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Original article

Investigation of Chemical Formulations for The Removal of Asphaltene–Resin–Paraffin Deposits

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ABSTRACT

Background: This article presents the results of laboratory investigations of newly developed high-performance formulations intended for the removal of asphaltene–resin–paraffin deposits (ARPD) formed during oil production and transportation. ARPD are solid organomineral deposits composed mainly of paraffins, asphaltenes, resins, and mechanical impurities that accumulate on the internal surfaces of oilfield equipment, thereby reducing its productivity and causing operational problems. This issue is particularly relevant for the oil fields of the Mangystau region, where crude oils are characterized by a high paraffin content.

Aim: To develop an effective, readily available, and low-toxicity formulation for the dissolution and removal of solid ARPD.

Materials and methods: A comprehensive set of laboratory studies was conducted to evaluate the dissolving, removing, and cleaning performance of the developed formulations in accordance with the approved methodology. The composition of ARPD samples was investigated in accordance with the applicable regulatory documents (GOST).

Results: Laboratory experiments conducted at 25°C and 60°C demonstrated that temperature significantly affects both the dissolution rate and the extent of ARPD removal. Formulations containing more than 6 wt% surfactants achieved ARPD dissolution and removal efficiencies of up to 100%. Even at lower temperatures of 25°C, ARPD removal efficiency exceeded 98%, indicating that the developed formulations are suitable for application across a wide temperature range.

Conclusion: Within the framework of this study, the composition of paraffinic-type ARPD samples collected from different sections of the investigated field was analyzed. Based on the obtained data, hydrocarbon-based formulations were developed using a pentane–hexane fraction (PHF) as the solvent, with the addition of nonionic and anionic surfactants (OP-10 and sulfonol). These components provide a synergistic effect in dissolving and dispersing the deposits. The proposed formulations demonstrated high efficiency in the destruction and removal of solid deposits within a short period of time, contributing to reduced operating costs and improved operational reliability of oilfield equipment.

Keywords: asphaltene–resin–paraffin deposits; hydrocarbon solvents; surfactants; solvent formulations; dissolution efficiency; removal efficiency; detergency; pentane–hexane fraction; oilfield deposits.

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Оригинальное исследование

Исследование композиционных составов для удаления асфальтосмолопарафиновых отложений

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АННОТАЦИЯ

Обоснование. В статье представлены результаты лабораторных исследований разработанных эффективных композиций, предназначенных для удаления асфальтосмолопарафиновых отложений (далее – АСПО), образующихся в процессе добычи и транспортировки нефти. АСПО представляют собой твёрдые органоминеральные осадки, состоящие преимущественно из парафинов, асфальтенов, смол и механических примесей, которые откладываются на внутренних поверхностях нефтепромыслового оборудования, снижая его производительность и вызывая осложнения в эксплуатации. Особенно актуальна данная проблема для месторождений Мангистауского региона, где нефть характеризуется повышенным содержанием парафинов.

Цель. Проведение научных исследований, направленных на разработку эффективной композиции для растворения и удаления твердых отложений АСПО, обладающей доступностью и не токсичностью.

Материалы и методы. Комплекс лабораторных исследований проводился по оценке растворяющей, удаляющей и моющей способности разработанных составов в соответствии с утверждённой методикой. Составы АСПО исследованы согласно утвержденным нормативным документам (ГОСТ).

Результаты. Результаты лабораторных исследований при температурах 25°C и 60°C показали, что температурный режим оказывает существенное влияние на скорость и глубину растворения АСПО. Композиции, содержащие более 6% поверхностно-активных веществ, обеспечивают эффективность удаления и растворения АСПО до 100%. При пониженных температурах (25°C) эффективность удаления АСПО превышает 98%, что делает составы пригодными для применения в широком диапазоне температур.

Заключение. В рамках работы изучен состав АСПО парафинового типа, отобранных с различных участков исследуемого месторождения. На основе полученных данных были разработаны композиции на углеводородной основе (пентан-гексановая фракция) с добавлением неионогенного и анионного поверхностно-активных веществ (ОП-10 и сульфонол), обеспечивающих синергетический эффект растворения и диспергирования. Предложенные композиции демонстрируют высокую эффективность в разрушении и удалении твёрдых отложений за короткое время, способствуют снижению эксплуатационных затрат и повышению надёжности работы нефтепромыслового оборудования.

Ключевые слова: асфальтосмолопарафиновые отложения, растворители, поверхностно-активные вещества, композиция, составы, растворяющая способность, удаляющая способность, моющая способность, пентан-гексановая фракция.

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Түпнұсқа зерттеу

Асфальт-шайыр-парафин шөгінділерін кетіруге арналған композициялық құрамдарды зерттеу

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АНДАТПА

Негіздеу. Мақалада мұнай өндіру және тасымалдау барысында пайда болатын асфальт-шайыр-парафин (АШП) шөгінділерін кетіруге арналған тиімді композициялардың зертханалық зерттеу нәтижелері келтірілген. АШП шөгінділері – негізінен парафиндерден, асфальтендерден, шайырлардан және механикалық қоспалардан тұратын қатты органоминалдық тұнбалар болып табылады, олар мұнай кен орындарындағы жабдықтардың ішкі беттеріне шөгіп, жүйенің өнімділігін төмендетіп, пайдалану үдерісінде түрлі қиындықтар туындатады. Бұл мәселе әсіресе парафин мөлшері жоғары болатын Маңғыстау өңіріндегі кен орындары үшін өзекті.

Мақсаты. Қолжетімді әрі уыттылығы төмен қатты АШП шөгінділерін ерітуге және жоюға арналған тиімді композицияны әзірлеуге бағытталған ғылыми зерттеулер жүргізу.

Материалдар мен әдістер. Әзірленген құрамдардың еріту, жою және жуу қабілеттерін бағалау бойынша кешенді зертханалық зерттеулер бекітілген әдістемеге сәйкес жүргізілді. АШП шөгінділер құрамы бекітілген нормативтік құжаттарға (МЕМСТ) сәйкес зерттелді.

Нәтижелері. 25°C және 60°C температурада жүргізілген зертханалық тәжірибелердің нәтижелері температуралық режимнің АШП еріту жылдамдығы мен тереңдігіне айтарлықтай әсер ететінін көрсетті. Құрамында 6%-дан астам ББЗ бар композициялар АШП-ны еріту мен кетірудің 100% тиімділігін қамтамасыз етеді. Төмен температураларда (25°C) АФР-ны алып тастау тиімділігі 98%-дан асады, бұл құрамдарды әртүрлі температура диапазонында қолдануға қолайлы етеді.

Қорытынды. Зерттеу аясында кен орнының әртүрлі учаскелерінен алынған парафиндік типтегі АШП шөгінділерінің құрамы зерттелді. Алынған мәліметтер негізінде көмірсутек негізіндегі (пентан-гексан фракциясы) және беттік-белсенді заттардың (ББЗ) – бейионды (ОП-10) және аниондық (сульфонол) қоспалары бар композициялар әзірленді. Бұл компоненттер АШП құрылымын ерітіп, диспергирлеу процесінде синергетикалық әсер береді. Ұсынылған композициялар қысқа уақыт ішінде қатты шөгінділерді тиімді ерітіп, кетіру арқылы мұнай өндірісіндегі жабдықтардың сенімділігін арттырып, пайдалану шығындарын азайтуға ықпал етеді.

Негізгі сөздер: асфальт-шайыр-парафин шөгінділері, еріткіштер, беттік-белсенді заттар, композиция, құрам, еріткіштік қабілеті, кетіру қабілеті, жуу қабілеті, пентан-гексан фракциясы.

Дәйексөз келтіру үшін:

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Introduction

The formation of asphaltene–resin–paraffin deposits (ARPD) is one of the most significant challenges affecting the uninterrupted operation of oil production systems. Throughout the entire lifecycle of oil and gas field development, paraffin deposition remains one of the most critical and technologically complex issues encountered during oil production, transportation, and processing [1, 2]. ARPD are formed as a result of the precipitation of high-molecular-weight components, including paraffins, resins, and asphaltenes, from crude oil due to changes in thermobaric conditions and phase composition during fluid flow from the reservoir to the wellhead, as well as during subsequent transportation through trunk pipelines. The accumulation of ARPD within the flow passages of oilfield equipment and on the internal surfaces of pipelines leads to a reduction in the overall productivity of the production system, a decrease in the time between well interventions, and diminished pumping efficiency [3, 4]. The issue of ARPD formation is particularly acute in regions such as the Mangystau Region, where crude oils are characterized by a high paraffin content.

It is well established [4–7] that ARPD constitute a complex multicomponent system comprising asphaltenes, paraffins, resins, and mechanical impurities, as well as minor amounts of oil and water retained within the pore space of the deposits. The principal components directly involved in ARPD formation are asphaltenes and paraffins. Resins do not participate directly in the deposition process; however, they co-precipitate with asphaltenes, contributing to the stabilization of the deposit structure. Paraffins are nonpolar, non-aromatic compounds that are incapable of spontaneous self-association. They are readily soluble in light alkanes and tend to crystallize as the temperature decreases. In contrast, asphaltenes are the most polar and aromatic constituents of crude oil and exhibit a strong tendency toward self-association even in highly diluted solutions. They are insoluble in alkanes and do not crystallize upon cooling. The removal of ARPD using organic solvents is a complex physicochemical process involving the simultaneous dissolution and dispersion of deposit components. Significant differences in the solubility behavior of paraffins, asphaltenes, and resins in various classes of hydrocarbons necessitate the careful selection of solvent formulations and deposit-removal compositions, taking into account the molecular characteristics of each ARPD component.

In oilfield practice, the mitigation of ARPD formation is generally carried out through two main approaches: preventing deposit formation and removing already formed deposits. Both approaches are implemented using physical and chemical treatment methods [8]. The use of solvents for the removal of already formed solid ARPDs is one of the most well-established and widely applied methods for enhancing oil production, transportation, and processing operations [9].

Patent studies reported in [10, 11] describe various formulations developed for ARPD removal. However, their application is associated with several limitations, including insufficient removal efficiency (less than 90% with respect to ARPD), high process duration and energy consumption, as well as considerable cost resulting from the limited availability of chemical reagents incorporated in the formulations. These reagents are generally petroleum-derived products or intermediates with high added value. An additional drawback is the use of toxic aromatic hydrocarbons as solvents.

Therefore, the development of efficient multicomponent formulations capable of exerting a comprehensive effect on all ARPD constituents at reduced temperatures and with minimal operating costs remains an important and relevant research challenge.

Materials and methods

To address the problem of ARPD dissolution and removal, a formulation based on a pentane–hexane fraction (PHF), a nonionic surfactant, and an anionic surfactant (sulfonol) was developed. The formulation was designed to reduce interfacial tension, improve surface wettability, and promote the dispersion of ARPD components through surfactant action.

A PHF, consisting of a mixture of low-molecular-weight aliphatic hydrocarbons, was employed as the primary solvent. The selection of this solvent was dictated by its high dissolving capacity toward paraffinic hydrocarbons and its relatively low toxicity compared with aromatic solvents.

An ethoxylated nonionic surfactant (OP-10) and an anionic alkylbenzene sulfonate surfactant (sulfonol) were used as surface-active agents. The use of a mixture of anionic and nonionic surfactants was motivated by the possibility of achieving a synergistic effect, resulting in reduced interfacial tension, enhanced dispersing ability, and disruption of the structural matrix of ARPD.

Characterization of the investigated ARPD samples

The physicochemical composition of ARPD samples collected from various locations within the investigated oil field was examined as part of the laboratory study. The analytical results obtained for the selected samples are presented in Tab. 1.

Table 1. Composition of ARPD samples collected from different locations of the investigated oil field

Sample	Content, wt%				P/(A+R)	Deposit type
	paraffins	asphaltenes	resins	mechanical impurities		
No.1	19.3	0.5	9.8	9.2	1.9	paraffinic
No.2	12.4	1.9	7.0	11.03	1.4	paraffinic
No.3	19.2	0.6	10.0	66.8	1.8	paraffinic
No.4	20.6	0.1	7.7	0.2	26	paraffinic
No.5	19.3	0.1	6.5	2.8	2.9	paraffinic

Sample	Content, wt%				P/(A+R)	Deposit type
	paraffins	asphaltenes	resins	mechanical impurities		
No.6	19.2	0.2	7.5	2.8	2,5	paraffinic
No.7	18.0	0.5	6.7	7.1	2,5	paraffinic
No.8	17.2	0.2	6.0	0.1	2,8	paraffinic
average	18.2	0.5	7.7	12.5	2,3	paraffinic

The laboratory analyses demonstrated that the investigated ARPD samples belonged to the paraffinic type, exhibiting a melting point of 74°C. Quantitative compositional analysis showed that the deposits contained 0.1–66.8 wt% mechanical impurities, 0.1–1.9 wt% asphaltenes, 12.4–20.6 wt% paraffins, and 6.0–10.0 wt% resins.

Experimental procedure for evaluating ARPD solvent performance

A series of laboratory experiments was conducted to evaluate the dissolving, removal, and detergency performance of the developed formulations in accordance with the methodology described in Measurement procedure No.05-2021, «Laboratory Investigation of Chemical Reagents Used in Oil Production and Treatment Processes», developed by the Branch of “KMG Engineering” LLP “KazNIPImunaigas” under No.KZ.06/01/00327-2021 dated October 8, 2021 [12].

To determine the dissolving capacity, model ARPD samples were prepared from solid deposits in the form of spherical pellets with diameters ranging from 10 to 15 mm. The prepared samples were weighed using an analytical balance both without and together with a metal mesh.

For evaluating ARPD removal efficiency, the mold surface was initially prepared by polishing with No.0 abrasive paper, degreasing with acetone, drying, and determining its initial mass. Subsequently, a weighed portion of ARPD was placed in a glass beaker and melted in a water bath at (80±0.5) °C. The molten material was homogenized by stirring and then poured into the prepared mold. Excess melt was removed using a metal plate to ensure complete filling of the mold cavity. The surface of the sample was required to be smooth, and the deposit had to completely occupy the mold volume. The prepared samples were maintained at room temperature for 24 hour until a constant mass was attained. Prior to testing, the masses of the mold coated with ARPD, the ARPD sample without the mesh, and the sample together with the mesh were determined.

To evaluate the dissolution and removal efficiencies, the prepared samples were immersed in 50–100 cm³ of the investigated formulation placed in a glass beaker.

The ARPD samples were exposed to the investigated formulations at temperatures of 25°C and 60°C. The temperature was maintained using a thermostatically controlled water bath.

The duration of the experiments was 3 hour for dissolution tests and 1 hour for removal tests. During the experiments, visual observations of deposit destruction and dissolution behavior were carried out.

Upon completion of the experiment, the samples were removed from the solution, dried at room temperature for 24 hour, and subsequently reweighed. At least two parallel measurements were performed for each formulation, and the arithmetic mean of the obtained values was reported as the final result.

The efficiency of dissolution or removal (E) was calculated according to:

$$E = \frac{m_1 - m_2}{m_1} \cdot 100, \text{wt\%} \tag{1}$$

where m_1 – the mass of the deposits used for the experiment, g; m_2 – the mass of the deposits after the experiment, g.

The average efficiency (E_{avg}) was determined as the arithmetic mean of two parallel measurements according to:

$$E_{avg} = \frac{E_1 - E_2}{2} \tag{2}$$

Results and Discussion

At present, the removal of ARPD from tubing strings recovered from wells at the Uzen oil field is carried out in dedicated cleaning units (washing baths), where a washing solution containing a surfactant additive (commercial grade «PAN») at concentrations ranging from 0.6 to 3.5 wt% is used at temperatures not exceeding 95°C.

To establish an appropriate temperature regime for the study, experimental investigations were conducted to determine the melting temperature of solid ARPD samples. The measurements were performed using an automatic MCR 702 rheometer (Anton Paar, Austria) (Fig. 1).

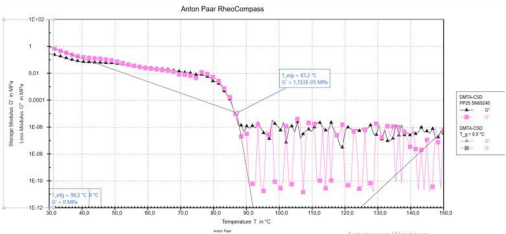


Figure 1. Determination of the melting temperature of solid ARPD deposits from the Uzen oil field

The experimental results demonstrated that the melting temperature of the solid ARPD deposits ranged from 82.9 to 87.2°C. Therefore, laboratory investigations aimed to evaluate the dissolution and removal efficiencies of the reference reagent, commercial-grade “PAN,” were conducted at temperatures not exceeding 90°C.

At the initial stage of the laboratory study, the optimal working concentration of the commercial surfactant «PAN» was determined based on the dispersion

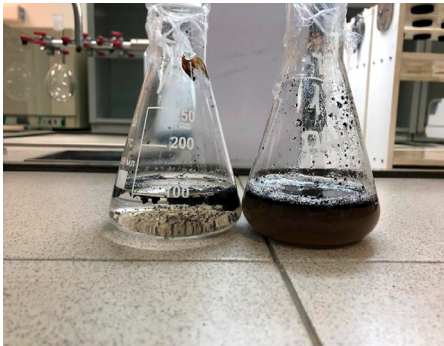
behavior of solid ARPD deposits. Representative photographs of the prepared washing solutions are shown in Fig. 2.

Subsequent laboratory investigations were performed to evaluate the efficiency of removing solid ARPD

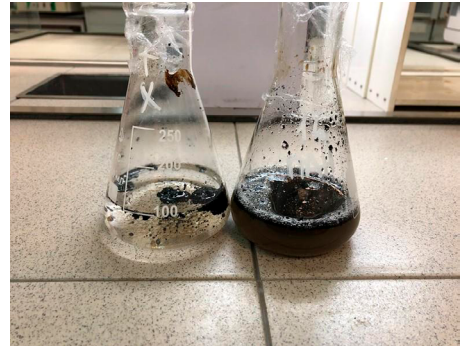
deposits using aqueous solutions of the commercial «PAN» surfactant at 60°C, 75°C, and 90°C. The obtained results are summarized in Tab. 2.

Table 2. Removal efficiency of aqueous PAN» surfactant solutions for ARPD deposits from metal surfaces after 1 hour of treatment

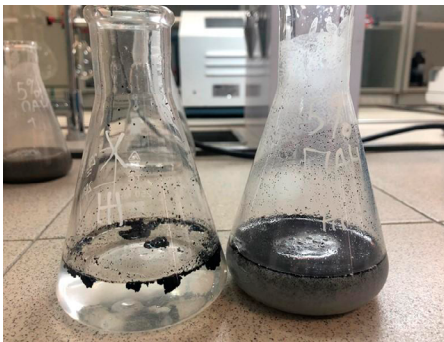
№	«PAN» concentration, wt%	T, °C	ARPD mass, g		Efficiency, %
			before	after	
1	Blank sample (without PAN)	60	0.5241	0.5229	0.23
		75	0.6076	0.6054	0.36
		90	0.6094	0.0181	97.03
2	0.25	60	0.5391	0.5297	1.74
		75	0.5080	0.4986	1.85
		90	0.4986	0.0011	99.78
3	1.0	60	0.7170	0.7031	1.94
		75	0.4935	0.4828	2.17
		90	0.4828	0.0008	99.83
4	10	60	0.3800	0.3777	0.61
		75	0.3553	0.3523	0.84
		90	0.4565	0.0046	98.99
5	30	60	0.3668	0.3608	1.64
		75	0.4646	0.4474	3.70
		90	0.5302	0.0015	99.72



a)



b)



c)



d)

Figure 2. Precision-Confidence Curve based on bounding rectangles

a) 1 wt% aqueous solution of the «PAN» surfactant; b) 1.5 wt% aqueous solution of the «PAN» surfactant; c) 5 wt% aqueous solution of the «PAN» surfactant; d) 10 wt% and 30 wt% aqueous solution of the «PAN» surfactant

The laboratory results demonstrated that the removal of solid ARPD deposits from the surface was achieved only at the investigated concentrations when the treatment temperature was increased to 90°C. However, removal efficiencies exceeding 90% were primarily attributed to the dissolution of ARPD at 90°C, which corresponds to the melting temperature range of the deposits themselves. Under all other test conditions, the efficiency of the aqueous PAN solutions did not exceed 4%. Furthermore, to achieve the objectives of the present study, a series of solvents commonly used for ARPD removal was investigated (Tab. 3). Both individual solvents and multicomponent formulations containing modifying additives were evaluated.

Table 3. Dissolution and removal efficiencies of individual solvents and solvent formulations

Solvent / Formulation	T, °C	Removal efficiency, %	Dissolution efficiency, %
PHF (pentane–hexane fraction)	30–60	<80	43–70
HS (hydrocarbon solvent)	30–60	<87	42–75
Gasoline	60	~81	~4
Acid formulations (HCl, H ₃ PO ₄ , CH ₃ COOH)	60	63–79	2,8–49
Blank systems (water, water + surfactant)	60–85	≤1	≤1,3

The hydrocarbon solvent and the PHF exhibited relatively high dissolution efficiency but low removal efficiency. Moreover, their application in pure form is associated with several drawbacks, including phase separation during temperature stabilization, partial precipitation of asphaltenes, and limited stability under field operating conditions. Thus, the conducted investigations demonstrated the limited effectiveness of both individual and modified solvents, which can be attributed to the complex multicomponent nature of ARPDs and the differences in the chemical characteristics of their constituents. These findings necessitated the development of a multicomponent formulation comprising a hydrocarbon solvent (PHF) in combination with various surfactants (OP-10 and sulfonol). The compositions of the investigated formulations intended for ARPD removal are presented in Tab. 4 [10].

Table 4. Composition of formulations developed for ARPD removal

Composition	Composition of the formulation, wt%					
	№1	№2	№3	№4	№5	№6
OP-10	–	1,75	3,5	10,5	21	33,25
Sulfonol	–	0,5	1,0	3,0	6,0	9,5
PHF	100,00	97,75	95,5	86,5	73,0	57,25

The experimental conditions and the results of evaluating the dissolution and removal efficiencies of the developed formulations are presented in Tab. 5 and Figs. 3–5.

The conducted investigations demonstrated that the temperature regime significantly affects the performance of formulations intended for ARPD removal. In all cases, increasing the temperature from 25°C to 60°C resulted in enhanced ARPD dissolution, removal, and detergency efficiencies. Analysis of the obtained results revealed that the individual hydrocarbon solvent, namely the PHF (Formulation No.1), provided ARPD dissolution and removal efficiencies of no more than 76%, indicating its limited capability to disrupt solid paraffinic and asphaltene structures (Figs. 3–5).

Table 5. Dissolution and removal efficiencies of the developed formulations toward ARPD

Formulation No.	T, °C	Degree of efficiency, %		Detergency, %
		ARPD dissolution (3 h)	ARPD removal (1 h)	
1	25	64.33	69.10	64.3
	60	71.65	75.77	71.7
2	25	96.40	95.92	96.4
	60	98.82	99.87	98.8
3	25	95.21	96.85	95.2
	60	100.00	100.00	100.0
4	25	95.77	96.09	95.8
	60	99.44	99.90	99.5
5	25	94.20	96.87	94.2
	60	99.26	100.00	99.3
6	25	97.05	97.69	97.1
	60	99.40	100.00	99.4

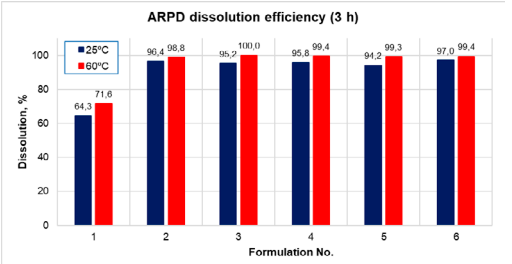


Figure 3. Evaluation of the dissolution efficiency of the formulations after 3 h at 25°C and 60°C

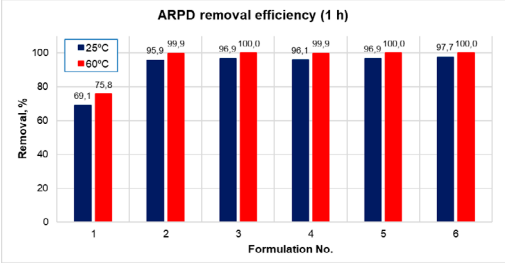


Figure 4. Evaluation of the ARPD removal efficiency of the formulations after 1 h of treatment at 25°C and 60°C

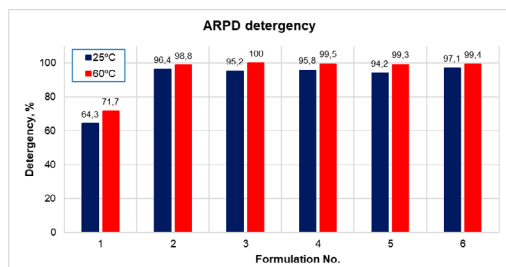


Figure 5. Evaluation of the detergency performance of the formulations at 25°C and 60°C

Thus, the use of a hydrocarbon solvent alone is insufficient to completely destroy the complex multicomponent matrix of ARPD. To improve performance, formulations with broad-spectrum action capable of affecting both paraffinic and resin-asphaltene components should be developed.

Formulations No.2–6 exhibited high efficiencies (> 99%) in both the dissolution and removal of solid ARPD deposits, which was further confirmed by their pronounced detergency performance. The high efficiency of these formulations can be attributed to the synergistic interaction between the solvent (PHF) and surfactants, as well as to the optimal balance between polar and nonpolar components. These formulations not only promote the dissolution of the solid ARPD phase but also ensure effective desorption of deposits from the surfaces of pipelines and oilfield equipment, thereby enhancing ARPD removal efficiency.

Visual observations of the interaction process revealed that the investigated formulations induced swelling and partial dissolution of ARPD samples within the first few hours of treatment, whereas complete dissolution was achieved within 3 hour. It was established that increasing the surfactant content in the formulations proportionally enhanced the dissolution, removal, and detergency efficiencies of ARPD, reaching values of up to 100%, in contrast to the control hydrocarbon solvent (Formulation No.1).

It should be noted that the investigated formulations maintained high efficiency even at 25°C, which represents an important practical advantage for their application under low-temperature

conditions and in situations where preliminary heating of the system is not feasible.

The test results demonstrated that the developed formulations provided ARPD removal efficiencies of up to 98% at 25°C and 100% at 60°C after 1 hour of treatment. Under otherwise identical conditions, the dissolution efficiency of solid ARPD deposits reached 95–97% at 25°C and complete dissolution was achieved at 60°C within 3 hours.

The authors suggest that the developed formulations have significant potential for reducing operating costs through lower energy consumption, shorter treatment times, and the use of readily available components.

Therefore, the developed formulations can effectively dissolve and remove solid organic deposits from the surfaces of oilfield equipment over a wide temperature range within a relatively short period of time. Their application is expected to reduce oil production operating costs and mitigate production-related problems associated with the high content of high-molecular-weight components in crude oil.

Conclusions

The results of the present study demonstrated that the developed multicomponent formulations based on a pentane–hexane fraction and a mixture of anionic and nonionic surfactants exhibit high efficiency in the removal of asphaltene–resin–paraffin deposits (ARPD).

It was shown that the use of individual solvents does not ensure complete destruction of the complex multicomponent structure of ARPD, whereas the application of combined formulations enables removal efficiencies of up to 100% owing to the synergistic interaction among the formulation components.

The developed formulations were found to retain high performance even at 25°C, which represents a significant advantage over conventional treatment methods that require heating to temperatures approaching or even exceeding the melting point of paraffins.

The practical significance of this work lies in the potential to reduce operating costs associated with ARPD mitigation, extend the maintenance interval of oilfield equipment, and improve the reliability and stability of oil production and transportation processes.

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Zhanetta A. Tleugaliyeva – conducting experimental works; Yerbolat O. Ayapbergenov – interpretation of data, writing and editing of the manuscript; Yevgeniy K. Ogay – checking the results, editing the manuscript; Maira B. Turkmenbayeva – interpretation of data, writing and editing of the manuscript; Anar Sh. Akkenzheyeva – concept of work, interpretation of data; Ainur K. Shakirova – analysis and interpretation of obtained data.

дение исследования и подготовку статьи, прочли и одобрили финальную версию перед публикацией). Наибольший вклад распределён следующим образом: Тлеугалиева Ж.А. – проведение экспериментальных работ; Аяпбергенев Е.О. – интерпретация данных, написание и редактирование рукописи; Огай Е.К. – проверка результатов, редактирование рукописи; Туркменбаева М.Б. – интерпретация данных, написание и редактирование рукописи; Аккензеева А.Ш. – разработка концепции работы, интерпретация данных; Шакирова А.К. – анализ и интерпретация полученных данных.

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