CHEMISTRY & CHEMICAL TECHNOLOGY

Chem. Chem. Technol., 2025,
Vol. 20, No. 1, pp. 092–097

Chemistry

EFFECT OF DEPRESSANT ON THE INHIBITION OF WAX DEPOSITION OF CRUDE OILS FROM THE WESTERN REGION OF UKRAINE

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https://doi.org 10.23939/chcht20.01.092

Abstract. Wax deposits create significant obstacles to the flow of crude oil, as they cause pressure anomalies in the pipeline, reduce its passage diameter, and lead to artificial blockages. For two types of oil from the Dolinske and Borislavske fields (the western region of Ukraine), the amount of deposits was determined using the Cold Finger method. The dynamics of the growth in the deposit amount within a time up to 24 hours were shown. The effectiveness of the Dodiflow 5236 pour point depressant as a wax deposition inhibitor was evaluated. It was found that the depressant reduces the amount of deposits formed by 1.5-2.5 times. In addition, the depressant reduces the rate of wax deposition and changes the structure of crystals. It was shown that the amount of deposits formed from crude oil and oil with a depressant depends not only on the physicochemical characteristics of the oil, but also on the temperature difference between the oil and the wall.

Keywords: Cold Finger method, depressor, wax, deposits.

1. Introduction

Oil deposits, which many oil companies around the world are striving to combat, are a complex system comprising many different components with ratios that vary widely. Wax deposits, which are particularly characteristic of high-waxy oils, are one of the main components of oil deposits. They create significant problems throughout the entire process from oil extraction to refining at the plant, and cause the so-called paraffinization of pipelines.

Pipeline paraffinization is the uneven deposition of a dense layer of paraffins, ceresins, asphalt-resinous substances, and mechanical impurities on the inner surface of the pipeline when pumping waxy oils and petroleum products that are cooled below the wax appearance temperature.[1] In the transportation system, crude oil enters the pipeline and comes into contact with the cooled surface. This is especially true during the cold season. The result of such contact is the formation of a temperature gradient directed toward the center of the flow perpendicular to the cooled surface. Due to the turbulence of the oil flow, its temperature decreases; this leads to two parallel processes: the isolation of wax crystals on the cold surface and the crystallization of wax in the oil volume. Paraffinization of the pipeline reduces the passage diameter of the pipeline and decreases its throughput capacity. As a result of the deposit accumulation on the inner surface of pipes, pipelines become clogged, which reduces the efficiency of pumping stations and lowers the overall productivity of the system.[2]

In the oil production industry, addressing the issue of wax deposits involves two primary approaches: preventing their formation and removing existing deposits.[3] In the first case, the internal surfaces of the pipelines are coated with anti-wax protective layers,[4] surfactants are introduced into the oil flow, pumping is carried out at temperatures higher than the wax appearance temperature (WAT), and solvents[5,6] or biological treatment methods[7] are used. Methods for removing deposits that have already formed include mechanical cleaning,[8,9] the use of magnetic or ultrasonic fields,[10] and the use of chemical reagents.[11]

Among the wide range of chemicals that can be used to improve pipeline throughput and solve problems associated with wax deposits are crystal modifiers: wax inhibitors, dispersants, surfactants, and depressants.[12]

Wax crystal modifiers contain hydrophobic fragments that interact with wax molecules and polar fragments that alter and modify the morphology of wax crystals through co-crystallization.[13] Modification of crystal growth and surface properties is possible by reducing the pour point and viscosity of the crystalline solution. This will reduce the tendency of crystals to adhere to metal surfaces, such as pipe walls. On the other hand, modifiers do not prevent deposition formation, but only slow down the deposition mechanism. The result is the formation of brittle wax, which allows it to be removed by the oil flow.[7]

Wax inhibitors are solvents or distillates of crude oil that reduce WAT, but are considered uneconomical due to the large volume required for their effective application. Dispersants and surfactants act on the surface of wax crystals, reducing adhesion between particles or between particles and walls. Depressants change the morphology of wax crystals. During crystal growth, the depressant molecule, which is incorporated into the crystal, can create spatial obstacles for paraffin molecules that continue to deposit on the crystals, leading to their deformation.[13]

Dolyna and Boryslav oils, which are produced in the western region of Ukraine, are high waxy oils with a high pour point. The transportation of these oils poses characteristic problems,[14-17] including the wax deposition. Since the choice of depressant for each specific oil is made individually when developing oil pipeline pumping technology,[18] the authors of this article previously conducted a series of studies on the selection of the optimal depressant specifically for western Ukrainian oils.[19] The result was Dodiflow 5236 depressant manufactured by Clariant, Switzerland, which is referred to pour point depressant (PPD). It is known from the literature that PPD that are most effective in improving the low-temperature properties of oil also have the highest ability to inhibit deposits.[12,20]

The purpose of this work was to determine the characteristics of wax deposits formed from Dolyna and Boryslav high waxy oil, and to study the effectiveness of the Dodiflow 5236 depressant as the deposition inhibitor.

2. Experimental

The study was conducted on samples of commercial oil collected at the Dolyna Naftogaz Oil and Gas Production Department, Ukraine (designated as oil I) and the Boryslav Naftogaz Oil and Gas Production Department, Ukraine (designated as oil II).

To evaluate the effect of pour point depressant (PPD) on the inhibition of wax deposits, Dodiflow 5236 depressant, manufactured by Clariant, Switzerland, was studied. According to the results of previous studies,[19] it significantly decreases the pour point of the studied high waxy oils (Table 1).

Table 1. Pour points of the studied oils

Index	Oil I	Oil II
Pour point of crude oil, °C	+19	+17
Pour point of oil with Dodiflow 5236 (1000 ppm), ºC	-2	-1

The content of paraffins, resins, and asphaltenes was determined in accordance with ASTM D6560-12.[21]

The amount of was deposits (wax residues) was determined using the Cold Finger method. Simple and easy to use, this method is often used in oil refining to simulate wax deposition under real flow conditions,[22-23] when hot oil comes into contact with cold pipeline walls.

It should be noted that during isothermal transportation, the rate of wax deposition depends on the oil temperature; during non-isothermal transportation, the deposition increases with a decrease in the temperature of the pipe wall.[24] The pipeline used to transport the oils under study is a non-isothermal pipeline, so during the research, the temperature of the Cold Finger was below the WAT and varied between 5 and 20 °C. Distilled water was used as a heat transfer. The temperature of the oil in the tank was 60 °C, *i.e.*, it was higher than the WAT. This provided a temperature differential of 40–55 °C.

The wax appearance temperature was determined using a differential scanning calorimeter (Mettler Toledo, Switzerland). The duration of the experiments was 6, 12, 18, and 24 hours.

The diagram of the laboratory setup is shown in Fig. 1.

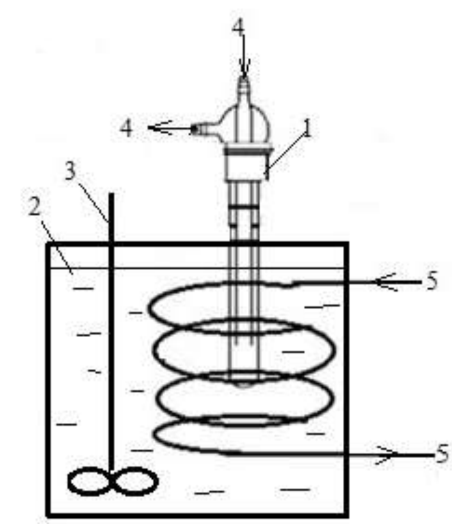


Fig. 1. Schematic diagram of the Cold Finger test setup: 1 – cold finger; 2 – tank with investigated oil; 3 – stirrer; 4 – cold flow; 5 – hot flow.

Before the beginning of the experiments, the crude oil was pretreated to melt the paraffin hydrocarbons, the same as in our previous experiments.[19] The Cold Finger is designed in such a way that the temperature of the inner cylinder *1* is maintained by a circulating water bath *4* in the range of 5−20 °C. The required temperature of oil (60 °C) in the tank is maintained by a hot flow *5*. The stirrer *3* ensures a rotation speed of 120 rpm during all experimental time. Wax is deposited on the cold surface due to the temperature difference between the Cold Finger and the hot oil. After reaching the necessary deposition time, the stirrer was turned off, and the oil was drained through the valve at the bottom of the tank. After 10 min, the Cold Finger was removed, and the deposit was scraped off its surface with a special tool and weighed on analytical scales. Each experiment was performed twice, and the average value was determined as the final experimental result for further analysis and comparison. The error was within the permissible range.

The effectiveness of the depressant (*E*, %) was calculated using the following formula:

$$E = \frac{m_1 - m_2}{m_1} \cdot 100$$

where m_1 and m_2 are the masses of deposits formed from oil without and with a depressant, respectively.

3. Results and Discussion

3.1. Physical and Chemical Characteristics of Oils

Table 2 shows the content of resinous, asphaltene, and paraffin components in oils I and II. The content of paraffin and asphaltenes in both oils under study is approximately the same, while the content of resins in oil I is approximately twice as high.

A sufficiently large amount of paraffin (9.5 wt% and 9.1 wt% for oil I and II, respectively) leads to the formation of deposits, especially when oil, which is moving through the pipeline, comes into contact with cold walls. When cooled, paraffin crystals transform their structure in such a way that, when connected to each other, they form a continuous lattice similar to a wide ribbon. In this form, the adhesive properties of paraffin increase many times over, and its ability to adhere to solid surfaces is significantly enhanced.[3]

Table 2. Content of resinous, asphaltene, and paraffin components in the studied oils

Index	Oil I	Oil II
Paraffins, wt%	9.5	9.1
Asphaltenes, wt%	1.2	0.9
Sulfuric-acid resins, wt%	19.0	10.1

Asphaltenes and resins interact with paraffins, and for high-resin paraffinic petroleum systems, the effect of asphaltenes and resins on deposit formation can be depressing.[25] The studied oils were compared according to the C/(P+A) criterion, which is 1.77 for oil I and 1.01 for oil II. Comparison of these criteria shows that as the resin content in oil increases, the amount of deposits formed decreases (*vide* Table 3). The results obtained are in agreement with the conclusions presented by other authors.[3,26,27]

The wax appearance temperature, determined by differential scanning calorimetry, was 27.8 °C for oil I and 24.3 °C for oil II.

3.2. Determination of the Amount of Wax Deposits

The experimental results obtained when determining the amount of wax deposits for crude oil and oil with the addition of Dodiflow 5236 depressant are presented in Table 3.

Table 3. Amount of wax deposits from oil with and without depressant at different Cold Finger temperatures. Oil temperature was 60ºC, deposition time was 6 hours

Cold Finger temperature, ºC	Temperature differential	Amount of deposits, g					
		Oil I			Oil II		
		Without depressant	With depressant	Effectiveness, %	Without depressant	With depressant	Effectiveness, %
5	55	3.91	2.55	35	4.30	2.86	33
10	50	3.22	1.87	42	3.58	2.10	41
15	45	2.24	1.03	54	2.75	1.29	53
20	40	1.60	0.64	60	1.95	0.89	54

For both oils without depressant, the amount of deposits formed is quite significant, decreasing with a decrease in the temperature differential (in this case, with an increase in the Cold Finger temperature to 20ºC) by 2.2–2.4 times. This pattern is confirmed by data from other researchers,[24,28-31] who note that the key factor determining the amount of deposits in pipelines is the temperature differential between the pipeline wall and the oil inside it.

Reducing this temperature differential leads to a decrease in the amount of wax deposited on the pipeline wall.

When PPD is added to oil, the amount of deposits at +5 °C decreases from 3.91 g to 2.55 g (by 1.5 times), and at 20 °C – from 1.60 g to 0.64 g (by 2.5 times). The effectiveness of the depressant in this case is 35-60%. For oil II, as a result of adding the depressant, the amount of deposits at T=5 ºC decreases from 4.3 g to 2.86 g (depressant effectiveness 33%). Raising the temperature to 20ºC increases the effectiveness of the depressant to 54%. In this case, the amount of deposits decreases from 1.95 g to 0.89 g. The pattern of decreasing deposits with decreasing temperature differential, which was observed for crude oils, remains the same when a depressant is added.

This pattern can be explained by the fact that when the pipe wall temperature is lower than the oil temperature and lower than the WAT, the thermal driving force generated by the temperature gradient enhances effects such as molecular diffusion.[24] The greater the difference between the temperature of the oil and the pipe wall, the more significant the temperature fluctuations in the latter, which leads to an increase in the concentration gradient of paraffin molecules, which in turn enhances molecular diffusion and accelerates the process of wax deposition.

If we compare the temperatures at which deposition occurs in crude oil and oil with a depressant, it becomes clear that depressants shift wax deposition toward lower temperatures (in this case, by approximately 10 °C).

It should be noted that during the experiments, a change in the structure of the obtained deposits was observed. When a depressant was used, the deposits formed were softer and looser than in experiments without PPD. The deposits were easier to scrape off the surface of the cold finger. The results obtained confirm that this effect is due to a change in the structure of paraffin crystals in the presence of depressants. That is, PPD acts as a structure modifier, reducing the size of crystals, changing the shape of the crystal lattice, and thus affecting the process of deposit formation.

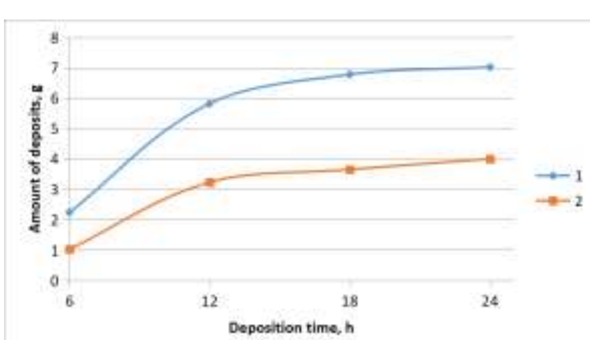
The mechanism of depressant action is determined by its ability to adsorb on paraffin crystals and prevent the formation of a dense crystal lattice. As a result, further crystal growth is impeded, and the ability of crystals to aggregate and form deposits is reduced.[13] In fact, the depressant acts as a dispersant. Most likely, the polar groups of the depressant, which are directed into the dispersion medium, create a kind of energy barrier and prevent the separation of crystals into large agglomerates. This contributes to the formation of a fine-crystalline structure, resulting not only in improved low-temperature and rheological properties of oil, but also in a reduction in the amount of wax deposits. Thus, the depressant has a dual effect: it inhibits wax deposits and changes their structure.

Asphaltenes and resins present in the studied oils can also react with the depressant, forming asphaltene-resin-depressant agglomerates, which reduce the amount of deposits. Chia *et al*.[32] concluded that agglomerates can act as paraffin crystal nuclei and modify the paraffin crystallization process, thereby improving the rheological properties of crude oil. However, the studies were conducted only on model waxy oil, so the existence of a synergistic effect for real oils remains in doubt. Liu *et al*.[33] noted that asphaltenes and resins present in crude oil can chemically bond with a depressant containing reactive groups, and the reaction products can act as a more effective nucleator of crude oil with PPD.

The effect of asphaltenes and resins on the effectiveness of the depressant is not in doubt, but further thorough research is needed for the oils from the western region of Ukraine.

3.3. Dynamics of Wax Deposit Formation

The next stage of research was to study the dynamics of wax deposit formation in crude oil and oil with the addition of a depressant. Fig. 2 shows the results of experiments at different times of wax deposition. The deposition times were 6, 12, 18, and 24 h.



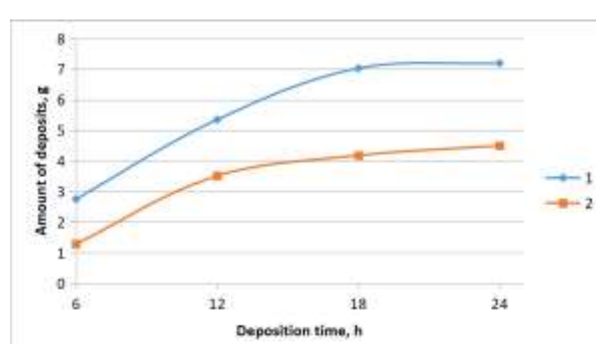


Fig. 2. Dynamics of wax deposition for oil I (a) and II (b): 1 – oil without depressant; 2 – oil with depressant. Oil/Cold Finger temperature = 60/15 ºC

For oil I without a depressant, the amount of precipitated deposits increases rapidly until approximately 14 h, then the deposition rate slows down as the thickness of the deposit layer increases, and after 18 h it changes insignificantly (from 6.78 to 7.02 g per 6 h). For oil II without a depressant, a more linear dependence of the deposits' amount on the deposition time is observed, and after 18 h, the deposition rate becomes practically constant. The depressant present in oil not only reduces the amount of deposits (Table 3), but also slows down their growth rate (Fig. 2, *curves* 2). The bulk of the deposits for oil I and oil II is formed in approximately 12 and 14 h, respectively.

The significant slowdown in deposition during the last 6 hours of the experiment is explained by the fact that the layer of wax formed on the surface of the Cold Finger begins to act as thermal insulation, oil loses its ability to dissipate heat outward, and this leads to a decrease in the effective temperature difference. Accordingly, the ability of paraffin crystals to continue depositing decreases. A similar trend is observed in works.[9,24,34]

4. Conclusions

The formation of wax deposits in oil pipelines is influenced by a complex interaction of various factors, including the temperature of crude oil, the temperature differential between the oil and the pipeline walls, the deposition time, and the physical and chemical characteristics of crude oil. Oils from the Dolynske and Boryslavske fields (western region of Ukraine) were examined to determine the amount of resins, asphaltenes and paraffins. The relationship between wax deposition and the factors that influence it was established. It was shown that as the temperature differential decreases, the amount of deposits decreases by 2.2–2.4 times. It was determined for the first time that the Dodiflow 5236 depressant, which was effective in improving the low-temperature properties of Dolyna and Boryslav oils, also inhibits wax deposits of the mentioned oils. Moreover, in the presence of Dodiflow 5236, a change in the structure of paraffin crystals was observed. The depressant effectiveness was found to be 35-60 % for Dolyna oil and 33-54% for Boryslav oil, depending on the temperature differential. The presence of a depressant in oil not only reduces the amount of deposits but also slows down their growth rate.

The results obtained may be useful during the actual transportation of high waxy oils at low ambient temperatures.

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Received: October 01, 2025 / Revised: October 19, 2025 / Accepted: October 20, 2025

ВПЛИВ ДЕПРЕСОРА НА ІНГІБІТУВАННЯ ПАРАФІНИСТИХ ВІДКЛАДІВ НАФТ ЗАХІДНОГО РЕГІОНУ УКРАЇНИ

***Анотація**. Парафіністі відклади створюють значні перешкоди в забезпеченні текучості сирої нафти, оскільки викликають аномалії тиску в трубопроводі, зменшують його прохідний діаметр, призводять до штучних засмічень. Для двох нафт західного регіону України, Долинського і Бориславського родовищ, за допомогою методу холодного пальця було визначено кількість відкладів. Показано динаміку росту кількості відкладів у часовому інтервалі до 24 год. Проведено оцінювання ефективності депресора Dodiflow 5236 як інгібітора утворення відкладів. Встановлено, що депресор зменшує кількість утворених відкладів у 1,5-2,5 разів. Крім того, депресор знижує швидкість осадження парафіну і змінює структуру кристалів. Показано, що кількість відкладів, утворених із сирої нафти і нафти з депресором, залежить не тільки від фізико-хімічної характеристики нафти, але й від різниці температур між нафтою та стінкою.*

***Ключові слова**: метод холодного пальця, депресор, парафіни, відклади.*