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DEVELOPMENT OF A DEEP EUTECTIC SOLVENT-BASED GREEN INHIBITOR FOR ASPHALTENE AGGREGATION CONTROL DURING CRUDE OIL TRANSPORTATION

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This article emphasizes that the transportation of heavy crude oil is accompanied by asphaltene deposition in pipelines, which leads to increased hydraulic flow resistance and, consequently, higher energy consumption for pumping. This, in turn, contributes to an increase in the carbon footprint of the oil production sector. The results of this study, aimed at developing energy- and resource-efficient technologies for the synthesis of a green inhibitor based on deep eutectic solvents (DESs), demonstrate the feasibility of preventing asphaltene precipitation during crude oil transportation. Within the framework of the study, asphaltenes were isolated from crude oils of the Zhanaozen and Karazhanbas fields of the Republic of Kazakhstan, model oil systems were prepared, and an environmentally friendly inhibitor based on betaine and ethylene glycol was synthesized. The model oil and the deep eutectic solvent were mixed at a 1:1 volume ratio, after which 0.15 mL, 0.25 mL, 0.45 mL, 0.65 mL, and 0.85 mL of n-heptane were sequentially titrated into the resulting mixture. Each sample was analyzed using ultraviolet (UV) spectroscopy. In samples containing the deep eutectic solvent as an inhibitor, a significant reduction in the degree of asphaltene aggregation was observed, as the inhibitor effectively slowed the agglomeration process. Accordingly, a decrease in UV absorbance intensity was recorded, indicating improved dispersion stability of the system. This effect was particularly pronounced for crude oil from the Zhanaozen field: upon the addition of 0.45 mL, 0.65 mL, and 0.85 mL of n-heptane, the absorbance values approached zero, indicating a near-complete absence of asphaltene aggregation that would otherwise hinder light transmission. The synthesis of an inhibitor based on betaine and ethylene glycol, biodegradable and low-toxicity compounds, represents a practical implementation of the «green inhibitor» concept, as confirmed by UV spectroscopic analysis and laboratory testing.

Keywords: green chemistry, deep eutectic solvents, asphaltene inhibitor, betaine, ethylene glycol, model oil, sustainable development, environmentally friendly technologies.

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Introduction

Oil remains the primary source for motor fuel production, and its extraction and transportation are largely carried out through pipeline systems. However, crude oil contains significant amounts of heavy resinous-asphaltene components that tend to aggregate

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and deposit on pipeline walls and processing equipment. Among these fractions, asphaltenes represent one of the most problematic constituents due to their high aromaticity, polarity, and tendency to precipitate under changes in pressure, temperature, or composition [1,2]. Asphaltene deposition leads to increased hydraulic resistance, reduced flow efficiency, equipment fouling, and substantial economic losses.

To mitigate these challenges, chemical inhibitors are commonly applied to stabilize asphaltenes in the oil phase. Conventional organic solvents such as toluene and xylene are effective, but suffer from major drawbacks, including high toxicity, flammability, and environmental impact. Consequently, modern research has increasingly focused on developing environmentally benign and cost-effective alternatives. In this context, deep eutectic solvents (DESs) have emerged as promising green inhibitors due to their low volatility, tunable physicochemical properties, biodegradability, and relatively simple synthesis routes [3–7].

DESs are typically formed by combining a hydrogen bond acceptor (HBA) with a hydrogen bond donor (HBD), resulting in a eutectic mixture with a melting point significantly lower than that of the individual components. Recent studies have demonstrated that DESs can effectively interact with asphaltenes through hydrogen bonding, electrostatic interactions, and π-π stacking, thereby suppressing aggregation and delaying precipitation [8,9]. These molecular interactions enable DESs to stabilize asphaltenes at the colloidal level, reducing their tendency to form large aggregates.

Several investigations have explored the application of DESs in petroleum systems. Hebbar et al. [9] demonstrated that glycerol- and choline-chloride-based DESs significantly reduced asphaltene aggregation in both toluene and n-heptane systems, which was confirmed by experimental and molecular simulation approaches. Kumar et al. [2] further advanced this concept by combining COSMO-RS modeling with experimental validation, identifying thymol-based DESs as highly effective inhibitors of asphaltene aggregation. These findings highlight the potential of DESs for rational design of next-generation asphaltene inhibitors.

From a technological and economic perspective, the development of efficient inhibitors is essential for improving oil production and transportation reliability. High-molecular-weight components such as asphaltenes, resins, and paraffins contribute to deposition in pipelines, wells, and storage tanks, leading to operational instability and increased maintenance costs. Effective inhibitors stabilize these components at the molecular level, enhance flow properties, prevent blockages, and extend equipment lifetime.

Overall, literature analysis confirms that deep eutectic solvents represent a promising, environmentally friendly, and versatile class of inhibitors for controlling asphaltene precipitation and deposition. Nevertheless, further systematic studies are required to optimize DES formulations, assess long-term stability, and evaluate large-scale industrial applicability under realistic oilfield conditions.

Experimental

For this study, two crude oil samples were collected from the Zhanaozen and Karazhanbas fields, located in the western part of the Republic of Kazakhstan:

– Zhanaozen – an oil and gas field on the Mangyshlak Peninsula (Mangystau region), belonging to the South Mangyshlak oil and gas-bearing area;

– Karazhanbas – an oilfield in the Tupkaragan district of the Mangystau region. Crude oil from this field was previously studied in ref. [10].

Three DES formulations were tested as solvents for asphaltenes:

– DES1 – citric acid:glycerol (1:4),
– DES2 – citric acid:ethylene glycol (1:4),
– DES3 – choline chloride:glycerol (1:2).

The selection of these oilfields was motivated by the high resin–asphaltene content of their crude oils, which increases the likelihood of isolating pure asphaltenes for further study.

Isolation of asphaltenes from crude oil

Asphaltenes were extracted from crude oil according to the state standard GOST 11851-2018 "Petroleum – Method for determining asphaltene content". A measured volume of crude oil (5 mL from each sample) was placed into a flask, and n-heptane was added. The mixture was kept in a dark place for 24 hours.

After 24 hours, a precipitate of asphaltenes formed at the bottom of the vessel, while the supernatant consisted of the lighter oil fraction. The precipitate was separated by filtration using filter paper placed in a funnel over a flask. The oil–n-heptane mixture was carefully poured onto the filter.

The filtrate contained the lighter oil fraction with a reduced asphaltene content, while the resin–asphaltene precipitate remained on the filter paper. Filtration was slow due to clogging of the filter pores by resinous components, which reduced the solution flow rate.

The next stage of asphaltene purification was performed by extraction using a Soxhlet apparatus. The filter paper containing the asphaltene residue was placed into the Soxhlet extractor, and toluene was used as the solvent. Since the boiling point of toluene is 116^0C, the heating plate was set to 120^0C. Within 3–5 minutes, toluene reached its boiling point.

During boiling, toluene rises in the form of vapor and, upon reaching the condenser, recondenses into liquid. Based on this condensation process, the condensed toluene accumulates in the chamber containing the filter paper. In its liquid state, toluene interacts with the asphaltenes deposited on the filter paper, forming a solution. Once the Soxhlet chamber containing the filter paper fills with toluene, the solution is siphoned back into the boiling flask through the upper tube.

The extraction process was repeated 4–5 times to remove asphaltenes from the filter paper completely. As a result, purified asphaltenes were collected at the bottom of the flask.

Preparation of model oil

For the subsequent investigation of the effect of the green inhibitor on asphaltenes, model oil was prepared from extracted asphaltenes. The isolated asphaltenes were dissolved in toluene (purity ≥99.8%) at a concentration of 0.5 wt.%. The mixture was stirred continuously for two hours and left to stand for 24 hours to ensure complete dissolution. In some experiments, asphaltenes were added to toluene at 0.1% (w/v), the samples were shaken on a platform shaker at 200 rpm for 1 hour, and then stabilized at room temperature for 24 hours [11].

This study used 150 mL of a solution prepared by mixing asphaltenes extracted from Zhanaozen crude oil with toluene. A solution containing asphaltenes from Karazhanbas crude oil in 100 mL of toluene was also prepared.

Both solutions were stirred on a magnetic stirrer for 1 hour at 80^0C and 200 rpm. A magnetic bar was placed inside the liquid, while the magnetic field source was positioned beneath the instrument base. The beakers were covered with foil, since toluene is a volatile liquid and its evaporation could alter the solution concentration. After stirring, the solutions were kept in a dark place, protected from sunlight, for 24 hours [11,12].

Synthesis of deep eutectic green inhibitor

Deep eutectic solvents (DESs) are eutectic mixtures composed of hydrogen bond acceptors (HBAs) and hydrogen bond donors (HBDs) mixed at specific molar ratios. Their physicochemical properties are governed primarily by hydrogen bonding, van der Waals forces, and electrostatic interactions, resulting in liquids with low vapor pressure, high thermal stability, and tunable solvating ability.

Compared with conventional ionic liquids, deep eutectic solvents (DESs) can be synthesized from biodegradable, low-toxicity, and inexpensive components using simple preparation procedures, which makes them particularly attractive for petroleum-related applications.

In this study, three DES formulations were prepared using the conventional heating–stirring method widely reported in the literature. The synthesis parameters, including component type, molar ratio, temperature, stirring speed, and synthesis duration, are summarized in Table 1 to ensure clarity and reproducibility.

Briefly, the selected hydrogen bond acceptor (HBA) and hydrogen bond donor (HBD) components were weighed according to the desired molar ratio, mixed in a glass flask, and heated under constant stirring until a clear and homogeneous liquid was obtained. The absence of solid residues or turbidity was taken as evidence of complete DES formation. The synthesized DESs were then cooled to room temperature and stored in airtight containers prior to use.

The final deep eutectic solvent obtained is shown in Figures 1 and 2.

Table 1

Synthesis parameters of deep eutectic solvents

Designation of DES	HBA component	HBD component	Molar ratio (HBA:HBD)	Temperature (^{0}C)	Stirring speed (rpm)	Synthesis time (min)	Appearance
DES1	Citric acid	Glycerol	1:4	80	200	120	Clear, colorless liquid
DES2	Citric acid	Ethylene glycol	1:4	80	200	120	Clear, colorless liquid
DES3	Choline chloride	Glycerol	1:2	80	200	120	Clear, colorless liquid
DES4	Betaine	Ethylene glycol	1:4	40→80	200	120	Clear, homogeneous liquid

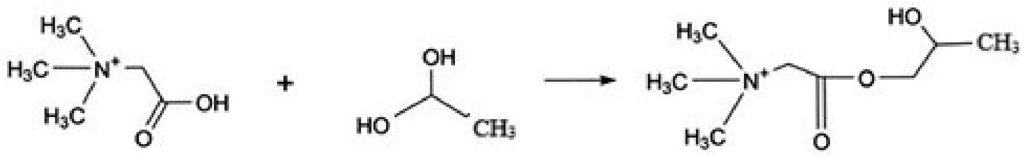

Fig. 1. Scheme of the DES prepared from betaine and ethylene glycol (author’s illustration)

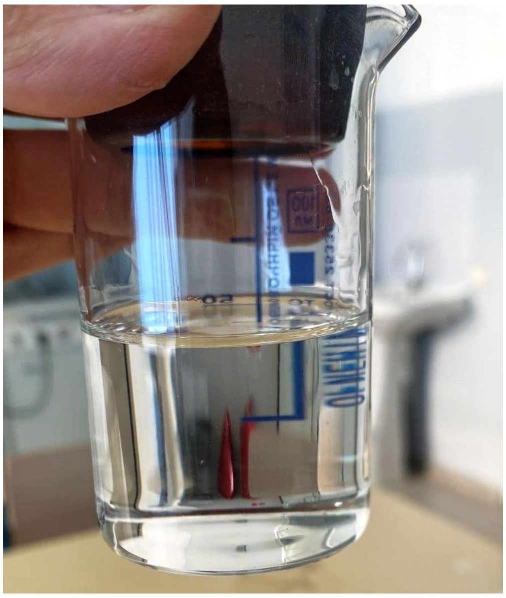

Fig. 2. Deep eutectic solvent (DES) prepared from betaine and ethylene glycol (author’s illustration)

Results and discussion

The next stage of the study involved the addition of the synthesized deep eutectic solvent to the model oil, followed by controlled titration with n-heptane. The model oil and DES were mixed at a 1:1 volume ratio, after which the mixture was sequentially titrated with 0.15, 0.25, 0.45, 0.65, and 0.85 mL of n-heptane. Each prepared sample was analyzed using ultraviolet (UV) spectroscopy.

Figures 3 and 4 present the UV absorption spectra of asphaltenes extracted from Zhanaozen and Karazhanbas crude oils, respectively. UV–Vis spectroscopy provides valuable insight into asphaltene behavior, as these compounds typically exhibit characteristic absorption in the 700–900 nm region, which is associated with aggregate formation.

The highest absorption intensity was consistently observed in samples without DES (0 mL), indicating extensive asphaltene aggregation. As aggregation increases, light transmission through the solution decreases, resulting in higher absorbance values. Upon DES addition, a noticeable reduction in absorption intensity was recorded, demonstrating the inhibitor’s ability to suppress aggregation.

This effect was particularly pronounced for Zhanaozen crude oil, where absorbance values approached zero at higher n-heptane additions (0.45–0.85 mL), indicating near-complete inhibition of asphaltene aggregation. In contrast, Karazhanbas crude oil exhibited consistently higher absorbance values, reflecting its greater resin–asphaltene content and stronger aggregation tendency, even in the presence of DES.

UV–Vis absorption spectra of asphaltenes extracted from Zhanaozen crude oil during titration with n-heptane at different volumes (0–0.85 mL), in the presence and absence of the deep eutectic solvent (DES). The peak observed near 750 nm corresponds to asphaltene aggregation. The progressive decrease in absorption intensity with increasing DES concentration indicates suppression of asphaltene agglomeration and improved dispersion stability.

UV–Vis absorption spectra of asphaltenes

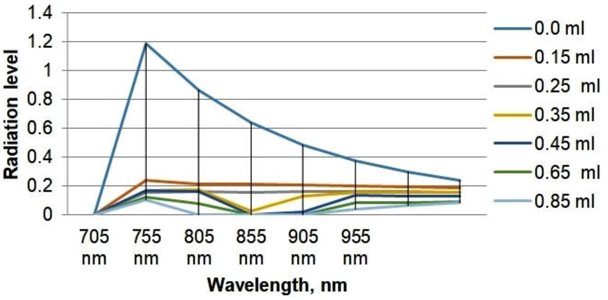

Fig. 3. UV absorption of asphaltenes in Zhanaozen crude oil

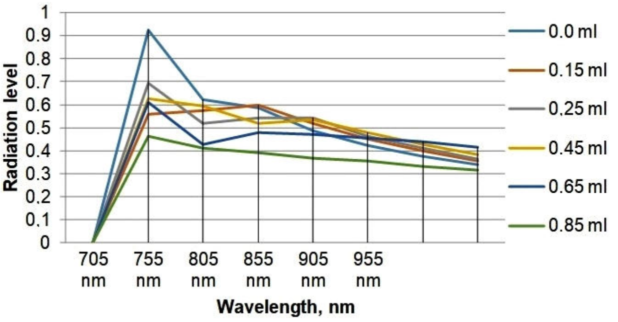

Fig. 4. UV absorption of asphaltenes in Karazhanbas crude oil

extracted from Karazhanbas crude oil during sequential n-heptane addition (0–0.85 mL), with and without the deep eutectic solvent. Higher residual absorbance compared to Zhanaozen crude oil reflects the greater resin–asphaltene content and stronger aggregation tendency of Karazhanbas crude oil, even in the presence of the inhibitor.

Conclusions

Based on the presented results, the application of green chemistry approaches shows strong potential for addressing the problem of asphaltene deposition during oil production and transportation.

The literature analysis and experimental findings confirm that deep eutectic solvents are environmentally safe, tunable systems capable of effectively interacting with asphaltenes. The synthesis of an inhibitor based on betaine and ethylene glycol, biodegradable, low-toxicity compounds, represents a practical implementation of the concept of green inhibitors.

The experimental results demonstrate that model oils prepared from asphaltenes extracted from Zhanaozen and Karazhanbas crude oils exhibit reduced aggregation and a lower tendency toward asphaltene precipitation after DES addition, indicating enhanced dispersion stability and improved reliability of crude oil transportation.

Importantly, although the experimental validation was carried out using crude oils from Kazakhstan oilfields, the inhibition mechanism is not region-specific. It is governed by universal physicochemical interactions between asphaltenes and DES components, suggesting that the proposed approach is applicable to a wide range of crude oils with high resin–asphaltene content. The simplicity of DES synthesis, the use of inexpensive and environmentally benign components, and compatibility with existing oilfield operations indicate strong potential for scaling up and industrial implementation of the proposed green inhibitor technology.

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Conflict of interest

The authors declare that they have no conflict of interest.

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РОЗРОБЛЕННЯ ЕКОЛОГІЧНО БЕЗПЕЧНОГО ІНГІБІТОРУ НА ОСНОВІ ГЛИБОКИХ ЕВТЕКТИЧНИХ РОЗЧИННИКІВ ДЛЯ КОНТРОЛЮ АГРЕГАЦІЇ АСФАЛЬТЕНІВ ПІД ЧАС ТРАНСПОРТУВАННЯ СИРОЇ НАФТИ

А.Ж. Керімкулова, С.Б. Риспаєва, Ш.С. Іслам, Х.С. Рафікова, М.А. Кожайсакова, Н.Н. Нургалієв, М.Е. Даріджан

У статті наголошено, що транспортування важкої сирої нафти супроводжується відкладенням асфальтенів у трубопроводах, що призводить до зростання гідравлічного опору потоку і, відповідно, до підвищення енергоспоживання на перекачування. Це, своєю чергою, сприяє збільшенню вуглецевого сліду нафтовидобувного сектору. Результати цього дослідження, спрямованого на розроблення енерго- та ресурсоефективних технологій синтезу екологічно безпечного інгібітору на основі глибоких евтектичних розчинників (DES), демонструють можливість запобігання осадженню асфальтенів під час транспортування сирої нафти. У межах дослідження асфальтени було виділено з нафт родовищ Жанаозен і Каражанбас Республіки Казахстан, підготовлено модельні нафтові системи та синтезовано екологічно безпечний інгібітор на основі бетаїну та етиленгліколю. Модельну нафту та глибокий евтектичний розчинник змішували в об’ємному співвідношенні 1:1, після чого в одержану суміш послідовно титрували 0,15 мл, 0,25 мл, 0,45 мл, 0,65 мл і 0,85 мл n-гептану. Кожен зразок аналізували методом ультрафіолетової (УФ) спектроскопії. У зразках, що містили глибокий евтектичний розчинник як інгібітор, спостерігалося істотне зменшення ступеня агрегації асфальтенів, оскільки інгібітор ефективно уповільнював процес агломерації. Відповідно фіксувалося зниження інтенсивності УФ-поглинання, що свідчить про підвищення дисперсійної стабільності системи. Особливо вираженим цей ефект був для сирої нафти родовища Жанаозен: за додавання 0,45 мл, 0,65 мл і 0,85 мл n-гептану значення оптичної густини наближалися до нуля, що вказує на майже повну відсутність агрегації асфальтенів, яка зазвичай перешкоджає проходженню світла. Синтез інгібітору на основі бетаїну та етиленгліколю, біорозкладних і малотоксичних сполук, є практичною реалізацією концепції «зеленого інгібітору», що підтверджено УФ-спектроскопічним аналізом і лабораторними випробуваннями.

Ключові слова: зелена хімія; глибокі евтектичні розчинники; інгібітор асфальтенів; бетаїн; етиленгліколь; модельна нафта; сталий розвиток; екологічно безпечні технології.

DEVELOPMENT OF A DEEP EUTECTIC SOLVENT-BASED GREEN INHIBITOR FOR ASPHALTENE AGGREGATION CONTROL DURING CRUDE OIL TRANSPORTATION

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