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Application of Bio Additive for Flow Assurance of Indian Waxy Crude Oil

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ABSTRACT

Wax deposition in pipelines during production, storage, and transportation is a very serious issue in the upstream petroleum industry. Lots of money and time need to be invested by the petroleum industries to restart the flow. The present study deals with the application of alkyd resin as bio-additive for flow assurance of waxy crude oil. Epoxidized Jatropa oil was co-polymerized with phthalic anhydride to prepare alkyd resin. FTIR and NMR spectrum analysis were performed to characterize the bio-additive JOAR. After dosing with alkyd resin, the pour point of Indian crude oil was decreased from 33°C to 24°C, and it also improves the flowability of crude oil by reducing its rheological properties including viscosity, thixotropic properties, etc. A significant reduction of pour point and viscosity was observed after the addition of 400 ppm bio-additive. Moreover, this bio-additive is biodegradable, eco-friendly, non-toxic, and cost-effective compared to commercial flow improver.

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Received: April 05, 2024; **Accepted:** April 11, 2024; **Published:** April 29, 2024

Keywords: Crude Oil, Bio Additive, Flow Assurance, PPD, Alkyd Resin

Introduction

In the petroleum industry, precipitation of wax in crude oil at lower temperature regions during its production as well as transportation through pipelines is a serious flow assurance problem. When the temperature of crude oil falls below wax appearance temperature (WAT), wax crystallization starts and with time it is agglomerated [1, 2]. The wax crystals start to deposit on the pipeline surface and resist the flow ability [3]. There are various methods of wax mitigation are extensively used by petroleum industries like scrapping off the deposited wax bounded of electrical wire on the pipeline surface, installation of infrared or radiant heaters, coating with insulating materials, circulating hot oil or water, placing stream tracing systems inside the pipeline are some of the conventional techniques are used. In addition to this, solvents like xylene, and toluene are used to dissolve wax. However, pigs can get stuck in the pipelines, and during wax removal wells are shut off for 2 to 10 days which greatly impacts the economic system [4]. For that reason, nowadays most of the industries prefer polymeric additives, known as pour point depressants (PPDs) or wax inhibitors to get rid of these wax deposition issues [5]. The efficiency of pour point depressants on waxy crude oil depends on various properties of crude oil such as wax content, carbon number distribution of wax, asphaltene, and resin content, etc [6,7].

It is generally considered that molecules of PPDs are also co-precipitated along with wax molecules and form a thin coating over them so that, wax particles cannot come close to each other and prevent the agglomeration of wax [8]. Consequently, the pour

point of crude oil is significantly reduced which in turn improves the rheological properties of crude oil. Commercial PPDs are usually employed to decrease wax deposition and pour point control. Polymeric compounds, often fall into one of the following three major chemical families, co-polymers of Maleic anhydride, co-polymers of ethylene vinyl acetate, and polyacrylate copolymers [6-11]. The major limitations of the PPDs derived from the above-mentioned chemicals are that these are costly and most importantly, their toxic chemical composition disturbs the ecological balance. Once PPD is mixed with crude oil, it is almost impossible to remove it, which can result in changes in the properties of crude oil. Due to these synthesized and commercial PPDs limitations, their use is decreasing, which raises the need for environmentally benign, economically viable, and sustainable green PPDs to ensure the flow of waxy crude oil. Additionally, using natural PPDs has several other advantages, including cost-effectiveness, biodegradability, non-toxicity, and environmental friendliness. Thus, this greatly opens up a new route for doing intuitive research on the synthesis of green PPDs.

To address the issue of wax deposition and enhance the flow of crude oil, coconut oil ethyl ester (COEE) was synthesized [12]. With an incredibly low concentration of 800 ppm, this biodegradable PPD was able to lower the pour point by 12°C. With this bio-additive, crude oil's viscosity was reduced by 94% and interfacial tension by 19%, respectively. Hence, the flowability of crude oil is greatly improved. Biodiesel derived from linseed oil can effectively increase the flow of Nigerian waxy crude oil. Nigerian crude oil's initial pour point was 23°C, it dropped to nearly 8°C after the addition of 0.1 V/V concentration of additive [13]. produced biodiesel using sunflower oil and studied its performance concerning the flow of

Nigerian waxy crude oil [14].

In this work, an alkyd resin was synthesized from jatropha oil, initially, epoxidation of jatropha oil with peracetic acid was carried out followed by alcoholysis with methanol and polyesterification with phthalic anhydride. Alkyd resin is a polyester that has a variety of applications as coating material to protect metal, wood, or concrete from weather and natural wear & tear. No work has been yet shown alkyd resin as a pour point depressant. Being a polyester, alkyd resin derived from jatropha oil shows a good performance in lowering the pour point as well as the rheological properties of Indian waxy crude oil. Moreover, its cost-effectiveness and ecofriendliness nature provide an additional benefit.

Materials and Methodology

Materials

The Jatropha oil, Sunflower, and sesame oils, Sulphuric acid, acetic acid, hydrogen peroxide, glycerol, sodium hydroxide pellets, methanol, and Phthalic anhydride were procured from Ranken Chemicals, Mumbai, India. The crude oils were collected from the Gamiz oil fields, Mehsana asset, Ahmedabad, ONGC. Toluene, methanol, acetonitrile, chloroform, and n-heptane were procured from Merk Life Sciences, Mumbai, India. All the reagents were used without any further purification.

Methods

Experimental Procedure of Jatropha Oil Alkyd Resin Synthesis

Epoxidation of Jatropha Oil

300gm of Jatropha oil was taken in a three-necked round bottom flask which is fixed with the reflux condenser, fitted in a water bath, and arranged with the mechanical stirrer. The oil in the flask was allowed to stand for 5 minutes by maintaining the temperature at 40°C with an agitation speed of 500 rpm. Then, 0.1% v/w (oil weight) of conc. sulphuric acid, and peracetic acid (mixture of acetic acid and hydrogen peroxide) were added dropwise into the flask [15]. The epoxidation reaction was allowed to stand for 6 hours at 55°C temperature and 500rpm.

Preparation Of Alkyd Polymer from Jatropha Oil Alcoholysis

After completing the reaction, 300 g of epoxidized Jatropha oil was again reacted with 70 g of glycerol in a reactor where 0.2% NaOH (w/w) was used as a catalyst. The reaction was carried out at room temperature to form monoglycerides [16]. After 30 minutes, the temperature of the reacted oil was increased up to 250°C in an

inert atmosphere. The reaction was carried until an aliquot became miscible by adding with 1:3 (v/v) ratio of anhydrous methanol and stored at 140°C.

Polyesterification

The polyester alkyd resin was prepared by reacting 95gm of phthalic anhydride with the prepared monoglycerides by maintaining the temperature at 250°C in the presence of N₂ atmosphere. Figure 1. Describes the flow chart of JOAR synthesis.

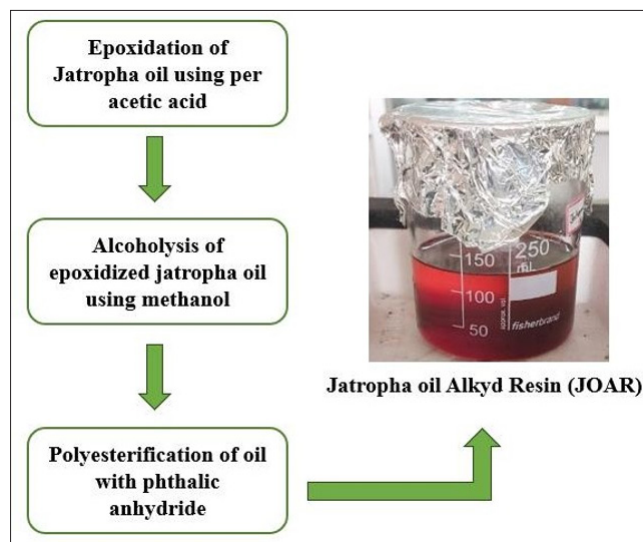


Figure 1: Flow Chart of Alkyd Resin Synthesis Derived from Jatropha Oil (Joar)

Characterization of Crude Oil

ASTM standard methods were followed to determine the specific gravity, API gravity, pour point, wax content, water content, and SARA analysis. Specific gravity was determined according to ASTM D 1298-12b (2017) with the hydrometer. API gravity was calculated from the value of specific gravity. Water content was measured in the Dean-Stark apparatus as per ASTM D4006-16e1 (2016) standard. The universal oil product (UOP) 46-85 method was followed to measure the wax content of crude oil. SARA analysis was performed to know the percentages of saturate, aromatic, resin, and asphaltene content of crude oil.

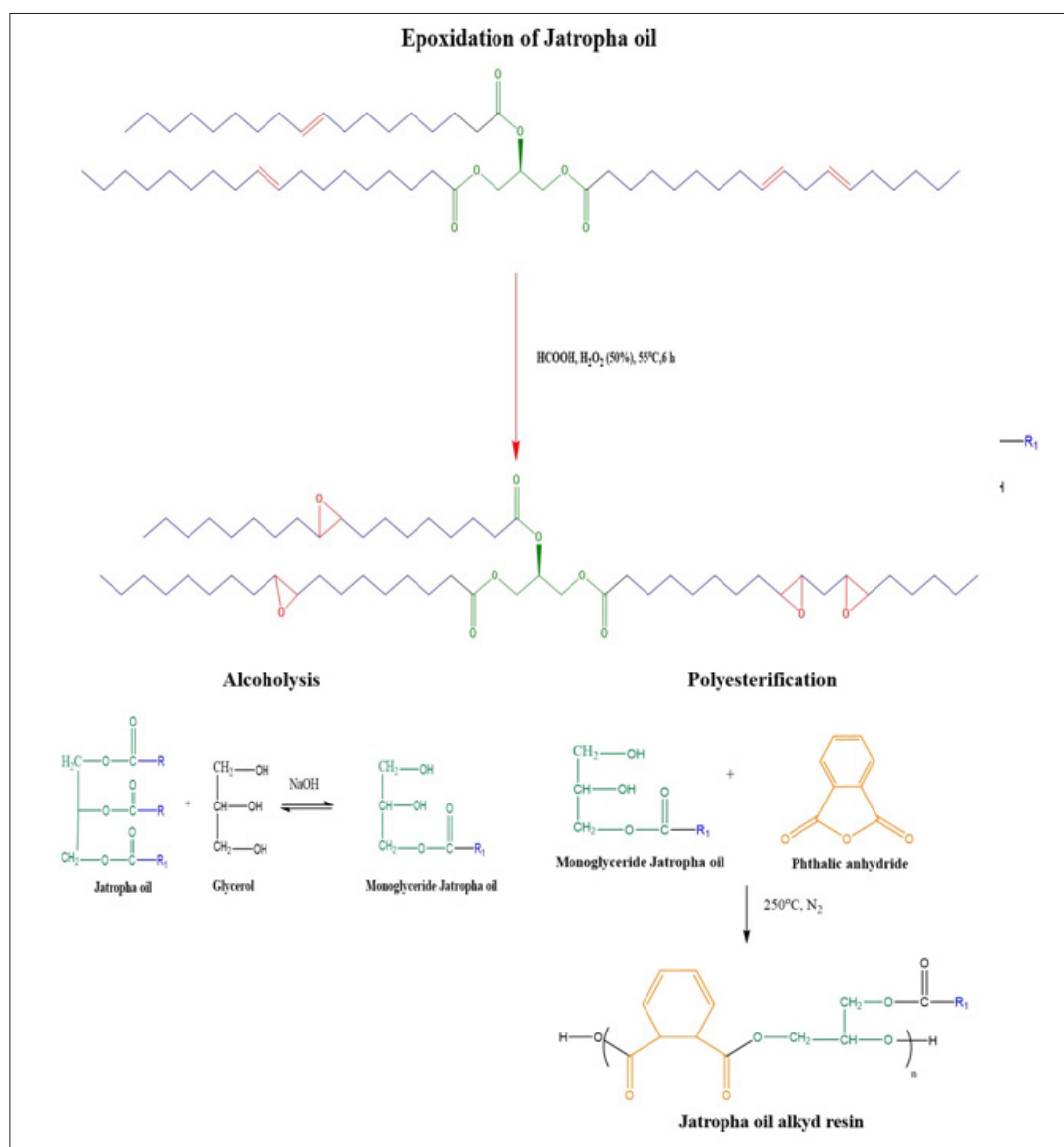


Figure 2: Reaction Scheme of Jatropa Oil Alkyd Resin (JOAR) Synthesis

Characterization of PPD

Fourier transform infrared spectroscopy (FTIR) was performed to characterize the synthesized pour point depressant, JOAR using Perkin Elmer Spectrum 2 FTIR spectrometer within 4000 to 400 cm^{-1} scanning range. ^1H NMR of JOAR was also carried out to ensure the formation of alkyd resin more precisely with Bruker Ascend TM NMR 400 MHz nuclear magnetic resonance spectrometer, using CDCl_3 solvent.

Preparation of Sample and Measurement of Pour Point

For dosing of additives crude oil was liquified by heating at 60°C . Samples were prepared after the addition of 100 to 500 ppm concentration of bioadditive. Prepared samples were allowed to heat in a thermostatic water bath for proper mixing of the additives in crude oil. According to the ASTM D5853- 17a (2017) method, the pour point of virgin crude oil and crude oil treated with several concentrations of PPD was measured.

Rheological Studies

All rheological properties such as viscosity, and thixotropic area of pure crude oil and crude oil dosage with PPD were measured using a Bolin Gemini 2 Advanced Air Bearing Rheometer equipped with a bob-cone cylinder system. Viscosity was determined within 35°C to 45°C with varying shear rate of 0.1 to 1000 s^{-1} . The thixotropic area was measured of pure and PPD added crude oil at two different temperatures, 35°C and 40°C at shear rate ranging from 0 to 600 s^{-1} .

Wax Deposition Thickness Measurement

The performance of JOAR in terms of wax deposition reduction was measured in laboratory-designed Cold Finger equipment. Glass bottles containing samples were placed in the hot water bath and cold fingers of stainless steel were immersed into bottles. The temperature gradient was maintained by Supervisory and Data Acquisition Software (SCADA). In this experiment, a cold finger represents the wall of a pipeline, and glass bottles with hot crude oil demonstrate the flow of crude oil that passes through pipelines.

Result and Discussion

Characterization of Crude Oil

Characteristics properties of crude oil are mentioned in Table 1. Specific gravity was measured using a hydrometer and its value is 0.8055. API gravity was found 21.75 which shows crude oil is light. Water content was obtained less than 1%. The pour point was attained at 33°C, so all the rheological analysis was done above this temperature range. Wax content was 19.8 %. The amount of saturate, aromatic, resin, and asphaltene were measured by following SARA analysis through column chromatography. The details result of the SARA analysis are listed in Table 1, where saturate is found in large quantity, it is almost 57.59 %.

FTIR Analysis of JOAR

In Figure 3, a broad absorption band appears at 3428 cm⁻¹ attributed to the -OH stretching of hydroxyl

group jatropha oil. The peaks at 2924 cm⁻¹, and 2854 cm⁻¹ denote the C-H stretching of the alkane group. The stretching bands of the alkene group (C=H Bonding) are attributed to the peak at 2955 cm⁻¹. The ester group which is the main component, was attributed to the band at 1737 cm⁻¹, which is again confirmed by the appearance of stretching of the C-O-C bond at 1237 cm⁻¹, and 1075 cm⁻¹. The peak at 1463 cm⁻¹ corresponds to the bending vibration of the -CH₂ group.

Table 1: Physical and Chemical Properties of Crude Oil

Characteristics	Units	Value observed	Method
Saturates	W/W%	57.59	SARA
Aromatic	W/W%	19.37	SARA
Resin	W/W%	14.26	SARA
Asphaltene	W/W%	8.77	SARA
Pour Point	°C	33	ASTM D 5853
API gravity	°API	21.75°	ASTM D 28
Wax content	W/W%	19.80	BP-237 ^[23]
Water content	V/V%	<1%	IP 74/57

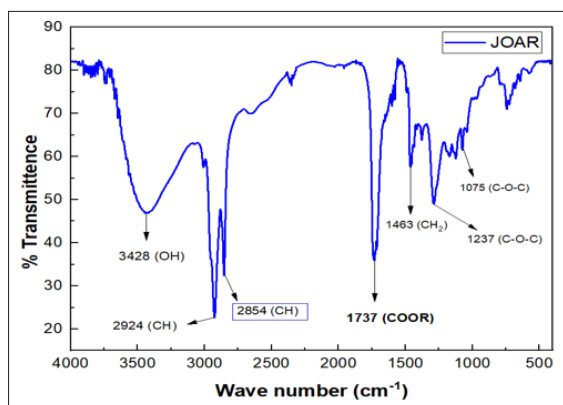


Figure 3: FTIR Spectra of JOAR

NMR Analysis of JOAR

¹H NMR spectra of alkyd resin JOAR is shown in Figure 4. The peak at δ 0.86 ppm is due to the proton of the terminal methyl group of the fatty acid chain. Peak at δ 1.60 ppm represents the proton of the methyl group adjacent to the terminal methyl group. Protons of the remaining methyl group (-CH₂) group appeared at δ 1.24 ppm. Peaks at δ 2.00, 2.31, and 2.77 ppm correspond to the epoxy protons of the alkyl chain. Protons of methylene group (-CH) of glycerol moiety are found at δ 3.65, 3.89, and 4.14 ppm, and the protons of the same glycerol moiety have also appeared at δ 5.33 ppm

which is due to the deshielding effect of the anhydride group. Appearance of the peak at δ 7.54 ppm represents the aromatic proton of the phthalic anhydride group [17].

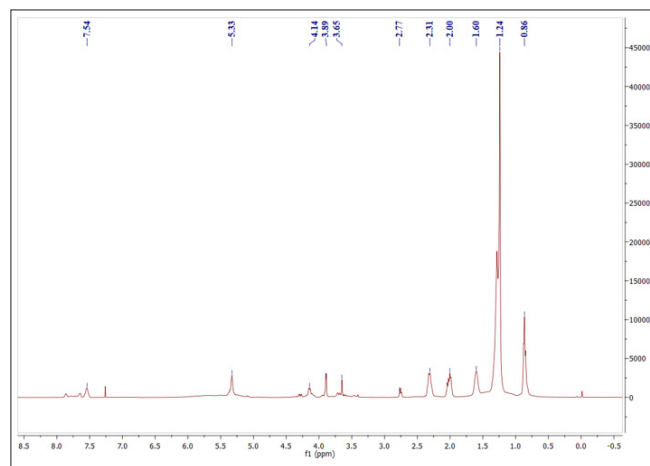


Figure 4: ¹H NMR spectra of JOAR

Measurement of Pour Point

The effect of alkyd resin JOAR on the depression of the pour point is shown in Table 2. From the study, it was observed that the pour point was gradually reduced with increasing concentration of bio additive and the best reduction was observed when crude oil was treated with 400 ppm dosage of JOAR which is 24°C. Alkyd resin JOAR helps to alter the morphology and size of wax particles of crude oil. Jatropha oil contains oleic acid and linoleic acid as major unsaturated fatty acids in its formulation along with a small amount of palmitic and stearic acid as saturated fatty acids [18]. These oleic acid and linoleic acids are subjected to epoxidation first, followed by alcoholysis to convert triglycerides into monoglycerides which are susceptible to polyesterification with phthalic anhydride. Therefore, the polar functional groups present in JOAR are epoxides and polyester. These polar functional groups of PPD and the polar group found in crude oil form an association of polar components and help to soluble the wax particles in crude oil. Additionally, oleic acid and linoleic acid have long aliphatic chains having carbon number C18 which interacts with the non-polar hydrocarbon part of wax particles via Van der Waals force of attraction and adsorbed onto the wax crystals surface to prevent further agglomeration of wax molecules. So, the flowability of crude oil is greatly improved.

Instead of interacting with the wax molecules, bio-additive molecules began to stick together after reaching their saturation point, and the pour point rose again.

Table 2: Pour Point Measurement of Crude Oil with the Addition of JOAR

Concentration of Additive	Pour Point (°C) WCO + JOAR
Crude oil	33
Crude oil + 100 ppm PPD	30
Crude oil + 200 ppm PPD	27
Crude oil + 300 ppm PPD	27
Crude oil + 400 ppm PPD	24
Crude oil + 500 ppm PPD	30

Viscosity Studies

Therefore, to check the best efficiency of the synthesized products of alkyd resin, the studies of the rheology were done on crude oil originating from the Gamiz field. However, the rheological properties of crude oil are greatly dependent on temperature. The presence of high wax content in crude oil forms an aggregate of semi-solid which is responsible for its higher viscous nature. The test of rheology was done at three distinct temperatures 35°C, 40°C, and 45°C at 0.1 to 1000 s⁻¹ shear rate with the geometry of cup and bob with and without addition of JOAR and the results were shown in Table 3. According to Figure 4, viscosity decreases significantly with the addition of 400 ppm JOAR. The percentages of viscosity reduction were calculated according to the following Equation 1,

$$\% \text{ Reduction in viscosity} = \frac{\text{Viscosity WCO} - \text{Viscosity WCO+PPD}}{\text{Viscosity of WCO}} \times 100 \quad (1)$$

The result showed that the reduction of viscosity was almost 45% when treated with 400 ppm of JOAR. Crude oil shows Newtonian behaviour after dosing with JOAR. Due to the increase of temperature from 35°C to 45°C wax particles get solubilized in crude oil, and as a result, this viscosity of crude oil decreased. In this experiment, viscosity reduction was accelerated with the presence of JOAR at 45°C. Therefore, the viscosity of crude oil was decreased by 45% at 35°C which in turn reduced the pumping cost. Interaction of both the hydrophobic alkyl chain of JOAR and polar ester group with wax molecules of crude oil helps to prevent the agglomeration process of wax molecules, increasing the flowability of crude oil.

Thixotropic Area Study

The amount of energy released to change a state and return to its original state is called thixotropic behaviour in crude oil. This behaviour in both samples treated with PPD and untreated crude oil is determined by the size of the hysteresis loop, as seen in Figure 5. The study was conducted with JOAR

additive with various concentrations of JOAR ranging from 100 ppm to 400 ppm at two different temperatures i.e., at 35°C and 40°C with increasing shear rate from 0 to 600 s⁻¹ and return to its starting position. The area under the hysteresis loop is thixotropic area and was calculated in each case [19]. found that crude oil has a unique microstructure in different phases and releases energy when transitioning between phases. Changes in the microstructure occur due to disintegration, accumulation without flow, or breakdown when flow begins. The hysteresis area of untreated crude oil at 35°C was found 7202.6 mPa.s and it is reduced when treated with several concentrations of JOAR. The percentages of area reduction of each hysteresis of different concentrations are calculated using Equation 2,

Table 3: VR% of Crude Oil with the Help of JOAR in Different Concentrations

Sample	35°C		40°C		45°C	
	Viscosity (mPa.s)	%VR	Viscosity (mPa.s)	%VR	Viscosity	% VR
Crude oil	3527.4	—	628.7	82.17	101.7	97.11
Crude oil + 100 ppm JOAR	3437.4	2.5	528.7	86.52	98.5	97.20
Crude oil + 200 ppm JOAR	3286.8	6.8	385.4	89.07	92.7	97.37
Crude oil + 300 ppm JOAR	2762.8	21.7	379.4	89.24	92.1	97.38
Crude oil + 400 ppm JOAR	1939.4	45.0	363.2	89.70	91.6	97.40

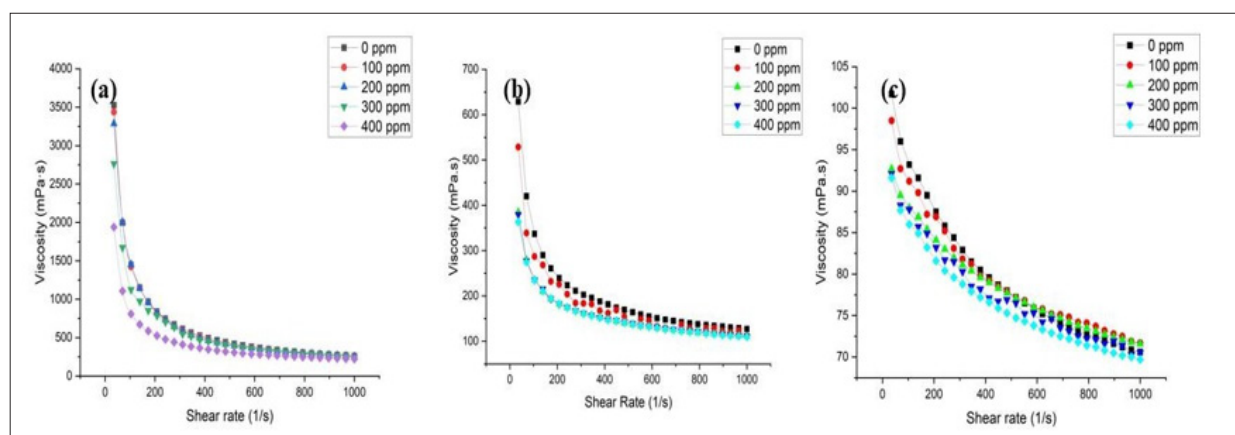


Figure 4: Effect on Viscosity of WCO after Addition of JOAR (a) at 35°C; (b) at 40°C; (c) at 45°C

$$\% \text{ Reduction in area} = \frac{\text{Area WCO} - \text{Area WCO} + \text{PPD}}{\text{Area WCO}} \times 100 \quad (2)$$

The results of the percentage reduction of viscosities are calculated at different concentrations and tabulated in Table 4. The thixotropic area of crude oil decreased with increasing concentration of JOAR. The thixotropic areas are decreased 86% when treated with 400 ppm at 35°C and the reduction is increased to 92% when at 40°C. Therefore, the required energy is less to alter the wax morphology in the case of 400 ppm JOAR treated crude oil [20]. According to, the WCO showed a larger area under the curve because more energy is required to break apart the wax molecules. Table 4 demonstrates that as the wax molecules break down into smaller particles and need less energy to come together at a decreasing shear rate, the additive hysteresis region decreases. This results in a reduction in the energy needed to break the bond for smooth flow through the pipeline with the addition of JOAR.

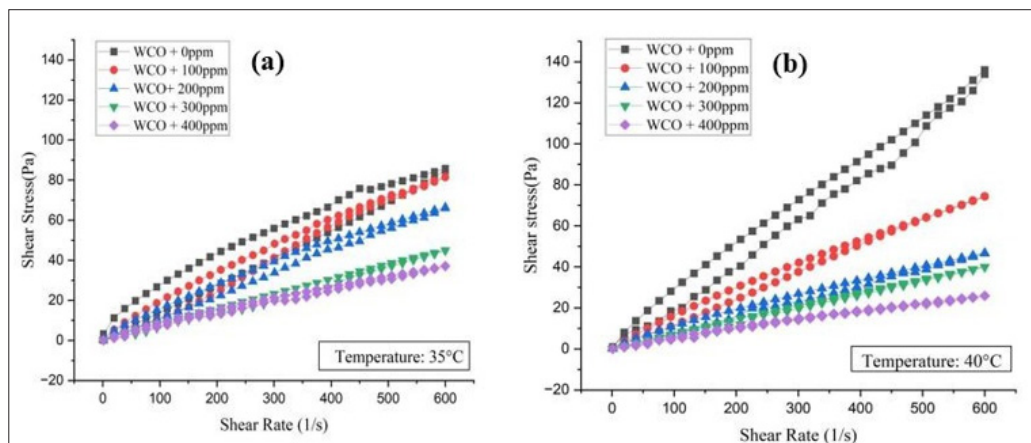


Figure 5: Effect on thixotropic area of WCO after addition of JOAR (a) at 35°C; (b) at 40°C Table 4. % Area reduction calculation of untreated and treated crude oils by varying concentrations at different temperatures 35°C, and 40°C

	35°C		40°C	
Sample	Area (mPa.s)	%VR	Area (mPa.s)	%VR
Crude oil	7202.6	—	5257.5	27.00
Crude oil + 100 ppm JOAR	3025.8	57.99	1782.2	75.25
Crude oil + 200 ppm JOAR	2576.8	64.22	1608.9	77.66
Crude oil + 300 ppm JOAR	1387.5	80.73	623.5	91.34
Crude oil + 400 ppm JOAR	1001.6	86.09	432.9	93.98

Wax Deposition Studies

In the cold finger apparatus, wax deposition was measured for untreated Gamiz crude oil and the effectiveness of JOAR towards flow assurance was also evaluated by varying the concentrations ranging from 100 to 400 ppm dosage [18]. The temperature of the water bath and finger was set at 55°C and 20°C respectively which was 10°C below the pour point temperature. Wax deposition thickness was evaluated by taking the difference between the weight of blank tissue paper with the wax-containing tissue paper. The experiment was carried out for 4 hours and crude oil samples were taken in screw-cap glass bottles equipped with fingers. Wax deposition thickness was calculated by the following Equation 3, $WT = W1 - W2$ (3) Where, WT represents the weight of wax deposited and W1 is the weight of deposited wax on tissue paper and W2 is the weight of blank tissue paper. Results of wax deposition inhibition after dosing with PPD are presented in Table 5. Wax deposition thickness was sufficiently decreased after treatment with 400 ppm JOAR. As mentioned in the previous section, that the non-polar hydrophobic alkyl chain of JOAR, adsorbed on to wax particles of crude oil and the polar groups of JOAR prevent the formation of wax association that is why, wax deposition is decreased by a significant amount after treatment with JOAR.

Table 5: Effect of JOAR on the Effect of Crude Oil Wax Deposition During 4 Hours Experiment At 20°C

	Weight of deposited wax (gram)	
Crude oil	WCO + JOAR	% Of wax inhibition
WCO	0.3008	-
WCO + 100 ppm PPD	0.2262	24.80
WCO + 200 ppm PPD	0.1616	46.27
WCO + 300 ppm PPD	0.1268	57.84
WCO + 400 ppm PPD	0.0582	80.65

Conclusions

In this research, JOAR(Jatropha oil alkyd resin) was synthesized with the help of vegetable oil which is a biodegradable compound. The synthesized product worked efficiently by reducing the pour point of Gamiz crude oil by 9°C. After comparing the results of pour point determination for both the treated and untreated crude oil, it was concluded that the JOAR works effectively as a PPD as well as a viscosity improver. The viscosity of the treated crude oil with the application of 400ppm JOAR decreased by

45% at 35°C from the viscosity of virgin crude oil which helped to a reduction in pumping cost. The thixotropic area of the polygon generated from the thixotropic studies was reduced to 86% and 92% at 35°C and 40°C showing the best performance of JOAR with the application at 400ppm in the virgin crude oil. The rheological studies gave a significant and promising result after the application of JOAR. Hence, this additive does not alter the properties of crude oil and no additional treatment is required to remove this additive.

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