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Research of the effect of a new composition against asphaltene-resin-paraffin deposits

Badanie wpływu nowej kompozycji na osady asfaltenowo-żywiczno-parafinowe

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ABSTRACT: New multifunctional compositions were prepared from chemical reagents of various purposes under laboratory conditions, and their effect on the rheological parameters of a heavy oil sample – such as freezing temperature, yield shear stress, viscosity, and oil deposits – was investigated for the first time. For comparison, the impact of the depressant additives Difron-4201 and Difron-3970, applied individually and as compositions, on the rheological parameters of the heavy oil was also examined. During the experiments, compositions with provisional names: AH-1, AH-2, AH-3, AH-4, AH-5, were applied at concentrations of 200, 300, 400, 500, 600 g/t. Difron-4201 was tested at 200, 300, 400, 500, 600, and 700 g/t, while Difron-3970 was tested at 250, 350, 450, 550, 750 g/t. The freezing temperature of the oil sample was reduced from 22°C to 5, 1, –4, –3 and –7°C due to the influence of the specified concentrations of the individual reagents and compositions. The effect of the reagents against oil deposits was investigated using the "coldfinger test" method. During the process, the amount of asphaltene-resin-paraffin deposits accumulated on the surface after 30, 60, 90, 120, 180 and 240 minutes at tube temperatures of 0, 5, 10, 20, 30°C was determined. To evaluate the effect of the reagents, the amount of oil deposits formed during the specified time and at given temperature without reagents was also measured. Using a known mathematical dependence, the effect was calculated for each concentration of the individual reagents and compositions at 5°C in the "coldfinger test". The highest effects were observed at concentrations of 700 g/t for Difron-4201, 750 g/t for Difron-3970, and 600 g/t for AH-1, AH-2, AH-3, AH-4, AH-5, with reduction rates of 75, 70, 78, 82, 87, 84 and 92%, respectively. The composition of oil deposits formed at each temperature over 240 minutes was also determined. The results showed that as the temperature increased, the amount of asphaltene and resin increased, while the paraffin content decreased. The values of rheological parameters K, n, τ_0 of the oil sample, both with and without reagents, were determined according to Herschel-Bulkley model. Thus, as a result of numerous laboratory tests, the optimal reagent concentrations were found to be 700 g/t for Difron-4201, 750 g/t for Difron-3970 and 600 g/t for AH-series compositions. It was revealed that, compared to individual additives, the compositions were more effective, with the AH-5 composition showing the highest efficiency.

Key words: composition, reagent, freezing temperature, yield stress, oil deposits, "cold finger".

STRESZCZENIE: W warunkach laboratoryjnych opracowano nowe środki wielofunkcyjne z odczynników chemicznych o różnym przeznaczeniu, a następnie po raz pierwszy zbadano ich wpływ na parametry reologiczne próbki ciężkiej ropy naftowej, takie jak temperatura krzepnięcia, graniczne naprężenie ścinające, lepkość oraz powstawanie osadów ropy. Dla porównania przeanalizowano wpływ dodatków depresatorów Difron-4201 i Difron-3970, stosowanych samodzielnie, jak i w postaci kompozycji, na parametry reologiczne ciężkiej ropy. W eksperymentach zastosowano mieszaniny o nazwach umownych: AH-1, AH-2, AH-3, AH-4, AH-5 w stężeniach 200, 300, 400, 500, 600 g/t. Środek Difron-4201 testowano w stężeniach 200, 300, 400, 500, 600 i 700 g/t, a Difron-3970 w stężeniach 250, 350, 450, 550, 750 g/t. Pod wpływem określonych stężeń zarówno pojedynczych odczynników, jak i kompozycji, temperatura krzepnięcia próbki ropy została obniżona z 22°C do 5, 1, –4, –3 oraz –7°C. Wpływ odczynników na powstawanie osadów ropy oceniano metodą „zimnego palca" (ang. *coldfinger*). Określano masy osadów asfaltenowo-żywiczno-parafinowych wytrąconych na powierzchni po 30, 60, 90, 120, 180 i 240 minutach przy temperaturach chłodzonej rurki wynoszących 0, 5, 10, 20, 30°C. Celem dokonania oceny wpływu odczynników zmierzono także masy osadów powstałych w określonym czasie i w danej temperaturze bez użycia dodatków. W oparciu o znaną zależność matematyczną obliczono skuteczność działania odczynników i mieszanin w temperaturze 5°C. Największy efekt usuwania osadów odnotowano dla stężeń: 700 g/t dla Difron-4201, 750 g/t dla Difron-3970 oraz 600 g/t dla środków AH-1, AH-2, AH-3, AH-4, AH-5, a wartości te wynosiły odpowiednio 75, 70, 78, 82, 87, 84 i 92%. Określono także skład osadów powstałych w każdej temperaturze po 240 minutach. Wyniki wykazały, że wraz ze wzrostem temperatury zwiększała się zawartość asfaltenów i żywic, a zmalała zawartość parafin. Wartości parametrów reologicznych K, n i τ_0 próbki ropy, zarówno bez odczynników, jak i z dodatkiem odczynnika, zostały wyznaczone zgodnie z modelem Herschela–Bulkleya.

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W wyniku licznych testów laboratoryjnych ustalono, że optymalne dawki wynoszą: 700 g/t dla dodatku Difron-4201, 750 g/t dla dodatku Difron-3970 i 600 g/t dla kompozycji z serii AH. Wykazano, że mieszaniny są skuteczniejsze niż pojedyncze dodatki, przy czym najwyższą efektywność osiągnęła kompozycja AH-5.

Słowa kluczowe: kompozycja, odczynnik, temperatura krzepnięcia, granica plastyczności, osady olejowe, metoda „zimnego palca".

Introduction

The formation of oil deposits on the internal surfaces of oil field equipment during the operation, development, preparation for transportation, storage, and transportation of the extracted product, along with efforts to mitigate this issue, constitutes one of the primary challenges (Akramov and Yarkeeva, 2017).

Currently, more than half of the oil extracted from oilfields in developed countries with well-established oil industries is heavy oil (Gurbanov and Gasimzade, 2003a). In formation conditions, oil components exist in a dissolved form within the oil-dispersed system (Gasimzade, 2024). A change in the thermodynamic state of the flow during oil extraction leads to oversaturation with asphaltene-resin-paraffin components, resulting in their crystallization and deposition on the inner surfaces of oilfield equipment (Gurbanov and Gasimzade, 2023b).

In general, the primary factors influencing the formation of oil deposits include environmental temperature conditions, production rate and pressure of flowing gas-oil mixture, the quantity of heavy components, physical and chemical properties, flow regime, pipeline diameter, surface roughness, the presence of water, gas, and mechanical impurities in the oil (Nikolaev and Plotnikova, 2023).

Investigations into the mechanism of oil deposit formation have shown that a complex set of physicochemical factors underlies structure formation. Numerous studies have demonstrated that the basis of such formation is the cooling of the flow due to changes in temperature, which depends on the thickness of the boundary layer (Nasibov et al., 2011). If there is no temperature difference between the pipelines and the flow, adhesion does not occur on the pipe walls. Depending on the characteristics of heavy oil wells, deposits are observed on various parts of the inner surface of mining equipment during oil extraction and transportation (Matiev et al., 2018).

Despite extensive research and the development of equipment and technologies, this problem remains relevant (Miller et al., 2021). Among the known mechanical, physical, and chemical methods used to combat oil deposits on the internal surfaces of oilfield facilities, the chemical method is considered the most effective (Mingalev et al., 2022). In this method, various chemical reagents are injected into the inner surface of the oilfield facilities. These reagents should be selected so that they are inexpensive, environmentally friendly, and effective against oil deposits (Ivanova et al., 2022). The main advantages of using chemical reagents against oil deposits include the prevention of deposition through low-dose injection, long-term effectiveness, simplicity of technology, and the potential for automation of technological processes (Khidr, 2011). It should be noted that while some reagents exhibit a positive effect against deposit formation in one specific heavy oil, they may be less effective or ineffective in others (Poletaeva et al., 2019). Therefore, no single reagent can be considered universally effective for all oil types (Gurbanov and Gasimzade, 2024). The variation in reagent performance is explained by differences in composition and technology used for their introduction into the oil. Their application involves general technological stages – heating, preparation of liquid solutions, and dosing into the oil. Consequently, it is necessary to take into account the variability in oil response to reagents from a technological standpoint (Gurbanov et al., 2024). In other words, for each type of heavy oil, an effective chemical reagent must first be selected experimentally under laboratory conditions. When determining the optimal characteristics of chemical reagents, it is also economically essential to select the minimum concentration that provides the maximum effect.

The objective of this research is to study the multifunctional properties of new compositions on parameters of heavy oil.

Methodology

The effect of the depressant additives Difron-4201 and Difron-3970, as well as AH-series compositions, on the rheological parameters of a heavy oil sample was investigated. The heavy oil sample used had a paraffin content of 9.1%, an asphaltene content of 5.6%, a resin content of 18.5%, a density of 967 kg/m^3, a viscosity of 2164 mP·s, and a freezing temperature of 22°C was used.

The efficiency of the individual reagents and compositions against asphaltene-resin-paraffin deposits was examined using the "coldfinger test" method (Jennings and Weispfenning, 2005). It should be noted that this method particularly well-suited to simulating mining conditions and allows for the accurate evaluation of the effectiveness of chemical reagents against oil deposits, both qualitatively and quantitatively. Furthermore, the method supports the development of application technology for the effective agents developed for paraffin deposit inhibition.

Using the "coldfinger test" method under laboratory conditions enables evaluation of reagent efficiency by measuring changes in the amount of oil deposits collected on the cold finger surface as a result of variations in the freezing temperature caused by the action of chemical reagents.

During the "coldfinger test", the procedure was carried out in the following sequence. First, a crude oil sample with a volume of 1.5 l was poured into the vessel. To prevent the initial formation of paraffin crystals, the oil was heated to 60°C and maintained at this temperature using a contact thermometer and thermostat.

When the experiments began, the mixer in the Dewar vessel was activated. The tests were continued by reducing the temperature to 0°C in the second thermostat. The oil sample was maintained under these conditions for 20 minutes, after which the amount of paraffin accumulated on the "coldfinger" surface was determined by weighing it on an analytical scale.

Subsequent experiments were conducted at temperatures of 0, 5, 10, 20 and 30°C. Predetermined amounts of reagents were added to the oil samples, and the procedure was repeated in the same sequence. At the end of the experiments, the amount of asphaltene-resin-paraffin deposits collected on the "coldfinger" at different temperatures was again determined by weighing on an analytical scale. In addition, the effect of the reagents on the freezing point and other rheological parameters of the oil sample was investigated using established methods (RD 39-3-812-82, 1982).

The sample names, content ratios, and component compositions of the new formulations developed for laboratory research purposes are presented in Table 1.

Table 1. Sample names and components of the developed compositions

Tabela 1. Nazwy próbek i składniki opracowanych kompozycji

Name	Component composition	Content ratio
AH-1	Difron 4201 + Difron 3970	1:1
AH-2	Difron 4201 + ND-12	3:1
AH-3	Difron 3970 + ND-12	3:1
AH-4	Difron 4201 + ND-12 + 50 g chloroprene	3:1 + 50 g
AH-5	Difron 3970 + ND-12 + 50 g chloroprene	3:1 + 50 g

Difron-4201 and Difron-3970, which are components of the compositions, are depressant additives produced by the Russian Federation company "EKOS" and are used to modify the rheological parameters of heavy oils. ND-12 is produced in the Republic of Azerbaijan and is a demulsifier used for the treatment of stable water-oil emulsions.

Results and discussion

First, the impact of new compositions and individual reagents on the freezing temperature of the high-paraffin oil sample was investigated under laboratory conditions. Experiments were conducted based on established methodology, using different reagent concentrations to determine the optimal dosage. Thus, concentrations of 200, 300, 400, 500, 600 g/t of Difron-4201 additive, 250, 350, 450, 550, 650, 750 g/t of Difron-3970 additive, and 200, 300, 400, 500 and 600 g/t of the compositions were tested. The results of these experiments are presented in Figures 1–7.

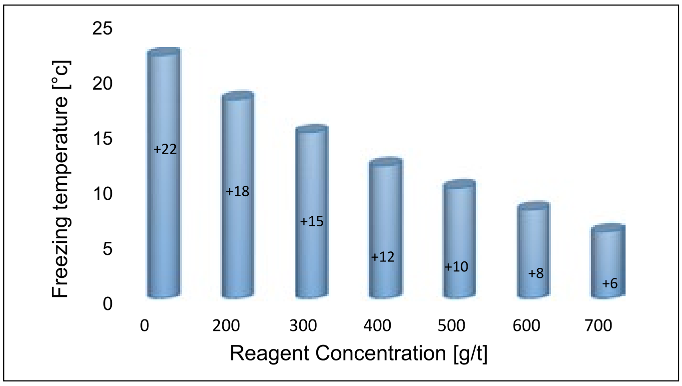

Figure 1. Effect of Difron-4201 additive on oil freezing temperature

Rysunek 1. Wpływ dodatku Difron-4201 na temperaturę krzepnięcia ropy naftowej

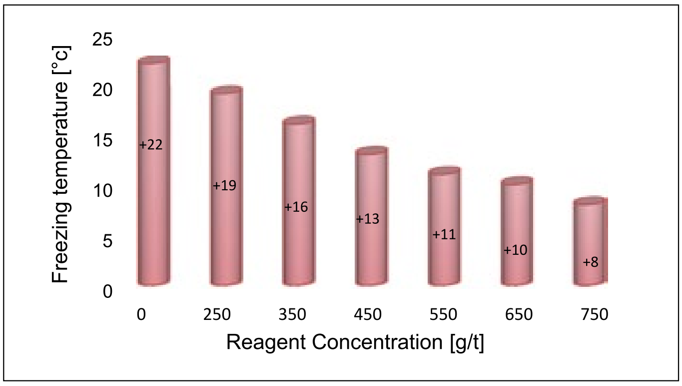

Figure 2. Effect of Difron-3970 additive on oil freezing temperature

Rysunek 2. Wpływ dodatku Difron-3970 na temperaturę krzepnięcia ropy naftowej

As shown in Figures 1–7, the freezing temperature of the investigated oil sample decreased from 22°C to 18, 15, 12, 10, 8 and 6°C with the addition of Difron-4201 at concentrations of 200, 300, 400, 500, 600,700 g/t, respectively. Similarly, with Difron-3970 at 250, 350, 450, 550, 650, 750 g/t, the freezing temperature decreased to 19, 16, 13, 11, 10 and 8°C, respectively. These results indicate that, of the two depressant

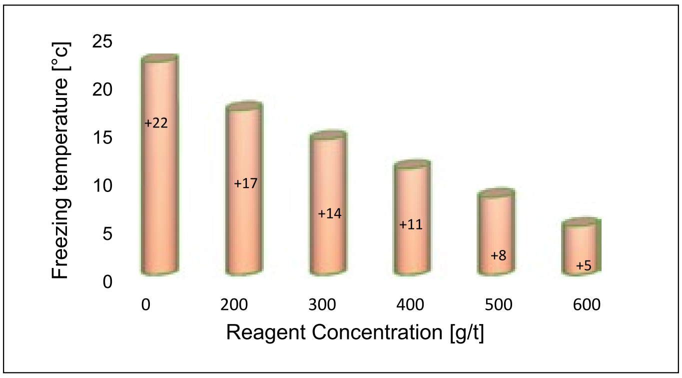

Figure 3. Effect of AH-1 composition on oil freezing temperature

Rysunek 3. Wpływ kompozycji AH-1 na temperaturę krzepnięcia ropy naftowej

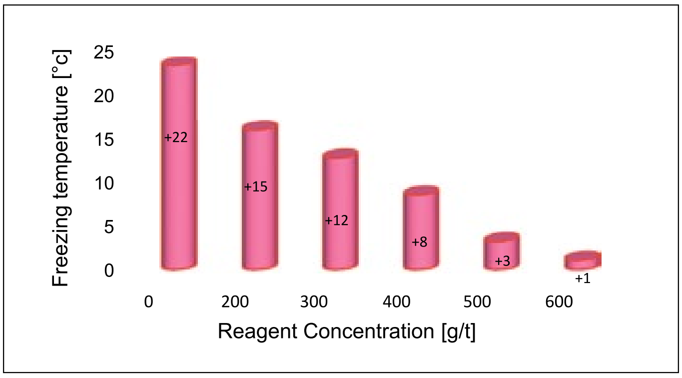

Figure 4. Effect of AH-2 composition on oil freezing temperature

Rysunek 4. Wpływ kompozycji AH-2 na temperaturę krzepnięcia ropy naftowej

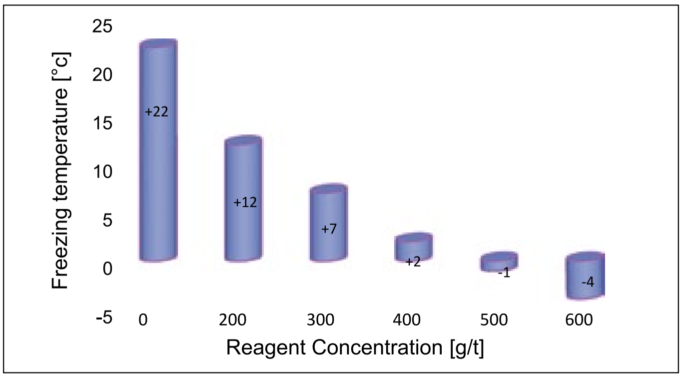

Figure 5. Effect of AH-3 composition on oil freezing temperature

Rysunek 5. Wpływ kompozycji AH-3 na temperaturę krzepnięcia ropy naftowej

additives tested, Difron-4201 had a stronger effect on the freezing temperature of oil.

The AH-series compositions were found to be more effective than the individual depressant additives. Their effects on the freezing temperature of the heavy oil sample can be summarized as follows: with concentrations of 200–600 g/t of AH-1, AH-2, AH-3, AH-4 and AH-5 compositions, the freezing temperature varies between 17–5°C, 15–1°C, 12–(–4)°C,

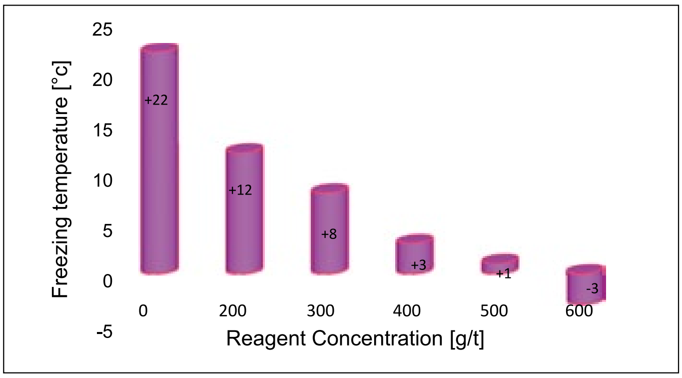

Figure 6. Effect of AH-4 composition on oil freezing temperature

Rysunek 6. Wpływ kompozycji AH-4 na temperaturę krzepnięcia ropy naftowej

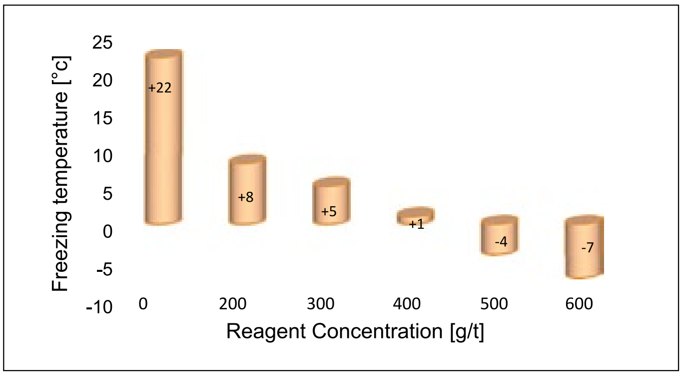

Figure 7. Effect of AH-5 composition on oil freezing temperature

Rysunek 7. Wpływ kompozycji AH-5 na temperaturę krzepnięcia ropy naftowej

12–(–3)°C, 8–(–7)°C, respectively. The optimal concentration for these compositions was 600 g/t, with AH-5 exhibiting the greatest effect at this concentration.

The effect of the individual reagents Difron-4201, Difron-3970, as well as the AH-series compositions, against paraffin deposits at different temperatures and exposure times were also studied. This part of the research was conducted under laboratory conditions using the "coldfinger test" method. First, the amount of asphaltene-resin-paraffin deposits accumulated on the "coldfinger" surface without any reagents was measured at 0, 5, 10, 20 and 30°C, at intervals of 30, 60, 90, 120, 180, and 240 minutes. After analyzing the results of multiple experiments, the processed data are presented in Table 2.

Table 3 presents the component composition of oil deposits accumulated on the cold surface at temperatures of 0, 5, 10, 20 and 30°C over a 240-minute period. As shown in the Table 3, as the temperature increases, the percentage of asphaltene and resin components in oil deposits increases, while the paraffin component decreases.

The amount of oil deposits was also determined in the oil samples to which the optimal concentrations of Difron-4201, Difron-3970, and AH-series compositions has been added, using

Table 2. Amount of deposits accumulated on the surface of the "coldfinger"

Tabela 2. Masa wytrąconego osadu zgromadzonych na powierzchni chłodzonej rurki

Time [minute]	"Coldfinger" temperature				
	0°C	5°C	10°C	20°C	30°C
	mass of ARPD[g]				
0	0.109	0.095	0.084	0.076	0.074
30	0.165	0.145	0.107	0.101	0.074
60	0.225	0.175	0.142	0.130	0.068
90	0.295	0.195	0.165	0.145	0.072
120	0.335	0.235	0.174	0.165	0.076
180	0.340	0.255	0.940	0.167	0.082
240	0.355	0.265	0.196	0.180	0.076

Table 3. Component composition of deposits

Tabela 3. Skład wytrąconych osadów

Temperature [°C]	Component composition of deposit [mass %]		
	asphaltene	resin	paraffin
0	20	44	36
5	24	46	30
10	27	48	25
20	30	50	20
30	33	51	16

the "coldfinger test" method. The efficiency of the individual reagents and compositions at 5°C was calculated using the following expression:

$$K = \frac{m_1 - m_2}{m_1} 100\% \text{ [mass\%]} \quad (1)$$

where:

K – reagent efficiency,

m_1 – mass of asphaltene-resin-paraffin deposits (ARPD) in an reagent-free medium,

m_2 – mass of ARPD in the medium containing the reagent.

Table 4 shows the calculated efficiencies of the reagents and new compositions against paraffin deposits at 5°C using the "coldfinger test" method, at the optimal concentration rates.

The dependence of reagent efficiency for Difron-4201, Difron-3970, and new compositions (AH-1, AH-2, AH-3, AH-4, AH-5) against paraffin deposits at 5°C, as determined using the "coldfinger test" method, and their optimal concentration was given in Figure 8. As shown, the efficiency of Difron-4201 is 75%, Difron-3970 is 70%, and the efficiencies of AH-1, AH-2, AH-3, AH-4, AH-5 compositions are 78%, 82%, 87%, 84% and 92%, respectively. These results demonstrate that AH-5 composition exhibits the highest efficiency against paraffin deposits.

The protection effect of individual reagents and compositions against paraffin deposits in intermediate concentrations at 5°C is given in Table 5.

It is known from the experimental results given in Table 5 that the effects of AH-1, AH-2, AH-3, AH-4, AH-5 reagents against paraffin deposits in the concentration ranges of 200–500 g/t are 55–74%, 57–77%, 60–83%, 58–79%, 65–88%. In the concentration range of 200–600 g/t of Difron-4201 reagent

Table 4. Effectiveness of reagents against the ARPD formation under laboratory conditions using the "coldfinger test" method at 20 ± 1°C ambient temperature and 0°C cooling surface, with reagent concentrations ranging from 600 to 750 g/t

Tabela 4. Skuteczność odczynników przeciwko tworzeniu się osadów asfaltenowo-żywiczno-parafinowych (ARDP) w warunkach laboratoryjnych przy użyciu metody "zimnego palca" w temperaturze otoczenia 20 ±1°C i powierzchni chłodzącej 0°C, przy stężeniach odczynników w zakresie od 600 do 750 g/t

Reagent concentration [g/t]		Reagent efficiency [mass %]
Difron-4201	700	75
Difron-3970	750	70
AH-1	600	78
AH-2	600	82
AH-3	600	84
AH-4	600	87
AH-5	600	92

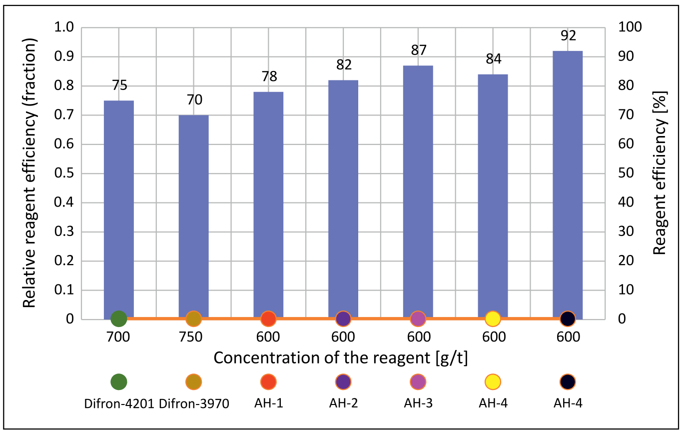

Figure 8. The effect of reagents against paraffin deposits at optimal concentration under laboratory conditions using the "coldfinger test" method at 20 ±1°C ambient temperature and 0°C cooling surface, with reagent concentrations ranging from 600 to 750 g/t

Rysunek 8. Wpływ odczynników na wytrącanie się osadów parafinowych przy optymalnym stężeniu w warunkach laboratoryjnych przy użyciu metody „zimnego palca" w temperaturze otoczenia 20 ±1°C i powierzchni chłodzącej 0°C, przy stężeniach odczynników w zakresie od 600 do 750 g/t

Table 5. Effect of reagents in different concentrations against paraffin deposits under laboratory test conditions

Tabela 5. Wpływ odczynników w różnych stężeniach na usuwanie osadów parafinowych w warunkach testu laboratoryjnego

Reagent	Concentration [g/t]	Effect [%]
Difron-4201	200	52
	300	56
	400	62
	500	67
	600	71
Difron-3970	250	49
	350	53
	450	62
	550	65
	650	68
AH-1	200	55
	300	59
	400	67
	500	74
AH-2	200	57
	300	62
	400	71
	500	77
AH-3	200	60
	300	65
	400	72
	500	83
AH-4	200	58
	300	64
	400	73
	500	79
AH-5	200	65
	300	77
	400	83
	500	88

and in the concentration range of 250–550 g/t of Difron-3970 reagent, the effect value varies between 52–71% and 49–68%, respectively.

The rheological parameters (K, n, τ_0) of the oil sample without adding the reagent were determined according to the Herschel-Bulkley model and are given in Table 6.

As it can be seen from the Table 6, the investigated oil sample exhibits non-Newtonian behavior with no fluidity at 15°C. It becomes a flowing non-Newtonian fluid between 20 and 25°C, and transitions to Newtonian fluid behavior from 50°C. After adding the optimal consumption rate of the AH-5 composition (600 g/t) to the investigated oil sample, the rheological parameters K, n, τ_0 were determined and the obtained results are presented in Table 7.

As seen from the Table 7, the oil sample with the optimal concentration of AH-5 composition regains its fluidity starting from 0°C. Starting from 15°C, the oil changes from a non-Newtonian fluid to a Newtonian one and its rheological parameters improve.

It should be noted that the compositions developed were formulated using reagents for various purposes. The oil sample used in the study is a heavy oil, which typically contains high percentages of asphaltenes, resins, and paraffin components. Due to the ease with which such oils easily form structures, they significantly influence rheological behavior by forming sediment. The compositions included emulsifier and depressant additives, which reduce viscosity by separating the water phase in the heavy oil and simultaneously removing salts. They also affect the surface tension between asphaltene, resin, and paraffin components, preventing structuring by forming a protective coating around these components. As a result, even if the high-molecular components are in Brownian motion, they cannot combine, thereby inhibiting structuring. It should also be noted that the AH-4 and AH-5 compositions contain chloroprene solvent, and their effect is

Table 6. Rheological parameters of the oil sample

Tabela 6. Parametry reologiczne próbki ropy naftowej

Temperature [°C]	Yield shear stress, τ_0 [Pa]	Consistency, K [Pa·s]	Non-Newtonian exponent, n	Note
0	76.1000	71.910	0.34	non-Newtonian fluid, does not flow, glassy state
5	53.6100	22.010	0.40	
10	38.4100	11.810	0.45	non-Newtonian fluid, non-flowing, glassy mass
15	18.8100	6.340	0.54	
20	10.6100	2.650	0.57	non-Newtonian fluid, flows, but does not comply with GOST
25	1.3500	1.370	0.73	non-Newtonian fluid, flows
30	0.1800	0.160	0.79	non-Newtonian fluid, flows
35	0.0024	0.038	0.84	
50	0.0015	0.013	1.03	Newtonian fluid, flows

Table 7. Rheological parameters of the oil sample at optimal concentration of AH-5 composition
Tabela 7. Parametry reologiczne próbki ropy naftowej przy optymalnym stężeniu kompozycji AH-5

Temperature [°C]	Yield shear stress, τ_0 [Pa]	Consistency, *K* [Pa·s]	Non-Newtonian exponent, *n*	Note
0	3.6100	3.290	0.80	non-Newtonian fluid, flows
5	1.9200	2.350	0.84	
10	1.0000	1.490	0.89	
15	0.0480	0.380	0.99	Newtonian fluid, flows
20	0.0310	0.140	0.99	
25	0.0120	0.063	0.99	
30	0.0090	0.054	1.00	
35	0.0070	0.036	1.00	
50	0.0009	0.005	1.04	

greater than that of the other three compositions. This is due to the strong softening effect of chloroprene, which reduces solid particles to a very small size, thereby lowering the colloidal degree of the oil.

Conclusion

1. New compositions with provisional names AH-1, AH-2, AH-3, AH-4, AH-5 were prepared in the laboratory for the first time using different proportions of reagents designed for different purposes. Their effects on certain rheological parameters of a heavy oil sample were investigated. Difron-4201 and Difron-3970 depressant additives were also used as individual reagents for comparative analysis.
2. In the study of the effects of individual additives and AH-series compositions on the freezing point, yield shear stress, viscosity, and paraffin deposits of the oil sample, optimal concentrations were determined, namely 700 g/t for Difron-4201, 750 g/t for Difron-3970, and 600 g/t for AH-1, AH-2, AH-3, AH-4, AH-5 compositions.
3. Analysis of the results of numerous laboratory experiments showed that the compositions exhibited a greater effect than the individual reagents. The highest effect was observed for the AH-5 composition.

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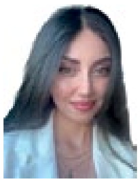

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