

6. Review of Solvents Based on Biomass for Mitigation of Wax Paraffin in Indonesian Oilfield



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Paint Removal with Pulsed Laser: Theory Simulation and Mechanism Analysis

by Haichao Zhao, Yulin Qiao, Xian Du, Sijie Wang, Qing Zhang, Yan Zang and Zhihai Cai

Appl. Sci. 2019, 9(24), 5500; <https://doi.org/10.3390/app9245500> - 13 Dec 2019

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by M. K. Afdhol, M. Abdurrahman, F. Hidayat, F. K. Chong and H. F. Mohd Zaid

Appl. Sci. 2019, 9(24), 5499; <https://doi.org/10.3390/app9245499> - 13 Dec 2019

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Review of Solvents Based on Biomass for Mitigation of Wax Paraffin in Indonesian Oilfield

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Abstract: This paper presents a review of the expectations and challenges of using biomass in the prevention and slowing of paraffin wax deposition that takes place during the crude oil production process. The inhibition of the deposition process involves the use of solvents from biomass that are generally available around the crude oil production field. The processes used to scale down the precipitation of wax include mixing crude oil with the manufacturer's solvent composed of toluene and xylene. The goal is to assess solvents sourced from biomass that are capable to slow down the wax deposition process. Wax appearance temperature is an important characteristic to evaluate the possible wax precipitation of a given fluid. Wax precipitation can be reduced by using some chemical additives, often called the pour point depressant. This additive is expected to be produced from local biomass which can compete with solvents currently produced on the market.

Keywords: biomass; crude oil; wax appearance temperature; pour point depressant

1. Introduction

The decline in crude oil production is a persistent challenge in the oil industry. The rate of production decreases over time, often caused by damage located in both the rock formation and the surface facilities. Close to 85% of those damages are resultant from oil wax congealing at the wellbore, in production string, and all through the surface pipelines [1]. The majority of crude oil and its products contain large amounts of oil wax called paraffin. Paraffin is a mixture of hydrocarbons formed from linear or normal chains, which are mainly composed of 20 to 40 carbon atoms [2]. Paraffin wax deposition causes equipment failure, upstream and downstream flow congestion, and loss of production, transportation capacity, and storage [3]. Due to the deposition of paraffin wax, plenty of wells are shut-in, countless pipelines blocked, transport vessels taken out of services, and refinery equipment closed at certain times, all resulting in a loss of income [1]. Once formed, the paraffin in which the liquid is transported is deposited on the wall of the pipes.

Paraffin deposition in hydrocarbon production systems is a major challenge for the development of oil and gas fields in cold environments or in deep waters [4–6] where nucleation and adhesion of the wax to the wall of the pipe occur [7]. Paraffin deposition in oil production is a critical problem that is difficult to control in terms of productivity and production operating costs. It begins from the reservoir then progresses to surface facilities [4]. A common method used to mitigate the problem is increasing the temperature of deposit wax. The problem must be handled as early as possible to prevent the decrease in oil production due to solids blocking the flow in the pipe. The primary target of heating the wax is to reduce the viscosity of the fluids. This can also be achieved by the use of some additives. Solvents generated from biomass can act as wax inhibitors.

Research on the application of biomass has mainly focused on the pharmaceutical industry, fuel reserves, and others [8]. However, there has been little discussion about the application of biomass to the crude oil in below surface conditions or near the wellbore. This study systematically reviews the expectations and challenges of using biomass in the prevention and slowing of paraffin wax deposition that occurs during the crude oil production process. Firstly, the review discusses the availability of raw materials in which the types and the area distribution of each material are shown. Secondly, this paper assesses the bioethanol production process consisting of three steps: pretreatment, hydrolysis, and fermentation. Thirdly, this paper examines the problems that occur due to the appearance of paraffin wax and how the solvent is frequently used in the oil industry. Biomass is a renewable energy source that has received attention; various types of biomass have been used such as coffee grounds [9], tea waste, and palm shells [10,11]. Bio-solvents are solvents that can be produced easily and economically where the production process consists of several stages: hydrolysis, fermentation, and the purification process through distillation [12–18]. One type of bio-solvent that can be produced from biomass is bioethanol [19–21]. The raw materials for bioethanol can be sourced from: (1) biomass starch such as corn, cassava, and sorghum [12]; (2) sugar sources, such as cane crops, sugar cane juice, coconut sap, and gum [22,23]; and (3) source of cellulose including rice husks, bagasse, and corncobs [24,25]. In general, biomass basically consists of cellulose, hemicellulose, and lignin [19,26,27] which the availability of its raw materials is a great potential to be utilized.

2. Availability of Raw Materials

The use of waste in waste to energy (WTE) technology involves a treatment process to recover energy in the form of heat, electricity, or transportation fuel from waste sources [28]. WTE is used to create various types of products or semi-finished products from various types of waste such as palm oil waste [29,30], coffee grounds [9], and tea pulp, and even the use of nonorganic waste such as plastic waste [10].

Lignocellulosic biomass, such as agricultural residues (corn stovers and wheat straw), wood, and energy crops, is an attractive material for bioethanol fuel production since it is the most abundant renewable resource on earth [31,32]. Lignocellulosic biomass could produce up to 442 billion liters per year of bioethanol [33]. Thus, the total potential bioethanol production from crop residues and waste is 491 billion liters per year, about 16 times higher than the current world bioethanol production. Rice straw is one of the most abundant lignocellulose waste materials in the world, with an annual production of about 731 million tons, which is distributed in Africa (20.9 million tons), Asia (667.6 million tons), Europe (3.9 million tons), the Americas (37.2 million tons), and Oceania (1.7 million tons) [10]. Food waste from the rest of the food production is used as animal feed. Food waste is composed of complex compounds lignin, hemicellulose, and cellulose (lignocellulose), and the compound has the potential to be biologically converted into other compounds so that they can be used as a solvent [34].

3. Bioethanol Production Process

Bioethanol is made from lignocellulosic biomass found in waste, where this raw material is available in many environments and is expected to increase the value of the waste [35]. Bioethanol is produced through a process of pretreatment, hydrolysis, and fermentation [36–39]. The bioethanol production process is shown in Figure 1.

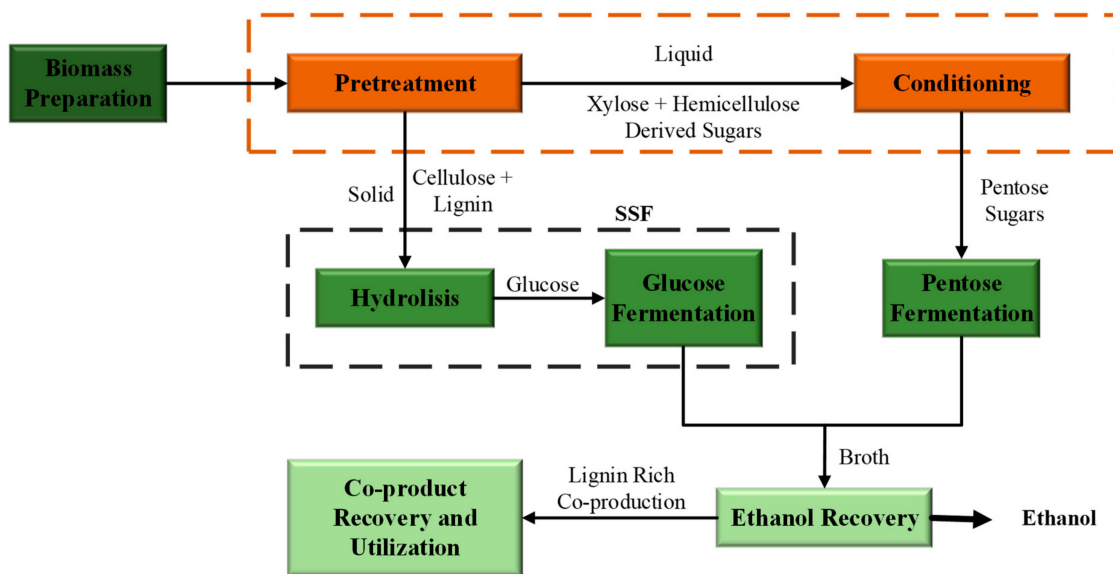


Figure 1. Schematic of the steps involved in conversion of lignocellulose biomass to ethanol [40].

3.1. Pretreatment

Pretreatment is an important step to break down the carbohydrates in which the process aim is to reduce the size of the materials used [31,41] and unlock the cellulose by releasing the lignin in the cellulose [42–44]. Commonly used methods for pretreatment include physicochemical methods (ozonolysis, steam treatment, wet oxidation, and ammonia fiber explosion), chemical methods (acid hydrolysis, alkaline hydrolysis, and organic solvent treatment) [41,45–47], electrical methods (such as pulsed electric field treatment), biological methods (using bacteria or fungi producing ligninolytic enzymes), or a combination of these [32,48].

3.2. Hydrolysis

Hydrolysis consists of the degradation of polysaccharides in aqueous media that can be catalyzed by enzymes, acids, or bases to produce glucose [13,36,42,49–63]. The hydrolysis result will depend on the reaction of enzymes and compounds [64–67].

The hydrolysis yield is determined as the amount of extracted reducing sugars per total amount of theoretical glucose in the biomass according to the following equation [68,69]:

$$\text{Hydrolysis yield (\%)} = \frac{\text{Extracted reducing sugar (g)}}{\text{theoretical glucose (g)}} \times 100. \quad (1)$$

For a total conversion, the glucose theoretically obtained is the same as the amount of cellulose in biomass waste. Total reducing sugars are obtained using the method as follows [70]:

$$\text{Extracted reducing sugar (mg)} = \text{Concentration (mg/mL)} \times \text{Volume}_{\text{Test}} \quad (2)$$

Fermentation yield (Y) is expressed as the obtained ethanol after the distillation process per the amount of extracted reducing sugars:

$$Y(\%) = \frac{\text{ethanol (g)}}{\text{Extracted Reducing Sugar (g)}} \times 100 \quad (3)$$

Fermentation efficiency corresponds to the ratio of the produced ethanol over the theoretically produced ethanol [71], as follows:

$$\text{Ethanol (\%)} = \frac{\text{Ethanol}_{\text{Observed}}}{\text{Ethanol}_{\text{theor}}} \times 100 \quad (4)$$

The bioethanol yield can be obtained using the following equation [29]:

$$\text{Bioethanol Yield} \left(\frac{\text{g}}{\text{g}} \right) = \frac{\text{Volume}_{\text{Hidrolisate}}(\text{L}) \times \text{Concentration}_{\text{bioethanol}}(\text{g/L})}{10 \text{ g Biomass Waste}} \quad (5)$$

Finally, the physical properties of the obtained residue after acid hydrolysis were tested. This analysis included moisture, calorific value, and elemental, inorganic compounds, and immediate analysis according to the standards [68].

3.3. Fermentation

The fermentation process consists of the conversion of glucose obtained from hydrolysis to bioethanol by microorganism media such as yeast, bacteria, and fungi [62,72,73]. *Saccharomyces cerevisiae* was used for fermentation assays [74–76]. This fermentation process lasts several days depending on the number of yeast cell culture starters used. The larger the number of starters, the faster the fermentation process. Besides producing ethanol, fermentation produces also other substances, including water. One measure of the quality of bioethanol is its water content. The lower the water content, the better the quality of the bioethanol [19,77], (Table 1).

Table 1. Advantages and disadvantages of methods for pretreatment lignocellulose materials.

Pretreatment Method		Advantages	Disadvantages	Ref.
Chemical	Acids	High glucose yield	High cost of acid, must be recovered	[78]
		Short reaction and residence time	Solvents need to be recycled, costly, causes corrosion to reactors, leads to formation of some inhibitors	[43,79]
	Base combined with oxidizing agents	Lignin and hemicellulose are removed		[79]
		Proceeds at ambient temperature	Generation of degradation products	[78]
	Organic solvents	Causes lignin and hemicellulose hydrolysis, useful for lignin recovery	Formation of toxic inhibitors	
			Reactor corrosion problems	
	Ozonolysis	High delignification efficiency High glucose yield	High cost	[41]
			Solvents need to be drained and recycled	[43]
	Ionic liquids	Mild environmental conditions (temperature and pressure)	Large amounts of ozone needed	[78]
			Process economically unviable	
No formation of toxic inhibitors			[78]	
Low generation of degradation products			[80]	
Physical	Microwave irradiation	Reduce cellulose crystallinity	[43]	
		Lack of studies of the impact of enzymes on the next stage	[41]	
	Milling	Faster heat transfer	Low penetration in bulk products	
		Shorter reaction times		[78]
		Low generation of degradation products		
Mechanical comminution	Reduces cellulose crystallinity	High power and energy consumption	[43]	
	Reduces of degree of polymerization		[79]	
Extrusion	Low generation of degradation products	High power and energy consumption	[41]	
	Low formation of toxic inhibitors	Temperature regulation difficulties, limited cooling capacities	[78]	
Physicochemical	Liquid hot water	Low formation of toxic inhibitors	Limited residence time	
		No catalyst required	High energy consumption	[41,81]
	Steam explosion	Low-cost reactor construction	Not developed on commercial scale	[78]
		Low corrosion potential		
		Low generation of degradation products		
Higher yield of glucose and hemicellulose in a two-step method	Causes lignin transformation and hemicellulose solubilization	Generation of toxic compounds	[80]	
	Cost effective	Partial hemicellulose degradation	[78]	

Table 1. *Cont.*

Pretreatment Method	Advantages	Disadvantages	Ref.
CO ₂ explosion	Increase accessible surface area Cost effective	Does not affect lignin and hemicelluloses Very high pressure requirements	[43] [33,41]
Ammonia fiber explosion	Does not generate of toxic compounds Increases accessible surface area Low formation of inhibitors	Not efficient for raw materials with high lignin content High cost of large amount of ammonia High cost of ammonia	[79] [78]
Soaking aqueous ammonia	Performed at lower temperature Glycan and xylene remain in the solid Low formation of inhibitors		[78]
Biological	Degrades lignin and hemicellulose	Low rate of hydrolysis	[41]
	Low energy consumption Low-capital cost No chemicals requirement Mild environmental conditions		[43] [78] [79]

4. Wax Problems and Use of Solvents in the Oil Industry

Most of the commercial wax inhibitors tested can reduce the deposition of paraffin with low molecular weight ($\leq C_{34}$), but have little effect on high molecular weight paraffinic wax (C_{35} – C_{44}) [82]. Wax deposition in oil production flow mainly consists of C_7 paraffin. Paraffin deposition is controlled by temperature, which is why heat application is preferable to manage the paraffin deposition [83,84]. When the temperature in a system decreases, paraffin in the liquid phase starts precipitating out of the solution as a solid. Wax deposition forms on the wall of the pipe where the temperature gradient is at its highest [3,85]. Pressure–temperature relationship of wax appearance in both reservoir fluid and stock tank oil (STO) condition is shown by Figure 2.

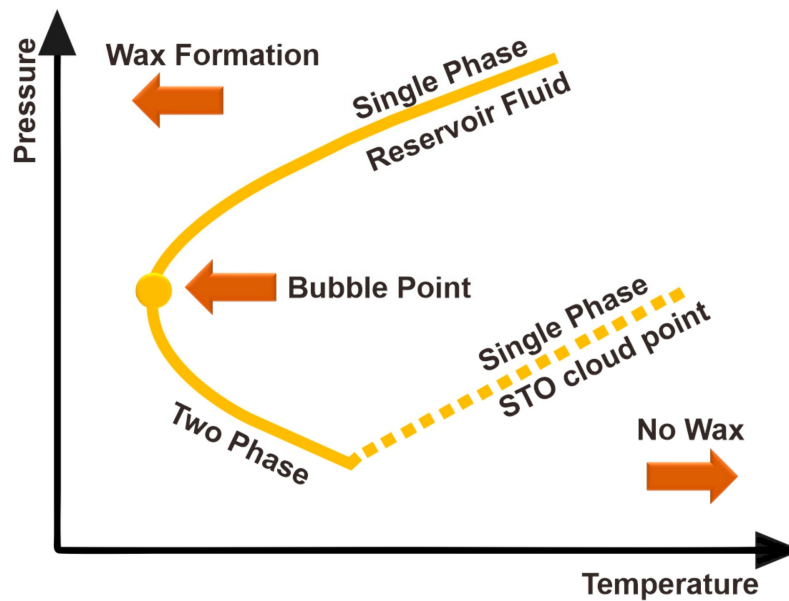


Figure 2. Pressure–temperature relationship of wax appearance in both reservoir fluid and stock tank oil (STO) condition.

The tendency and deposition rate can be adequately predicted by calculating the rate of diffusion of wax molecules onto the wall with the following equation:

$$\frac{dm}{dt} = -\rho D_m A \frac{dC}{dr} \quad (6)$$

where m is the mass of deposit (kg), ρ is the density of wax (kg/m^3), D_m is the molecular diffusion constant (m^2/s), A is the deposition area (m^2), C is the concentration of wax (%), and r is the radial position (m) [86].

The radial concentration gradient can be easily calculated if it is broken down into two components by applying the chain rule as shown in the following equation:

$$\frac{dm}{dt} = -\rho D_m A \frac{\partial C}{\partial T} \frac{dT}{dr} \quad (7)$$

where T is temperature. The concentration gradient can be calculated from the wax concentration predicted by the thermodynamic model for the temperature range [3,86].

Additional chemicals considerably affect the process of paraffin wax deposition, where measurements show that wax deposition is rather high for all crude oil [87]. No correlation exists between the temperature and levels of total wax of crude oil [4]. The deposition of paraffin or wax can be called a depositor of carbonaceous material, which is insoluble or dispersed by crude oil under normal conditions [88,89].

The effectiveness of the inhibitor must be tested for each application. Tests must be conducted under all possible operating conditions. Chemical inhibitors for prevention of wax include:

- (1) Thermodynamic wax inhibitor (TWI)—pressing cloud point, reducing viscosity and pour point, requires a high volume;
- (2) Depressant/pour point—modifies the wax crystal structure, reduces viscosity, and produces stress, but does not reduce the rate of wax deposition;
- (3) Dispersants/surfactants coat wax crystals to prevent wax growth and change the wetting characteristics to minimize wax adhesion to the pipe wall or other crystals [3,90].

Wax characteristics are outlined in Table 2.

Table 2. Physical characteristic carbon distribution analysis and molecular type composition of slack wax crudes [91].

Characteristic	Slack Waxes		
	Light	Middle	Heavy
Congealing point, °C	48	59	62.5
Kinematic viscosity, 98.9 °C	3.04	4.30	6.00
Refractive index, 98.9 °C	1.4224	1.4270	1.4402
Density, 70 °C	0.7920	0.8035	0.8107
Mean molecular weight	384	446	477
Oil content, wt.%	5.32	6.23	23.05
Cone penetration, 25 °C	17	13	28
Needle penetration, °C	43	40	59
Sulfur content, wt.%	0.09	0.10	0.22
Color (ASTM D-1500)	1.0	1.5	3.0
Carbon Distribution Analysis			
% C _A	5.93	8.41	10.46
% C _N	12.85	14.20	29.44
% C _R	18.78	22.61	39.90
% C _P	81.22	77.39	60.10
Molecular Type Composition			
Total saturates, wt.%	97.63	96.97	86.18
n-paraffin content, wt.%	74.71	62.89	35.07
Iso- and cyclo-paraffin content, wt.%	22.92	34.08	51.11
Total aromatics, wt.%	2.37	3.03	13.82
Mono-aromatics, wt.%	2.37	3.03	11.52
di-aromatics, wt.%		-	2.30
% C _A —percentage of aromatic carbon per average molecule % C _N —percentage of naphthenic carbon per average molecule = % C _R - % C _A % C _R —% C _N + % C _A % C _P —percentage of paraffinic carbon per average molecule = 100% C _R			

4.1. Pour Point Depressants (PPDs)

Pour point depressants (PPDs) are a solution in heavy or extra-heavy crude oils where the main problem is flowability, especially from offshore to ground facilities [92,93]. A crystal candle modifier, also called a PPD, is a chemical that determines the solubility of wax, adsorption, and nucleation. The modification of wax crystallization may help depress the crude oil's pour point, viscosity, and yield stress appreciably [94].

Many polymers have been used as pouring depressants. In heavy waxed oils, high-branched poly- α -olefins, long-chain fatty acid amides, poly-n-alkyl, acrylates, methacrylate copolymers [95], alkyl ester copolymer carboxylic acids, unsaturated α -olefin, ethylene-vinyl fatty acid esters, vinyl acetate- α -olefins, and maleic anhydride copolymers are used. A number of patents have been generated for these polymers [92].

Examining the effect any different PPDs on the viscosity, pouring point, and temperature of the candle selection proved that PPD substances are able to reduce the wax crystals, changing the growth

characteristics and surface of the crystal at metal surfaces that are attached like pipe walls [96], as shown in Figure 3. Studies were able to reduce crude oil from 1000 cP to 10 cP at 10 °C–20 °C in all of these additives, which increased the flow of crude oil. The following effect on the viscosity of crude oil was successfully modeled by regulating lower melting temperatures for wax molecules in the range of C₂₁ to C₄₅ in the presence of additives that increase flow [95].

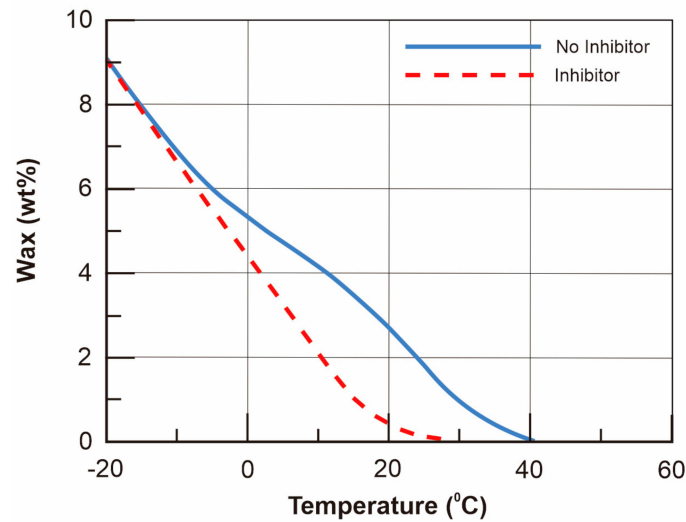


Figure 3. Precipitated weight of paraffin from the oil with (dashed line) and without (solid line) inhibitor [95].

Several studies have been conducted in Mexico to test the rheological properties of light and heavy oils by measuring pouring depressant performance using a combination of vinyl acetate, styrene, and n-butyl acrylate, and viscosity was reduced at 25 °C and 50 °C [97]. In other studies, the viscosity in heavy crude oil dropped at 25 °C and in light crude oil at 313 K due to the depressant pour point [92].

Figure 4 shows the effect of concentration on the depressant pour point. The pour point can be reduced at 11 °C and a dose of 400 ppm. At doses above 400 ppm, no further reduction in pour point occurs, which causes it to be uneconomical [94].

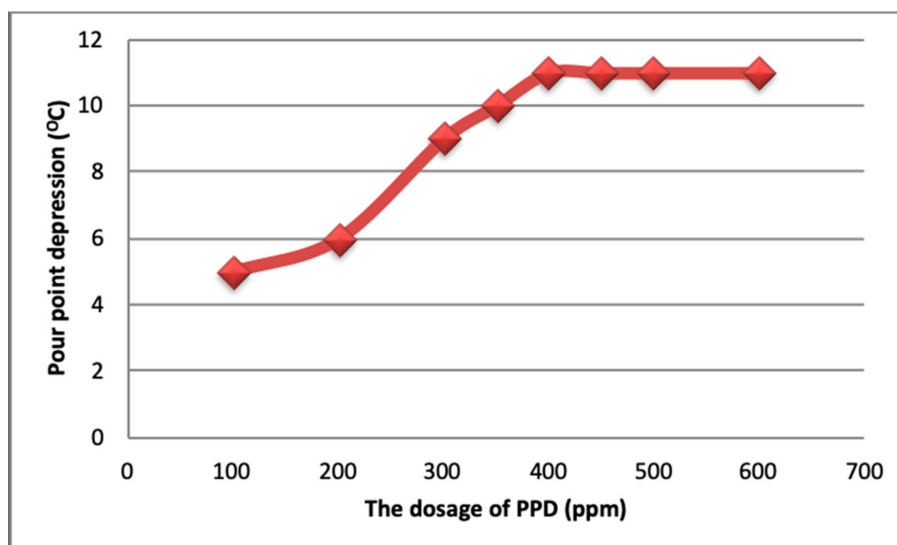


Figure 4. Effects of pour point depressant (PPD) dosages on pour points.

The pour point of pure crude oil is calculated by the following equation:

$$\text{Pour point reduction } (\Delta P) = PP_{\text{pure}} - PP_{\text{additive}} \tag{8}$$

where PP_{pure} is the pour point of the pure crude oil and PP_{additive} is the pour point of the crude oil containing additives [98].

4.2. Rheological Studies

The depressant pour point mixed into crude oil at 400 ppm is depicted in Figure 5. At temperatures above 55 °C, the viscosity of crude oil decreases and remains constant at subsequent temperatures [99], meaning that at this temperature, the crude oil flow benefits because it can flow without depressant pour points [100,101]. When the temperature decreases, the viscosity of crude oil increases, thereby increasing the viscosity of the oil, which is then accompanied by deposition [102,103]. Then, at temperatures below 50 °C, the wax appearance temperature (WAT) is higher without the addition of pour point depressant [104]. The addition of pour point depressant was proven to reduce the viscosity of crude oil so that it can increase the flow of low-temperature crude oil [105].

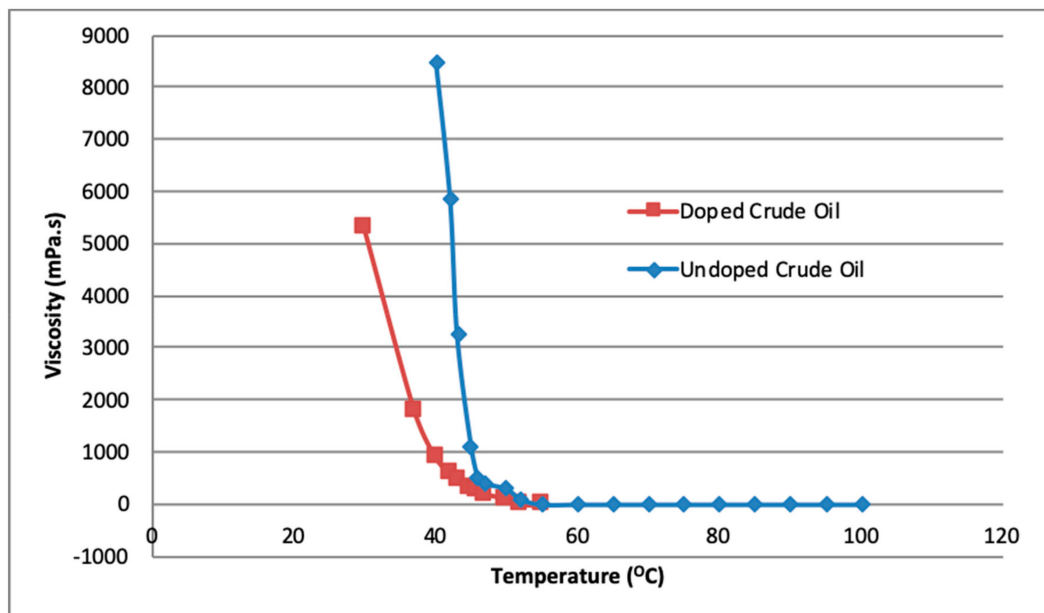


Figure 5. Comparison of wax appearance temperature with doped–undoped PPD to crude oil.

4.3. Evaluation of Paraffin Inhibition Efficiency

WAT Determination (ASTM Standard D5773-04, 2005)

WAT is also called the cloud point [7,106]. Lately, some researchers have shown the advantage of calculating wax disappearance temperature (WDT) compared to WAT. WDT is the temperature at which the last wax crystal melts. The WDT is thermodynamically more precise because crystallization requires some saturation [107]. The efficiency of paraffin inhibition (total PIE) for total wax deposition can be calculated as [88]:

$$PIE_{\text{total}} (\%) = \frac{W_{n(C11-C44)} - W_{i(C11-C44)}}{W_{n(C11-C44)}} \times 100 \tag{9}$$

where $W_{n(C11-C44)}$ is the total amount of paraffin deposits without inhibitors (mg) and $W_{i(C11-C44)}$ is the amount of paraffin deposit with added inhibitors (mg) [4,82]. The effects of commercial wax inhibitors on ΔWAT and inhibitor efficiency in wax deposition is shown by Table 3 below.

Table 3. Effects of commercial wax inhibitors (200 ppm) on Δ WAT, and inhibitor efficiency in paraffin deposition [82].

Inhibitor	Chemical Name	Δ WAT ($^{\circ}$ C)	PIE _{light} (%)	PIE _{heavy} (%)	PIE _{total} (%)
A	Poly alkyl enamine	−2.7	71	13	52
B	Proprietary	−3.3	87	75	83
C	Polyolefin amide alkene amine	−0.1	18	−9	9
D	Polyolefin amide alkene amine	−0.2	33	3	23
E	Poly acrylate	0.0	42	28	37
F	Poly alkyl acrylate	−3.4	72	18	54
G	Olefin amide copolymer	−1.7	55	−3	36
H	Proprietary	−4.2	73	29	58

5. Discussion

Solvent treatments of wax deposition are often the most successful remediation methods but are also the most costly. Therefore, solvent remediation methods are usually reserved for applications where hot oil or hot water methods have shown little success. When solvents contact the wax, the deposits are dissolved until the solvents are saturated. If they are not removed after saturation is reached, the waxes may precipitate, resulting in a situation more severe than prior to treatment [3].

5.1. Production of Solvents for the Oil Industry

The process of producing a solvent from biomass waste certainly has the potential for development, especially with the increasing amount of waste produced by people today. Very little of that waste can be recycled back into a valuable product, which is a complex problem for the industrial world that must be addressed. This is especially true in the oil industry, as the chemical treatment needed to overcome this problem is enabling the development of the world of industry by optimizing petroleum production [108].

5.2. Solvent Application in the Oil Industry

One important challenge in crude oil production and fuel handling is the risk of wax deposition. This deposition is of particular concern in the production of crude oil from deep-water fields where the liquid produced can cool to near the surrounding water temperature, which is 4 $^{\circ}$ C or less. The paraffin deposits that can occur in these long transportation routes can endanger these projects due to partial or even complete blockages of these pipes [82,89].

Paraffin hydrocarbon liquids, including crude oil and condensate, form a paraffin solid phase when the temperature drops below the liquid cloud point [4]. Cast points are indicators of temperature where oil will solidify into a gel. At the pour point, the fluid still flows under gravity; at the next lower measurement temperature (lower than 3 $^{\circ}$ C), the fluid has already formed a gel and does not flow. In the ambient temperature, where the fluid is at or below the temperature of the pour point, special pouring strategies are needed to maintain fluid flow capability. So, the ambient temperature condition can be analyzed in general so that the analysis is conservative. The extreme 100-year minimum seabed temperature is used along the pipeline route [3].

6. Conclusions

In this study, we examined the feasibility of bio-solvent application to the inhibition of the deposition of wax that forms in oil production facilities. Generally, we found that the bioethanol production process, used to create a solvent, shows potential, especially bio-solvents which are created from biomass in the surrounding environment. The ability of bioethanol to act as a suitable solvent to prevent the deposition of paraffin wax must be determined. This study has been one of the first attempts to thoroughly assess this wax problem. The process of assessment for a wax problem in an oilfield can be summarized as follows: obtain a good sample; determine the cloud point or WAT based on solid–liquid equilibrium; determine the rheology including the viscosity, pour point, and

gel strength; the crude oil composition must be quantified, including standard oil composition; wax deposition rates must be identified, including flow loop, wax melting point, interfacial tension; and the use of wax inhibitors from biomass or organic waste should be considered. Using a wax paraffin solvent produced from biomass is an intriguing challenge that could be explored in further research.

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Nomenclature

TWI	Thermodynamic wax inhibitor
WAT	Wax appearance temperature
WDT	Wax disappearance temperature
WTE	Waste to energy
PIE	Paraffin inhibition efficiency

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