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https://orcid.org/0000-0002-7680-7084
https://orcid.org/0000-0001-5442-4763
https://orcid.org/0009-0001-3091-970X
https://orcid.org/0000-0002-1957-5168
https://orcid.org/0009-0001-7364-0901
https://orcid.org/0000-0003-1947-2449

WAX PRECIPITATION PREDICTIVE MODEL MODIFICATION ON THE EXAMPLE OF A KAZAKHSTAN OIL

D.A. ISMAILOVA[1],
PhD, associate professor,
j.ismailova@satbayev.university

D.N. DELIKESHEVA[1],
Master, senior lecturer,
d.delikesheva@satbayev.university

A.S. BAIBEKOVA[2],
Master's student of SEPI, KBTU,
a_baibekova@kbtu.kz

A.A. ISMAILOV[2],
Professor, Candidate of Technical Sciences,
Dean of the SEPI, KBTU,
a.ismailov@kbtu.kz

G.Zh. SMAILOVA[3],
General director of NCC Petroleum Engineering Consulting LLP,
gulbagdamsm@gmail.com

A.Kh. SYZDYKOV[1],
Candidate of Technical Sciences, PhD, Professor of the Department of «Petroleum Engineering»,
a.syzdykov@satbayev.university

[1]SATBAYEV UNIVERSITY
22, Satbayev str., Almaty, 050013, Republic of Kazakhstan

[2]KAZAKH-BRITISH TECHNICAL UNIVERSITY
59, Tole bi str., Almaty, 050000, Republic of Kazakhstan

[3]"NCC PETROLEUM ENGINEERING CONSULTING" LLP
12, Dostyk dst., Almaty, 055551, Republic of Kazakhstan

Currently, the oil and gas segment of Kazakhstan is facing such a problem as paraffin deposits. The problem associated with the deposition and crystallization of paraffin causes large-scale difficulties, and models for predicting wax precipitation are limited to specific types of oil, and therefore there is a need to improve them.

The authors of this article analyzed and developed a predictive model of paraffin deposition from crude oil for Kazakhstani fields.

The work is based on the study of Lira-Galeana et al., which considers the multicomponent deposition of immiscible solids from an oil mixture - a multi-solid model. During the study, it was found out that the Lira-Galeana model uses the equation of state, the volatility properties of the components and the activity coefficient. The Poynting effect was considered at high pressures in the system.

For experimental studies, oil samples were taken from two fields. The component composition of the oil, the pour point and loss of fluidity of the oil, as well as the melting point temperatures were determined in laboratory conditions. The amount of paraffin sludge from the oil was estimated experimentally. The results obtained are fully consistent with the analytical solution. During laboratory tests, it was found that the oil from the second field was heavier, and the degree of crystallization of paraffin was more significant than that of oil from the first field under the same conditions.

Using this predictive model, the authors were able to predict the amount of solid precipitation from crude oil during the year, depending on changes in ambient temperature.

KEY WORDS: *crude oil, wax precipitation, paraffin deposits, crystallized paraffin, fugacity, predictive model*

ҚАЗАҚСТАНДЫҚ МҰНАЙ МЫСАЛЫНДА ПАРАФИН ТҮЗІЛУІН БОЛЖАУ МОДЕЛЬДІҢ МОДИФИКАЦИЯСЫ

Д.А. ИСМАИЛОВА[1], PhD, қауымдастырылған профессор, *j.ismailova@satbayev.university*
Д.Н. ДЕЛИКЕШЕВА[1], магистр, аға оқытушы, *d.delikesheva@satbayev.university*
А.С. БАЙБЕКОВА[2], ҚБТУ ШЭиНГИ магистранты, a_baibekova@kbtu.kz
А.А. ИСМАИЛОВ[2], профессор, техника ғылымдарының кандидаты, ҚБТУ энергетика және мұнай-газ индустриясы мектебінің деканы, *a.ismailov@kbtu.kz*
Г.Ж. СМАИЛОВА[3], "Petroleum Engineering Consulting ҰБО" ЖШС Бас директоры, *gulbagdamsm@gmail.com*
А.Х.СЫЗДЫКОВ[1], техника ғылымдардың докторы, PhD докторы, «мұнай өндірісі» кафедрасының профессоры, SU, *a.syzdykov@satbayev.university*

[1]SATBAYEV UNIVERSITY
Қазақстан Республикасы, 050013, Алматы қ., Сәтбаев қ., 22

[2]ҚАЗАҚСТАН-БРИТАН ТЕХНИКАЛЫҚ УНИВЕРСИТЕТІ
Қазақстан Республикасы, 050000, Алматы қ., Төле-би қ., 59

[3]"PETROLEUM ENGINEERING CONSULTING" ҰБО ЖШС
Қазақстан Республикасы, 055551, Алматы қ., Достық ш.а., 12

Қазіргі уақытта Қазақстанның мұнай-газ сегменті парафинді шөгінділер сияқты проблемаға тап болып отыр. Парафиннің тұндырылуы мен кристалдануына байланысты проблема ауқымды қиындықтар туғызады, ал парафиннің тұндырылуын болжау модельдері мұнайдың белгілі бір түрлерімен шектеледі, сондықтан оларды жетілдіру қажет.

Осы мақаланың авторлары қазақстандық мұнай үшін парафиннің түсуін болжау моделін талдап дамытты.

Жұмыс Лира-Галеана және т.б. зерттеуге негізделген, ол мұнай қоспасынан араласпайтын қатты заттардың көп компонентті тұндыруын қарастырады – "multi-solid" модель. Зерттеу барысында Лира-Галеана моделі күй теңдеуін, компоненттердің құбылмалылық қасиеттерін және белсенділік коэффициентін қолданатыны анықталды. Пойнтинг әсері жүйеде жоғары қысымда қарастырылды.

Эксперименттік зерттеулер үшін екі кен орнынан мұнай сынамалары алынды. Зертханалық жағдайда мұнайдың құрамдас бөлігі, қатаю температурасы және мұнай сұйықтығының жоғалуы, сондай-ақ балқу температурасы анықталды. Мұнайдан алынған парафин шөгінділерінің мөлшері эксперименталды түрде бағаланды. Алынған нәтижелер аналитикалық шешімге толығымен сәйкес келеді. Зертханалық сынақтар екінші кен орнынан алынған мұнайдың ауыр екенін және парафиннің кристалдану дәрежесі бірінші кен орнынан алынған мұнайға қарағанда дәл осындай жағдайда маңыздырақ екенін анықтады.

Осы модельді қолдана отырып, авторлар қоршаған орта температурасының өзгеруіне байланысты жыл бойына шикі мұнайдан қатты жауын-шашынның мөлшерін болжай алды.

ТҮЙІН СӨЗДЕР: *шикі мұнай, парафинді тұндыру, парафиннің түзілуі, кристалданған парафин, құбылмалылық коэффициенті, болжау моделі*

МОДИФИКАЦИЯ МОДЕЛИ ПРОГНОЗИРОВАНИЯ ОСАЖДЕНИЯ ПАРАФИНА НА ПРИМЕРЕ КАЗАХСТАНСКОЙ НЕФТИ

Д.А. ИСМАИЛОВА[1], PhD, ассоц. профессор, *j.ismailova@satbayev.university*
Д.Н. ДЕЛИКЕШЕВА[1], магистр, старший преподаватель, *d.delikesheva@satbayev.university*
А.С. БАЙБЕКОВА[2], магистрант ШЭиНГИ КБТУ, *a_baibekova@kbtu.kz*
А.А. ИСМАИЛОВ[2], профессор, кандидат технических наук, декан ШЭиНГИ КБТУ, *a.ismailov@kbtu.kz*
Г.Ж. СМАИЛОВА[3], генеральный директор ТОО «НПЦ Petroleum Engineering Consulting», *gulbagdamsm@gmail.com*
А.Х. СЫЗДЫКОВ[1], к.т.н., PhD, профессор кафедры «Нефтяная инженерия», SU, *a.syzdykov@satbayev.university*

[1]SATBAYEV UNIVERSITY
Республика Казахстан, 050013, г. Алматы, ул.Сатпаева, 22

[2]КАЗАХСТАНСКО-БРИТАНСКИЙ ТЕХНИЧЕСКИЙ УНИВЕРСИТЕТ
Республика Казахстан, 050000, г. Алматы, ул. Толе би, 59

[3]ТОО "НПЦ PETROLEUM ENGINEERING CONSULTING"
Республика Казахстан, 055551, мкр. Достык, 12

В настоящее время, нефтегазовый сегмент Казахстана сталкивается с такой проблемой, как парафиновые отложения. Проблема, связанная с отложением и кристаллизацией парафина вызывает масштабные трудности, а модели прогнозирования осаждения парафина ограничены конкретными типами нефти, в связи с чем существует потребность в их усовершенствовании.

Авторами данной статьи была проанализирована и разработана модель прогнозирования выпадения парафина для Казахстанской нефти.

Работа основана на исследовании Лира-Галеана и др., в которой рассматривается многокомпонентное осаждение твердых несмешивающихся веществ из нефтяной смеси - мультитвердая модель. В ходе исследования было выяснено, что модель Лира-Галеана использует уравнение состояния, свойства летучести компонентов и коэффициент активности. Эффект Пойнтинга рассматривался при высоких давлениях в системе.

Для экспериментальных исследований были взяты пробы нефти с двух месторождений. В лабораторных условиях был определен компонентный состав нефти, температура застывания и потеря текучести нефти, а также температуры точки плавления. Количество парафинового осадка из нефти было оценено экспериментально. Полученные результаты полностью соответствуют аналитическому решению. В ходе лабораторных испытаний было обнаружено, что нефть со второго месторождения была тяжелее, а степень кристаллизации парафина была более значительной, чем у нефти с первого месторождения при тех же условиях.

С использованием этой модели авторы смогли спрогнозировать количество твердых осадков из сырой нефти в течение года в зависимости от изменения температуры окружающей среды.

КЛЮЧЕВЫЕ СЛОВА: *сырая нефть, осаждение парафина, образование парафина, кристаллизованный парафин, коэффициент летучести, модель прогнозирования.*

Introduction. Petroleum reservoir fluids are complex mixtures of hydrocarbons. The light fraction of these fluids contains associated gasses and low molecular weight hydrocarbons (e.g., CO_2, H_2S, N_2, C_I, C_2, C_3, n-C_4, i-C_4), while the rest of the oil contains a large number of paraffinic hydrocarbons, naphthenic and aromatic compounds whose molecular weights range from about 100 to 2000. When the temperature is lowered, the heavy hydrocarbon components in this range can crystallize and precipitate as a solid mass known as paraffin, which is deposited on the walls of a pipe or process unit. This precipitation has operational and economic implications that are important to the oil industry.

While the molecular and thermodynamic behavior of the light fraction of these fluids is relatively well known, the situation is quite different for the heavier hydrocarbon fractions, whose complexity and crude oil-dependent properties mean that the general behavior still needs to be better understood and difficult to predict.

The huge amount of crude oil in Kazakhstan contains heavy hydrocarbons that precipitate as a solid paraffin phase at low temperatures. Paraffin is a heavy component of crude oil released as a solid phase below the solidification point.

The accumulation of deposited paraffin on the pipe walls is one of the complex flow assurance problems that lead to a reduction and complete blockage of the oil flow rate due to a decrease in the flow cross-sectional area in the pipelines [1]. In addition, ground structures require higher energy consumption, and paraffin plugs cause equipment failure. Wax precipitation also increases the viscosity of the oil mixture, leading to an increase in the energy required to transport crude oil. Paraffin deposition can occur anywhere from reservoirs to ground structures and pipelines.

This work aims to develop a theoretical model for reliable prediction of paraffin deposition for Kazakhstani crude oil based on the chemical composition of hydrocarbons and thermobaric conditions. A modified thermodynamic model is developed to predict wax deposition based on a mathematical approach and an experimental procedure.

The new model is based on calculations for specific Kazakhstani oil, which will increase the prediction efficiency by developing it from experimental data.

Materials and methods. The correct approach for predicting wax deposition is justified by robust models that are valuable in the design and operation of production lines. To predict the precipitation of paraffin, two types of models are available that are applicable for calculations: solid and multisolid (*Fig.1*). The first model is based on the fact that the fallen wax phase is a solid solution (Won 1986, 1989; Hansen et al. 1988; Pedersen et al. 1991; Zuo et al. 2001; Ji et al. 2004). The second model, in turn, assumes that the wax that is deposited consists of several solid phases (Lira-Galeana et al. 1996; Pan and Firoozabadi 1997; Nichita et al. 2001; Escobar-Remolina 2006).

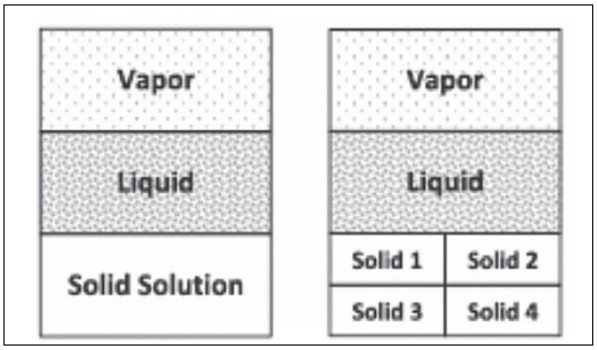

Figure 1 – **The forecasting models: solid solution and multisolid**

The WAT at which the paraffin begins to transition to a solid-state from the first minutes of cooling, is usually measured in laboratories. But the peculiarity of these thermodynamic models is the possibility of predicting the precipitation of paraffin using a modified model approach that uses various correlations to calculate a model for forecasting paraffinization.

Experimental studies. Field data were collected and analyzed from Kazakhstan's *X* and *Y* fields to conduct the experiments. Three crude oil samples were collected from each field to do precise laboratory experiments. The crude oil from two fields represented as a Black oil having different compositions. According to the obtained six samples of two different oils from the *X* and *Y* fields, laboratory experiments were carried out to determine the component composition of the oil necessary to carry out the following work tasks. In addition, the physicochemical properties of the oil sample on the surface were determined, which are necessary for calculating the multi-solid solution model.

The group composition of the oil was investigated by the SARA (saturated, aromatics, resins, asphaltene) analysis method. This method allows crude oil to be fractionated according to four main solubility classes, which are generally called SARA: saturated hydrocarbons, aromatic hydrocarbons, resins, asphaltene (*Fig.2*). The method is based on the separation of a mixture of certain components by thin-layer chromatography (TLC) on quartz rods with silicagel applied. The fractionation of crude oil is based on the solubility of hydrocarbon components in various solvents. In this experimental study were used to separate groups of compounds solvents of heptane, a mixture of toluene:heptane (80:20), a mixture of dichloromethane:methanol (95:5). The results of the group composition of SARA oil are presented in *Table 1*.

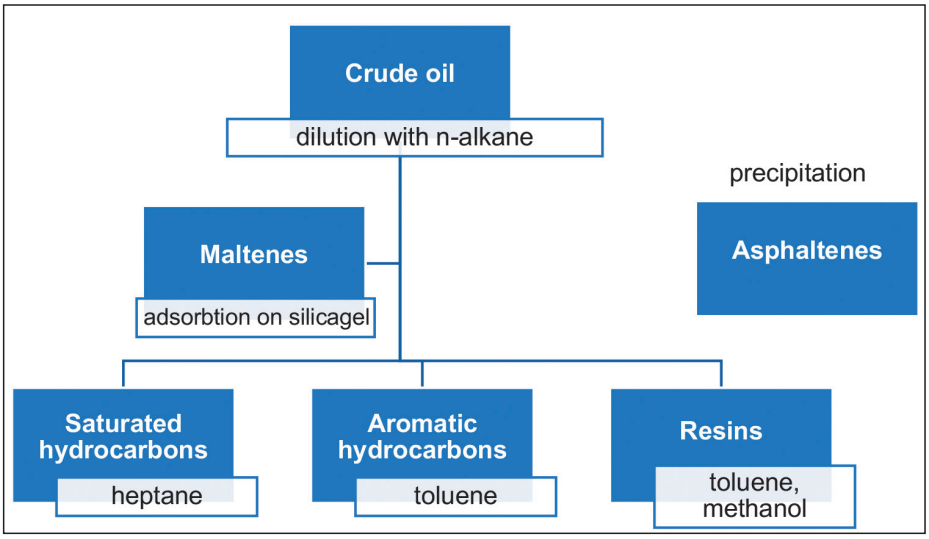

Figure 2 – **General scheme of SARA analysis**

Table 1 – **The results of the SARA oil group composition for oil samples from the Field X**

Crude oil group composition - SARA analysis				Units
Content of saturated hydrocarbons	57,56	58,11	59,10	weight %
Content of aromatic hydrocarbons	20,83	20,24	19,93	weight %
Resin content	14,75	14,91	14,87	weight %
Asphaltene content	6,86	6,74	6,10	weight %
Balance	100,00	100,00	100,00	

Table 1 – **The results of the SARA oil group composition for the oil sample from Field Y**

Crude oil group composition - SARA analysis				Units
Content of saturated hydrocarbons	61,42	60,86	62,30	weight %
Content of aromatic hydrocarbons	21,63	21,74	20,49	weight %
Resin content	12,44	12,71	12,89	weight %
Asphaltene content	4,51	4,69	4,32	weight %
Balance	100,00	100,00	100,00	

The second analysis determines the loss of fluidity and the solidification temperature. The method of operation with the "ATZ-01" device. The instrument automatically determines the state of mobility and immobility of the product without the need to remove the tube. Also, the following parameters were determined: the wax's melting point and the oil's molecular weight. The results are illustrated in *Table 3* and *4*:

Table 3 – **Results of solidification, melting point temperature, WAT and molecular weight for oil samples from Field X**

Parameters	Results			Units
Solidification and loss of fluidity temperature	19,0	20,0	20,0	°C
WAT	41,0	43,0	42,0	°C
Melting point temperature	62,0	61,0	62,0	°C
Molecular weight	273,85	270,33	266,75	g/mole

Table 4 – **Results of solidification, melting point temperature, WAT and molecular weight of oil sample from Field Y**

Parameters	Results			Units
Solidification and loss of fluidity temperature	28,0	28,0	27,0	°C
WAT	40,0	38,0	39,0	°C
Melting point temperature	65,5	65,0	66,0	°C
Molecular weight	248,87	243,18	238,55	g/mole

Figure 1 – **The forecasting models: solid solution and multi-solid**

Analytical part.

The stability criterion states that each component (i) can exist as a pure solid (solid assumption) if it is complete [6]:

$$f_i(P,T,z) - f^s_{pure\,i}(P,T) \geq 0, (i = 1,2\ldots,N) \quad (1)$$

where $f_i(P, T, z)$ is a fugacity of the component (i) with feed composition z_i.

Assuming constant temperature and constant pressure, the multi-solid model for any component (i) should satisfy the following equation:

$$f_i^v = f_i^l = f^s_{pure\,i},\ (i = 1,2\ldots,N) \quad (2)$$

Fugacity characterizes molecules' degree of escaping inclination from one state to another. Thus, in a multi-component system, if the fugacity of a component is the same in two different states, there is no net transition of molecules between the states, and they are in equilibrium.

$$f_i = \varphi_i\, z_i\, P \quad (3)$$

$$f_i^l = \varphi_i^l x_i^l P \quad (4)$$

where φ_i is a coefficient of fugacity.

Equation (1) cannot indicate the number and does not designate the components that will precipitate out. It is assumed that the process has a "continuous" character, and the following equation is derived (Escobar 2006):

$$z_i - x_i^l \left(\frac{\varphi_i^l}{\varphi i}\right) \geq 0, (i = 1,2\ldots,N) \quad (5)$$

Considering equation (2) and a model of the activity coefficient, the general form of the constant of the solid-liquid phase equilibrium is defined as follows:

$$K_i^{sl} = \frac{\varphi_i^l\, P}{\gamma_i^s f^s_{pure\,i}} = \frac{s_i}{x_i} \quad (6)$$

where $f^s_{pure\,i}$ and φ_i^l are calculated at mixture pressure at temperature; γ is an activity coefficient, s_i – solid fraction.

A complete immiscibility for all species in the solid state [20] is achieved when the pure solid ($s_i = 1$) and ideality of solid state ($\gamma_i^s = 1$) is assumed. Considering these assumptions and equation (6), they get:

$$K_i^{sl} = \frac{1}{x_i} = \frac{\varphi_i^l P}{f_{pure\ i}^s} \tag{7}$$

The equation (7) is not general - it is specific, which means that the solid state consists of several immiscible components that describe the K-factor $\left(\frac{1}{x_i}\right)$.

From the thermodynamic point of view, it could be shown as [21]:

$$\ln\left(\frac{f^s}{f^l}\right)_{pure\ i} = -\theta \tag{8}$$

where θ is calculated from following equation:

$$\theta = \frac{\Delta h_i^f}{RT}\left(1 - \frac{T}{T_i^f}\right) + \frac{1}{R}\Delta C_{pi}\left(1 - \frac{T_i^f}{T} + ln\frac{T_i^f}{T}\right) + Poynting \tag{9}$$

This equation allows us to calculate, using an iterative process, the composition of the liquid and the volatility coefficient *xi* and *φil*. Carrying out the material balance for liquid-multi-solid equilibrium: By considering the equations (7) - (9) and knowing that:

$$f_{pure\ i}^l = \varphi_{pure\ i}^l P \tag{10}$$

the following relation is derived:

$$K_i^{sl} = \frac{1}{x_i} = \frac{\varphi_i^l}{\varphi_{pure\ i}^l}\exp(\theta) \tag{11}$$

Reddy S.R. in 1986 [22] has derived the formula for determining the mole fraction of component *(i)* in the liquid phase:

$$x_i^l = exp\left[\frac{\Delta h_i^f}{T}\left(\frac{1}{T_i^f} - \frac{1}{T}\right)\right] \tag{12}$$

By comparing the equations (4) and (11), the new relation is found [17]:

$$s_i = z_i - x_i^l\left(\frac{\varphi_i^l}{\varphi_i}\right) \tag{13}$$

$$(i = 1,2..., N)$$

The parameter s_i determines the components that precipitate at a given pressure and temperature. The component precipitates if the value exceeds 0; otherwise, the component does not precipitate – the Poynting correction factor.

The fusion properties must be determined to calculate the amount of wax precipitated. First, the fusion temperature is the temperature of a solid body at which it transitions to a liquid state, and the substance formed can be either a solid or a liquid state. Second, Won's correlation was chosen as a generally accepted equation for determining the melting point of each component using the molecular weights of the components [1]. Calculation of crystallized paraffin:

At the last stage, the paraffin crystallized is calculated using the formula suggested by Escobar et al. [17]:

$$P.c.(\%) = \frac{\Sigma_j^N M_j * S_j}{(\Sigma_N^i M_i * Z_i)_{T_{WAP}}^{T_f}} * 100\% \tag{14}$$

Results and discussions. The oil samples from two Kazakhstani fields were examined in this paper. Each test tube of crude oil was characterized based on the fraction composition. The fusion properties were determined as input parameters for different formulas.

In all calculations, it was assumed that the mixture had only liquid and solid phases. Therefore, the vapor phase should have been addressed. In this context, the constant of the solid-liquid equilibrium phases K_i^{sl} is determined for each component. The values vary between 99 and 102, increasing with the number of carbon atoms and the increase of the potential solid phase.

As the pressure increases, the paraffin deposition increases. The pressure of the system was changed from 0.1 to 6 MPa. The effect of high pressure on paraffin crystallization at a constant temperature of 298.15 K is shown in the *Fig. 3* and *4*:

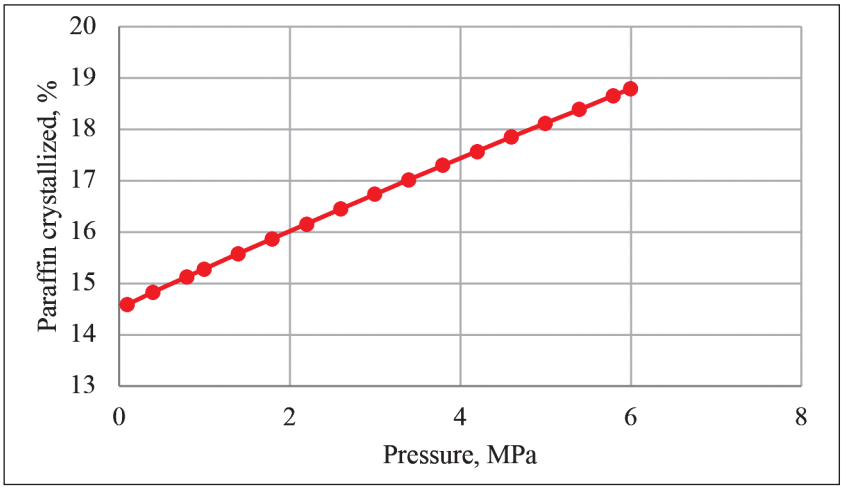

Figure 3 – **The effect of pressure on calculated paraffin crystallized results (weight %) of Oil 1, Field X, T=298.15 K**

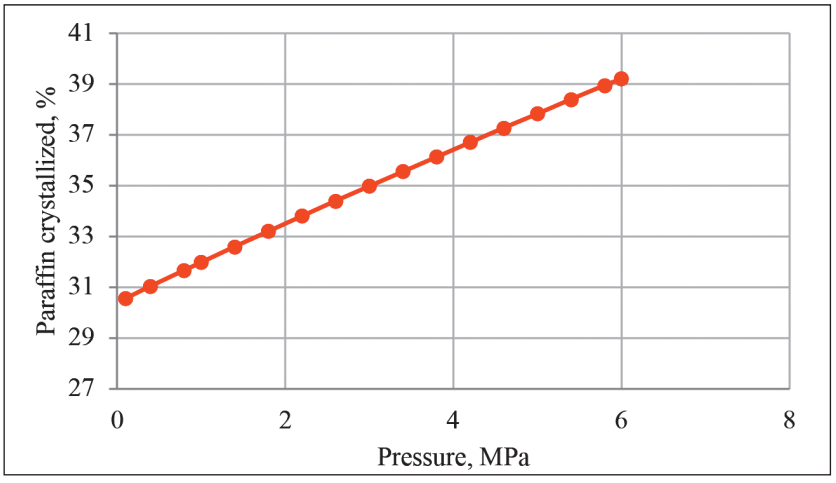

Figure 4 – **The effect of pressure on calculated paraffin crystallized results (weight %) of Oil 2, Field Y, T=298.15 K**

It is clear from the figures that the dependence on these parameters is direct and linear, but we have found that this tendency persists in both fields.

The study of the Poynting correction factor could be helpful when we cannot change the temperature range, but the pressure is under our influence. Therefore, the system pressure should be reduced as much as possible to minimize the paraffin deposition from crude oil at a constant temperature. The paraffin crystallized (by weight) was estimated for both Fields *X* and *Y*. The calculated results for three crude oil samples in each field at atmospheric pressure and temperature of 298.15 K.

Table 2 – **The calculated results of paraffin crystallized for both Fields X and Y at atmospheric pressure and temperature of 298.15 K as displayed in Table 7**

Samples	P.c., %
Field X	
1st sample	14,594
2nd sample	13,743
3rd sample	14,792
Field Y	
1st sample	26,271
2nd sample	30,560
3rd sample	24,389

In order to follow the temperature effect during the year, the amount of paraffin crystallized (in %) was calculated, assuming that the atmospheric pressure does not change. It was found that in cold weather, paraffin deposition increases, and vice versa. The coldest atmospheric temperature during the year is -9 ºC (Field *X*) and -8 ºC (Field *Y*); the warmest temperature is 34 ºC and 35 ºC, respectively. Since both fields are located in the West of Kazakhstan, the temperature change during the year is almost the same. Thus, if the system pressure is constant, the maximum paraffin crystallization from the crude oil is achieved in winter and is about 23% at Field X and 41% at Field Y. The lowest wax precipitation occurs in summer: at least 11% in Field X and 20% in Field Y as seen in table 5 and 6. Consequently, the most favorable period for production is summer when the air temperature is high.

By constructing the predictive model, we could always assume the expected amount of wax precipitation from crude oil during the year. Therefore, additional work could be done to prevent incidents.

In Field X, the composition of the oil samples and their physical properties are identical. Therefore, in the last phase, the number of paraffin depositions from three oil tubes is almost the same. On the other hand, in Field Y, the second sample showed a gradient in the amount of precipitated wax compared to the other two tubes. This fact can be attributed to the different compositions and temperatures of the turbidity and solidification points.

The above discussion suggests that the oil from Field X has similar characteristics from well to well, but this is different for the second field.

Comparison was made according to wax appearing temperatures experimental and calculated, where the calculated was equal to 317.5 K with 315.15 K experimental.

Table 5 – **The amount of paraffin crystallized at the Field X across the year, P=0.1 MPa (calculated results)**

T, °C	T, K	P.c., % (1st sample)	P.c., % (2nd sample)	P.c., % (3rd sample)
-9	264,15	22,407	21,128	22,581
-6	267,15	21,875	20,625	22,052
-3	270,15	21,313	20,094	21,492
0	273,15	20,721	19,535	20,902
3	276,15	20,099	18,946	20,282
6	279,15	19,446	18,329	19,631
9	282,15	18,763	17,683	18,95
12	285,15	18,049	17,009	18,239
15	288,15	17,304	16,305	17,496
18	291,15	16,528	15,571	16,722
21	294,15	15,721	14,808	15,916
24	297,15	14,881	14,015	15,078
27	300,15	14,009	13,19	14,208
30	303,15	13,103	12,335	13,304
33	306,15	12,164	11,447	12,367
34	307,15	11,844	11,145	12,047

Table 6 – **The amount of paraffin crystallized at the Field Y across the year, P=0.1 MPa (calculated results)**

T, °C	T, K	P.c., % (1st sample)	P.c., % (2nd sample)	P.c., % (3rd sample)
-8	265,15	39,674	46,275	36,995
-5	268,15	38,725	45,162	36,102
-2	271,15	37,723	43,986	35,159
0	273,15	37,026	43,168	34,502
3	276,15	35,935	41,889	33,476
6	279,15	34,79	40,547	32,399
9	282,15	33,592	39,141	31,272
12	285,15	32,339	37,673	30,093
15	288,15	31,032	36,14	28,864
18	291,15	29,669	34,543	27,583
21	294,15	28,251	32,88	26,249
24	297,15	26,776	31,151	24,862
27	300,15	25,243	29,355	23,422
30	303,15	23,652	27,49	21,927
33	306,15	22,002	25,557	20,376
36	309,15	20,291	23,552	18,769

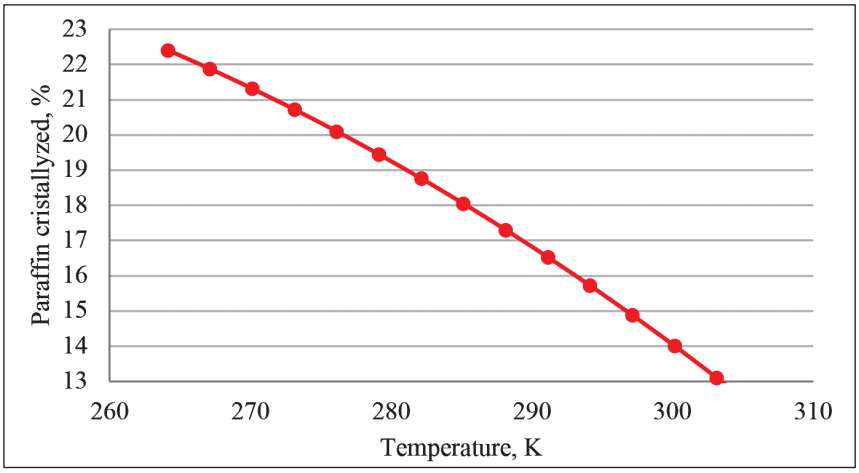

Figure 5 – **The effect of temperature on calculated paraffin crystallized results (weight %) of Oil 1, Field X. Pressure=0.1 MPa**

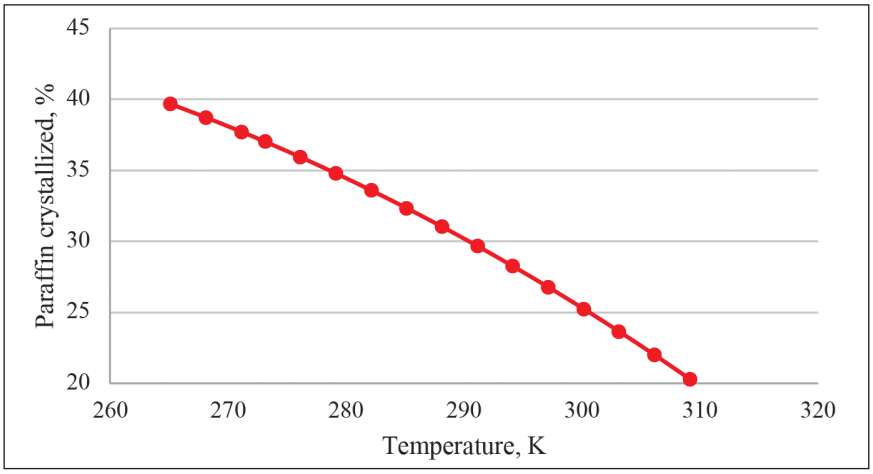

Figure 6 – **The effect of temperature on calculated paraffin crystallized results (weight %) of Oil 1, Field Y. Pressure=0.1 MPa**

Conclusion. In conclusion, it is worth noting that the problem of the formation of paraffin deposits is gaining serious proportions. This is justified by the fact that many deposits are in the late stage of development. When eliminating this problem, it is necessary to be guided primarily by the causes of this problem. After that, it is worth conducting an analysis that will be directly aimed at predicting wax deposits.

Based on the analysis results, the correlation of melting and solidification temperatures for Kazakhstani oil was changed. It is based on the intensive properties of solution components, such as their molecular weights and melting points, and external properties, such as mass fractions. In other words, the independent variables are solute weight fraction, solute molecular weight, melting point temperature, and the molecular weight of the solution. The obtained results show that correctly applied paraffin accumulation preventive methods are profitable, and the help of a multi-solid predictive model for

paraffin deposition from crude oil is undoubtedly valuable. By analyzing the existing literature and available results of laboratory studies, the multi-solid model for Kazakhstani oil was be modified.

By creating the predictive model, we can always assume the expected amount of wax precipitation from crude oil during the year.

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