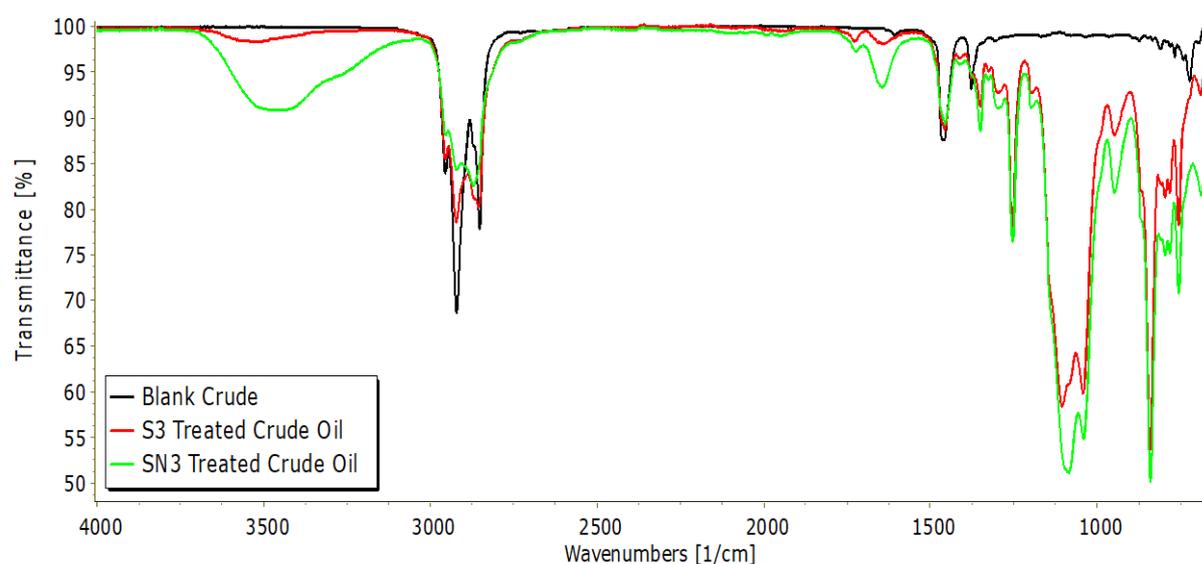
Figure 5. Comparison on FTIR spectra of blank crude oil, S3 treated crude oil, and SN3 treated crude oil.

The blank sample and S3 treated sample revealed significant reduction in the intensity of the absorption peaks for the fingerprint region (C-C bond, C-O bond, and C-N bond), which possibly indicates the distinct effect of silane-based surfactant to disintegrate these bonds. As implied by Naiya et al [19], Banerjee et al [29], and Coto et al [30], the breaking of C-C bond in crude oil (by the surfactant) subsequently reduces the viscosity of the crude oil. Furthermore, the presence of oxygen atoms in the polyethylene oxide (PEO) side chains of the silane-based surfactant increases the wax solubility (inhibits the wax growth) and eventually enhances flowability of the crude oil [31].

Apart from that, the comparison of FTIR spectrum of S3 treated sample and SN3 treated sample showed evident stretching at the peak of approximately $3472cm^{-1}$, which could be derived from the vibration of hydroxyl bond (O-H groups) of SiO_2-NPs [32]. The intensity of the absorption peak of the O-H bond in SN3 treated sample decreased at $3472cm^{-1}$ (weakening of O-H groups), which reduces the viscosity of crude oil [33]. In addition, the previously identified dominants peaks in S3 treated sample also demonstrated reduction with the addition of SiO_2-NPs.

4. Conclusion

This study demonstrated the effects of the surfactant and NPs towards WAT, pour point, and viscosity. The silane-based surfactant and SiO_2-NPs were evaluated (individually and in mixture) as FIs, which successfully enhanced the overall flowability of the Malaysian light crude oil (42.4° API). The addition of 400 ppm silane-based surfactant alone successfully reduced the viscosity of the crude oil by 51%. The addition of 400 ppm SiO_2-NPs in the blended surfactant (SN3) resulted to superior flowability performance (DVR of 58%) of the crude oil. Furthermore, the combination of 400 ppm of silane-based surfactant and 200 ppm of SiO_2 NPs in SN3 blend exhibited the highest efficiency to depress both WAT (to 15.6°C) and PP (to 4°C). This particular combination also offered the highest DVR (67%) at the temperature of 10°C and the shear rate of 35 s⁻¹. Following the addition of S3 and SN3, the fall in the band intensity of C-C group, C-O group, and C-N group of crude oil (wavelength $< 1400cm^{-1}$) and O-H group ($3472 cm^{-1}$) indicated that these groups are responsible for the variation in the viscosity of the crude oil. Generally, there are substantial studies on polymeric WIs. Thus, future research should focus on the development of green products for flow assurance, such as the agriculture surfactants and the bio-surfactants (which are synthesized from plants). After all, the aforementioned surfactants are non-toxic in nature with lower extraction cost and the ability to recover in the refining process. Thus, the inclusion of surfactants (with NPs) to replace polymers provides an added advantage as a multipurpose specialty chemical in the crude oil industry. These surfactants can serve as demulsifier, dispersant, inhibitor, or FI at lower cost. Finally, the resultant outcomes of this study are expected to contribute to the petroleum

industry based in the region of relatively warmer sea temperature, such as the Southeast Asia region, with the identification of this specific blend, which offers immense potential as FI.

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