Thermal treatment of model water-in-oil emulsions by coal fly ash particles

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Al-Farabi Kazakh National University, Almaty, Kazakhstan *E-mail: saidullakentau@mail.ru One of the primary challenges in the oil industry is the formation of stable crude oil emulsions, which cause equipment and pipeline corrosion due to the inorganic salts dissolved in the aqueous phase. This study investigated the effect of coal fly ash on model crude oil emulsions. The model emulsions were prepared using crude oil samples from the Kyzylorda and Atyrau regions. Thermal demulsification at 60°C resulted in a low dewatering rate, with only 16% of water separated from a 50% water-in-oil emulsion. Higher efficiency in oil emulsion separation was observed using the microwave treatment method. The highest dewatering degree was achieved for the Atyrau crude oil emulsion, reaching 75% after 155 seconds for a 50% emulsion with a 1% ash concentration at a microwave power of 700 W, while the dewatering for the Kyzylorda oilfield emulsion was 50% under the same conditions. This difference in demulsification efficiency is attributed to the composition of the crude oil samples. It has been demonstrated that the microwave treatment method somples. It has been demonstrated that the microwave treatment method somples. It has been demonstrated that the microwave treatment method somples. It has been demonstrated that the microwave treatment method combined with the use of coal fly ash enables the separation of water-in-oil emulsions within a short period.

Keywords: water-in-oil emulsions; coal fly ash; thermal treatment; demulsification; microwave irradiation.

Мұнайдағы су модельді эмульсияларын көмір ұшпа күлінің бөлшектерімен термиялық өңдеу

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Әл-Фараби атындағы Қазақ ұлттық университеті, Алматы қ., Қазақстан *E-mail: saidullakentau@mail.ru Мұнай өнеркәсібіндегі негізгі мәселелердің бірі — тұрақты мұнай эмульсияларының түзілуі, олар су фазасында еріген бейорганикалық тұздар салдарынан жабдықтар мен құбырлардың коррозиясын тудырады. Бұл зерттеуде көмір ұшпа күлінің модельді мұнай эмульсияларына әсері қарастырылды. Модельді эмульсиялар Қызылорда және Атырау облыстарынан алынған шикі мұнай үлгілері негізінде дайындалды. 60°С температурадағы термиялық деэмульсиялау төмен сусыздану дәрежесін көрсетті — 50% су-мұнай эмульсиясынан тек 16% су бөлінді. Мұнай эмульсияларын бөлү тиімділігі микротолқынды өңдеу әдісінде жоғары болды. Ең жоғары сусыздану дәрежесі Атырау мұнайының эмульсиясында байқалды — 700 Вт микротолқынды қуатта 1% күл концентрациясы бар 50% эмульсия үшін 155 секүндтан кейін 75%-ға жетті, ал Қызылорда кен орнының эмульсиясы үшін сусыздану деңгейі 50% болды. Деэмульсиялау тиімділігіндегі бұл айырмашылық шикі мұнай үлгілерінің құрамына байланысты. Микротолқынды өңдеу әдісі мен көмір ұшпа қүлін қолдану арқылы су/мұнай эмульсияларын қысқа уақыт ішінде бөлуге болатыны дәлелденді.

Түйін сөздер: су-мұнай эмульсиялары; көмір ұшпа күлі; термиялық өңдеу; деэмульсиялау; микротолқынды сәулелену.

Термическая обработка модельных водонефтяных эмульсий частицами угольной золы уноса

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Казахский национальный университет имени аль-Фараби, г. Алматы, Казахстан *E-mail: saidullakentau@mail.ru Одной из основных проблем нефтяной промышленности является образование стойких нефтяных эмульсий, вызывающих коррозию оборудования и труб из-за растворенных в водной фазе неорганических солей. В данной работе исследовалось влияние угольной золы уноса на модельные нефтяные эмульсии. Модельные эмульсии были приготовлены на основе образцов сырой нефти из Кызылординской и Атырауской области. Термическая деэмульгирование при 60°С приводит к низкой степени обезвоживания, количество отделившейся воды составило 16% для 50% эмульсии типа вода-нефть. Более высокая эффективность разделения эмульсий нефти была выявлена для метода микроволновой обработки. Самая высокая степень обезвоживания достигнута для эмульсии нефти Атырау, она достигает 75% через 155 секунд для 50% эмульсии с концентрацией золы в 1% при мощности микроволновой печи в 700 Вт, тогда как для эмульсии Кызылординского месторождения обезвоживание составляет 50%. Данное различие в степени деэмульгирования зависит от состава образцов сырой нефти. Показано, что метод микроволновой обработки в комбинации с применением золы уноса позволяет разделять эмульсии типа вода/нефть за короткий промежуток времени.

Ключевые слова: водно-нефтяные эмульсии; угольная зола уноса; термическая обработка; деэмульгирование; микроволновое облучение.

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1. Introduction

The formation of crude oil emulsions is inevitable during oil extraction and transportation. Some physical properties of crude oil, such as density and viscosity, are usually aggravated during emulsification, which incurs additional expenses for the oil industry. In addition, crude oil emulsions significantly contribute to the corrosion of multiphase flow pipelines, primarily due to their complex interactions with water and corrosive agents, which can accelerate the deterioration of pipeline materials [1]. Eventually, emulsions can significantly impact the capacity of separators, pumps, and pipelines and present significant flow assurance issues [2]. The arguments above induce the oil industry to seek effective, fast, cheap and environmentally friendly methods of breaking the crude oil emulsions.

Demulsification is a process of breaking down the emulsion into two separate phases: water and oil. Chemical demulsifiers are applied to break the interfacial film of natural stabilizers surrounding the water droplets in water-in-oil emulsions. The high cost and possible environmental hazard of commercial synthetic demulsifiers demand the search for more costeffective and environmentally friendly alternatives.

Different demulsification methods are available today, including chemical treatment, heating, gravity separation, centrifugation, filtration, and membrane separation [3-5]. These techniques, in combination or separately, are widely used in the oil industry to destabilize water-in-oil emulsions.

Recent findings indicate that introducing coal fly ash (CFA) powder has potential demulsifying action and causes the separation of two phases [6-8]. CFA is a waste of coal-fired powerplants composed of minerals such as mullite and quartz, aluminium oxide and iron (III) oxide [9-10]. Using coal ash as a demulsifier is a relatively new approach that has attracted

attention due to its simplicity, ash recycling capability, high efficiency, and low operating cost. Adewunmi et al. [6] studied the efficiency of coal ash as a demulsifier for highly stable water/oil emulsions under different conditions. The results showed that adding coal ash in various amounts (1 to 7%) resulted in the separation of water from the oil phase, with the highest dewatering efficiency of 96.67% observed with the addition of 7% ash. Studies have shown that coal ash has more dewatering efficiency at elevated temperatures and provides a strong alternative to commercial demulsifiers. The demulsifying capacity of coal ash is associated with the presence of a large amount of Al₂O₂ and SiO₂ in its particles. The authors [11] proposed a demulsifying mechanism in which ash particles interact with asphaltenes and destroy the protective film at the oil/water interface, which leads to the separation of water and oil phases.

Microwave irradiation technology has emerged as an alternative method for emulsion separation [12-13]. However, little has been done to apply microwave effects on separating crude oil from Kazakhstan oil fields. Usually, to separate water from freshly recovered crude oil, it is heated at 50-60°C in the presence of the demulsifying agents [14]. Microwave heating for dewatering crude oil is considered a very effective technique. This results in the separation of water from crude oil in a short period. Many authors have considered microwave irradiation for demulsification purposes because of its advantages, such as high efficiency, low energy consumption, and being environment-friendly [15].

Therefore, the aim of this research was devoted to developing a new cost-effective solid demulsifier based on the powder of coal fly ash for water-in-oil emulsions and researching the effect of CFA particles and microwave treatment on crude oil emulsion of domestic oilfields. The crude oil samples were obtained from oilfields in the Atyrau and Kyzylorda regions. The

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powder of CFA was collected from Almaty thermal powerplant N° 2; CFA powder has been used to study its potential as a solid demulsifier in separating water from a crude oil emulsion to replace chemical demulsifiers [8].

2. Experiment

2.1 The research objects

1) Samples of crude oil taken from the oilfields of the Kyzylorda and Atyrau regions were used. The samples of crude oil do not contain the water or contain traces of water (Dean-Stark method GOST 2477-65). The main characteristics of oil samples are shown in Table 1.

2) Preparation of crude oil emulsion. 50 ml crude oil emulsions of 50% were prepared by mixing crude oil with distilled water for 10 minutes using the homogenizer IKA T 10 basic ULTRA TURAX at room temperature (25°C) until a homogeneous mixture formed. The emulsions were left at room temperature for 1 week to get stable emulsions.

3) CFA was collected from the Almaty thermal power plant N° 2 as industrial waste and sieved through a 125 – 63 μ m sieve. The CFA samples were dried before demulsification experiments. The electrokinetic potential of CFA was measured using Anton Paar zeta potential analyzer, and it equals to ζ = -25 mV.

2.2 Methods of research

1) Oil emulsion stability test. The stability of a model emulsion was studied using a conventional separation test, in which the amount of water separated is observed with time at $50-60^{\circ}$ C. Crude oil emulsion was transferred into 50 ml graduated glass test tubes and placed into a thermostat. The aqueous phase separation was monitored at regular time intervals (10 min). The water separation percentage (W, %) was calculated as the ratio of the volume of separated water to the initial volume of water in the emulsion.

2) Microwave demulsification tests. 40 ml of emulsions were prepared in the measuring cylinder in the same way as the stability tests. Then 0.4 g (1%) of CFA was added to the prepared emulsions, and the mixture was homogenized for 5 minutes. The domestic microwave oven was used as a microwave source to test microwave irradiation. The emulsion samples were placed inside the microwave oven at 400 and 700 W power, and the water separation was studied. The demulsification efficiency (DE) of oil emulsions in per cent (W, %) was calculated as the

relation of the volume of separated water to the initial volume of water in the emulsion.

All measurements were repeated three times for each sample.

3) IR-spectrometry. Fourier-transform infrared spectroscopy analysis was used. The IR analysis of crude oil samples was conducted by the FTIR spectrometer Spectrum-65. The analysis was conducted at 4000-500 cm⁻¹ wavenumber.

3. Results and Discussion

It is important to enhance the recovery, transportation, processing, stabilization, and dewatering of crude oil systems due to the development of the oil sector. The separation of water and salts dissolved from crude oil is required to increase economic and technological indicators since oil's chloride and sulfide salt content causes a decline in the quality of petroleum products using special techniques to break down water-in-oil emulsions [11]. As mentioned above, the research aims to develop new demulsifiers for the separation of water-in-oil emulsions and to research the effect of CFA and microwave treatment on oil emulsion from crude oil of domestic oilfields. The demulsifying action of CFA has been studied to reach the aim of the study.

The crude oil model emulsions were prepared by mixing with distilled water. The concentration of the model emulsion was 50 %. The model emulsions remained stable without any phase separation at room temperature for 1 week. The thermostatically controlled heating at 50°C did not show the breaking of model emulsions. At 60°C, water separation from the emulsions was observed. However, the amount of water released was not significant, with the highest DE being 16% for the 50% emulsion.

Emulsion stabilization due to the presence of natural stabilizers can explain the low DE (16 %). For crude oil emulsion of the Kyzylorda region, the effect of the thermal treatment without demulsifying agents was studied. It was found that the thermal treatment was not enough for the dewatering of the w/o emulsion because the DE did not exceed 10% at 60° C.

Naphthenic acids, their salts, resins and asphaltenes, highly dispersed particles of paraffin and ceresin, and particles of mechanical impurities are usually natural crude oil emulsifiers [16]. Some of them were identified in crude oil samples (Table 1).

Table 1 – Physico-chemical properties of crude oil samples

Crude oil sample	Density, kg/m³	Dynamic viscosity, mPas, 20 °C	Amount of as- phaltenes, % (±0.01) wt. (GOST 11858-66)	Amount of resins, % (±0.01) wt. (GOST 11858-66)	Amount of carbenes, carben- oids and mechanical impurities, % (±0.01) wt. (GOST 11858-66)
oil field of Atyrau region	860	58.8	3.06	0.280	1.12
oil field of Kyzylorda region	850	11.6	2.47	2.94	1.55



Figure 1 – IR-spectra of oil samples and asphaltenes

To justify the presence of asphaltenes in the crude oil samples, the IR spectra of two samples and asphaltenes separated from the second crude oil sample were analyzed (Figure 1). All samples have medium bands corresponding to methyl groups ($-CH_3$) and methylene groups ($-CH_2$), stretching and bending vibrations at 2923.09 cm⁻¹ (Kyzylorda) and 2930 cm⁻¹ (Atyrau), 1461.17 cm⁻¹ (Atyrau) and 1463.51 (Kyzylorda) [19]. The similarity between the peaks of both oil samples with asphaltenes suggests that asphaltene stabilizers are present in both oil samples.

A microwave treatment was applied to increase the DE. Compared with conventional heating, the dewatering efficiency increases under microwave irradiation. Microwave irradiation has many advantages, such as high efficiency, energy savings, and environmental friendliness. A lot of work has been developed recently regarding the application of microwave irradiation for breaking emulsions [17]. In this work, microwave irradiation and the addition of CFA were tested simultaneously to demulsify the water-in-oil emulsions.

The physicochemical characteristics of coal fly ash particles were studied earlier [18]. CFA is mainly represented by crystals of mullite and quartz, as well as aluminium and iron (III) oxides.

As mentioned before, according to Adewunmi et al. [6, 8], the CFA particles come into contact with w/o stabilizers, damaging the protective layer of the water phase. The destabilized layer generates a coalescence of water droplets. As a result of increased coalescence and aggregation, water droplets settle at the bottom of the vessel. In [18], the authors also stated that the demulsifying capability of CFA is connected to the destabilization capability of alumina (Al_2O_3) and silica (SiO₂) oxides. The effectiveness of fly ash (CFA) in breaking down emulsions is attributed to its interaction with natural stabilizers in crude oil, such as asphaltenes and other organic compounds. The CFA particles disrupt the stabilizing interfacial film around water droplets, leading to coalescence and subsequent phase separation. The demulsifying capability of CFA is related to its adsorption on stabilizers and the weakening of the water/oil interface, which enhances the coalescence of water droplets and facilitates demulsification under microwave irradiation.

The treatment of the Atyrau region w/o emulsion at 400 W showed phase separation. The sample without CFA displayed the highest DE of 15% after 70 seconds, while the highest percentage of separated water in the sample with CFA was 40% after 120 seconds (Figure 3).



Figure 2 – Demulsification of 50% emulsion (Atyrau region oilfield) at 400W

The choice of microwave power is related to efficiency and energy input. At power lower than 400W, the demulsification process is inefficient and very slow. In contrast, at a power higher than 700W, water droplets in the emulsions boil off rapidly with crude oil and cause experimental errors. The experiments at maximal microwave power (700 W) demonstrated the highest DE of 75% after 155 seconds for the sample with CFA addition and 45% after 120 seconds for the emulsion without CFA (Figure 3).



Figure 3 – Demulsification of 50% emulsion (Atyrau region oilfield) at 700W

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The experiments for the second sample of the same concentration from the Kyzylorda region oilfield at microwave power 400W demonstrated 37.5% of DE over 265 seconds and 27.5% of DE over 280 seconds for the sample with and without CFA, respectively (Figure 4).

As can be seen from Figures 2-5, increasing microwave power from 400 to 700 W increases the DE. For a water-in-oil



Figure 4 – Demulsification of 50% emulsion (Kyzylorda region oilfield) at 400W



Figure 5 – Demulsification of 50% emulsion (Kyzylorda region oilfield) at 700W



Figure 6 – Demulsification of 40% emulsion (Kyzylorda region oilfield) at 700W

emulsion with a 40% concentration at 700 W, the DE does not change much compared with a 50% concentration (Figure 6). The difference in demulsification efficiency between Kyzylorda oil and Atyrau oil is attributed to the distinct physicochemical properties of the crude oil samples from these regions. The Kyzylorda oil has a higher total content of asphaltenes and resins (Table 1), which act as natural stabilizers for water-in-oil emulsions, making them more resistant to demulsification. In contrast, the Atyrau oil has a lower stabilizer content, resulting in less stable emulsions that are more easily disrupted.

At 400 W DE for 40% emulsion was insignificant (Figure 7).



Figure 7 – Demulsification of 40% emulsion (Kyzylorda region oilfield) at 400W

The influence of the amount of CFA added was studied for a 40 % emulsion at 400 W (Figure 8). Increasing the CFA amount from 1% to 2% leads to a twofold increase in demulsification efficiency, and the DE is close to 40% at 700 W.



Figure 8 – Demulsification of 40% oil emulsion (Kyzylorda region oilfield) under the action of different concentrations of CFA at 400W

While promising, this study has limitations. Laboratoryscale experiments necessitate further research on large-scale feasibility, including equipment, costs, and process control. Microwave irradiation accelerates demulsification but may cause crude oil vaporization, requiring efficiency comparisons with conventional heating. Variations in crude oil composition, particularly high asphaltene and resin content in Kyzylorda crude, reduced efficiency. CFA performance also varies with coal source and combustion conditions, highlighting the need for standardization or optimization. Addressing these challenges is crucial for adopting microwave-assisted CFA demulsification in the oil industry.

4. Conclusion

The model emulsions were prepared using crude oil from the Kyzylorda and Atyrau regions, with physicochemical properties analyzed according to State Standards. Asphaltenes and resins, acting as natural stabilizers, were confirmed via Standards and IR spectra.

Thermal demulsification at 60 °C resulted in low efficiency, with only 16% water separation for a 50% water-in-oil emulsion. However, increasing microwave power from 400W to 700W significantly improved demulsification efficiency (DE). The highest DE of 75% was achieved for Atyrau crude oil emulsion (50% water-in-oil, 1% CFA, 700W, 155 seconds), while Kyzylorda crude oil reached 50% DE under the same conditions. CFA particles contributed to demulsification by adsorbing stabilizers and promoting water droplet coalescence.

These findings demonstrate that microwave-assisted demulsification with CFA is an efficient and cost-effective alternative to chemical demulsifiers, offering an environmentally friendly solution for the oil industry. However, further research is needed to assess large-scale feasibility, optimize CFA composition, and compare energy efficiency with conventional heating.

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