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Relaxation Process in Crude Oil after Ultrasonic Treatment

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P A P E R I N F O

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A B S T R A C T

This paper presents an investigation on changes in viscosity and group composition of heavy oil samples after ultrasonic upgrading process. Heavy oil samples correspond to different oilfields (located in Russia) were processed under various ultrasonic mode, the cavitation consequence of which was controlled by acoustic method. The viscosity of samples was measured just after ultrasonic treatment, 10 minutes, and 1-34 days. The saturate, aromatic, resin and asphaltene (SARA) analysis was carried out after ultrasonic treatment with 34 days of relaxation and the achieved results were compared with the SARA fractions of original crude oil. The results of viscosity measurement showed viscosity reduction after the ultrasonic treatment. However, the viscosity was regressed after 1-4 days of relaxation with further reduction in 7 days. The degree of viscosity increase after 34 days was only 10% in contrast to viscosity of original crude oil. The power and the sonication time did not influence the relaxation process. In conclusion, attention was drawn to the results of SARA analysis, the content of saturates decreased and the relative content of heavy fragments such as resins and asphaltenes was increased, which determines the degree of viscosity increase after relaxation period.

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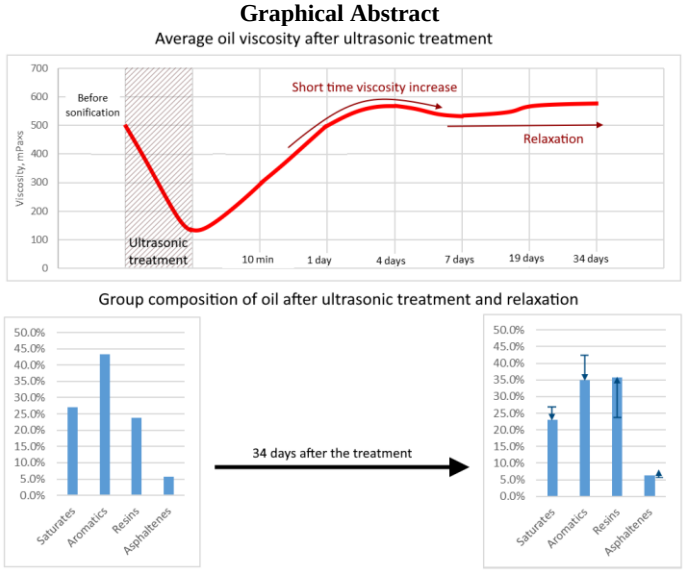
Graphical Abstract



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1. INTRODUCTION

Due to growing interests in the heavy oil reservoirs (1-3), enhanced oil recovery methods became more popular (4-6). Ultrasound-assisted heavy oil upgrading is one of the methods of enhanced oil recovery. It allows to reduce viscosity of oil and paraffins and asphaltenes sedimentation (7-9). It is widely used during oil recovery and processing (10-12).

Asphaltenes are the heaviest molecules of oil and they mainly determine the viscosity of crude oil. However, the asphaltenes have a tendency to precipitate and aggregate. The size of the last depends mainly on its concentration. It is well known, that asphaltene molecules are present in toluene at concentrations less than 1 mg/l as monomers and above 5 mg/l as dimers and trimmers, etc. With increasing concentration, asphaltenes are present as supermolecules and solid-like aggregates (13-15).

The presence of large structures leads to non-Newtonian behavior of fluids. In such fluids, viscosity depends on the aggregate size, shape, interactions between structures, flexibility of aggregate elements, etc. (16). In heavy crude oils, asphaltenes structure, size and interaction can change due to external conditions, such as temperature and pressure (17-19). Thus, the ultrasound energy can be employed to destruct the aggregation of the asphaltenes, which significantly reduces the viscosity of the fluid (20-22). However in some cases, it has been reported that viscosity of crude oil can be increased up to the untreated levels or even more after sonification (23-25). Others argue that this phenomenon is due to the formation of new molecular structure in normal conditions (26, 27).

Relaxation phenomenon in chemistry is related to the delay between the external effect and system response. After an abrupt change of pressure or temperature it takes time for the molecular or atomic structure to re-equilibrate under new conditions. This effect can be caused by the redistribution of energy among electronic, nuclear, rotational and vibrational molecular energy states or by a shift in the ration of the number of product molecules to reactant molecules in chemical reactions. The relaxation time depends on the atomic and molecular structure, on the rate and mechanisms of chemical reactions (28).

Relaxation phenomenon in crude oil and similar organic fluids have been studied in several where magnetic field influence on the oil viscosity was investigated. It was concluded, that viscosity changes due to ferromagnetic aggregates destruction in the alternating magnetic field. Volkova et al. (29) investigated the destruction of polymers after sonification. It was observed that destructed polymer chains recombined after sonification. Cavitiation process highly increases heat production during sonification (30, 31).

The aim of this study is to reveal the influence of ultrasonic relaxation on viscosity and group composition of heavy oil. Moreover, the light fragments were evaluated in detail by GC-MS analytical tool. The cavitation intensity was controlled using an acoustic method.

2. MATERIALS AND METHODS

Two crude oil samples were used in this study, one of which was isolated from Ashal'chi oil reservior (Republic of Tatarstan, Russia) and the second one from North Komsomol oil reservior (Yamalo-Nenetsk Autonomous Region, Russia).

LLC «VOLNA» (Moscow, Russia) developed the experemental set-up for the sonification of samples. This set-up allows to control the acoustic multifrequency fields at the given amplitude (32, 33). The schematic drawing of the set up is illustrated in Figure 1. The reactor (3) is connected to the thermostat (1), which allows it to maintain constant temperature. 100 mL of crude oil sample was maintained at temperature of 40°C for 1 hour before the sonification. The temperature of the samples during ultrasonic treament is controlled by thermocouples (6). Temperature controlled reactor has a pizecoceramic transucer mounted at the bottom (4) to produce the ultrasonic field.

The generator (9) was used to apply the high frequency signal to the tranducer, it allows to regulate the aplitude of the waves by adjusting the power of the electric signal. Transformer (5) and oscilloscope (8) was used to detect and monitor the applied signal. The frequency of acoustic signal in the sample during sonification was also monitored using the hydrophone (7). Monitor for obseration of data acquisition system (10) was used to record the signal processing. The spectrum of applied signal is shown in Figure 2. The main harmonics of the applied signal was 20 kHz.

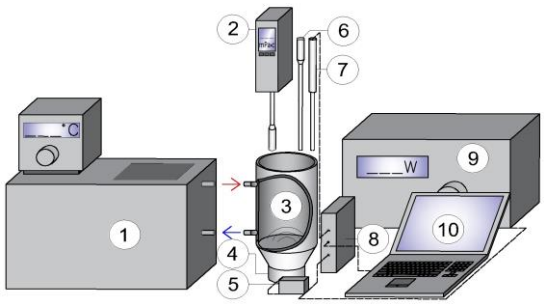


Figure 1. Experimental set-up for ultrasonic processing of oil 1. thermostat 2. viscosimeter 3. temperature-controlled reactor 4. piezoceramic ultrasonic transducer 5. current transformer 6. thermometer 7. hydrophone 8. digital oscilloscope 9. electric signal generator UZG-22 10. workstation

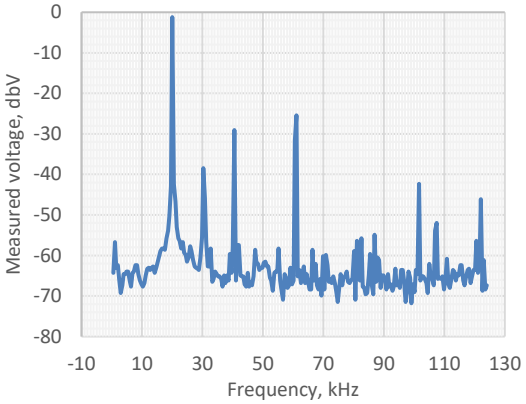


Figure 2. Applied signal spectrum

To determine viscosity changes during sonification PCE-RVI 2 viscosimeter was used. It was immersed into the reactor filled with crude oil immediately after sonification for the viscosity control during thermal conditioning. The viscosity of crude oil during viscosity relaxation was determined using a Fungilab Alpha L viscosimeter at 20 °C, 40 °C and 60 °C.

SARA analysis method was used to investigate the composition of crude oil after ultrasound treatment and relaxation. 40 ml of hexane was used to preipitate the asphaltene fraction from a gram of oil sample. The maltenes (de-asphalted oil sample) were extracted in Soxhlet extractor from the precipitate after filtration. The liquid adsorption chromatography in aluminum oxide was used to separate maltenes. The chromatography column had a diameter and length of 20 × 500 mm. Neutral aluminum oxide calcined at temperature of 450 ◦C for 3 h (as per TU 6-09-3916) was used to fill the column. After that maltenes were poured into the column and washed with hexane, toluene and toluene and methanol mixture to obtain saturates, aromatics and resins. All of the fractions were recovered from the solvents by distillation.

After SARA analysis saturates and aromatics were analyzed using Gas Chromotography and Mass Spectroscopy (GC-MS) in a Chromatec-Crystall 5000.2 chromatograph (“Chromatec”, Yoshkar-Ala, Russia) and Mass-spectrometer 214.2.840.083-10 (ions source ADVIS). NIST (National Institute of Standards and Technology) library and relevant literature sources were used for the identification of molecules and compounds.

3. RESULTS AND DISCUSSION

An acoustic method of cavitation control was used during this study. Acoustic spectra for different oils during ultrasound treatment is shown in Figure 3, measured voltage is corrected for the ease of reading.

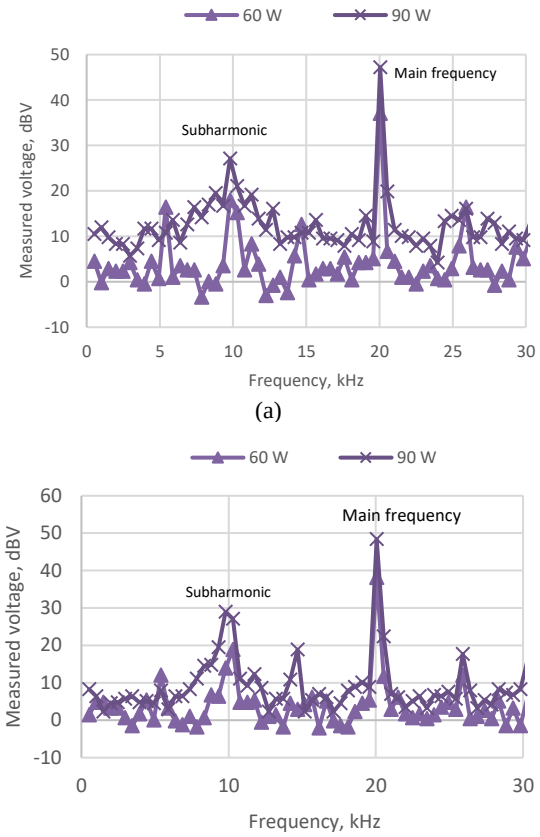


Figure 3. Acoustic spectra during sonification in: a) Ashal’chi crude oil, b) North-Komsomol crude oil

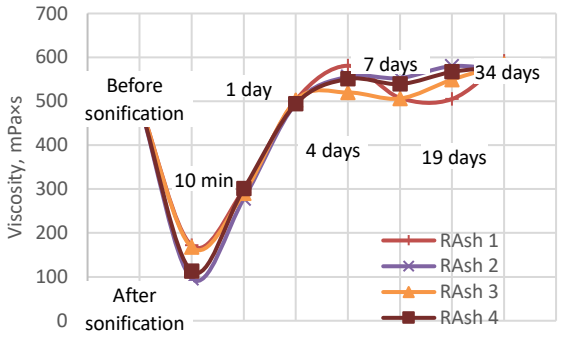
The highest peak on the spectra corresponds to the ultrasound signal (20 kHz). Subharmonic peak (10 kHz) corresponds to the intensity of cavitation process (34). Subharmonic peak appears at the power of ultrasound of 60 W. Due to the results of spectra measurements it was decided to perform all the tests in the range of 60-90 W. Treatment conditions are summarized in Table 1. It was decided to include the treatment duration of 10 min with pauses to avoid sample heating. Total duration of sonification in this case was 10 min.

Table 1 demonstrates that the biggest amount of heat was generated during the treatment at 90 W for 3 min. Pauses during the treatment allow significantly reduce heating.

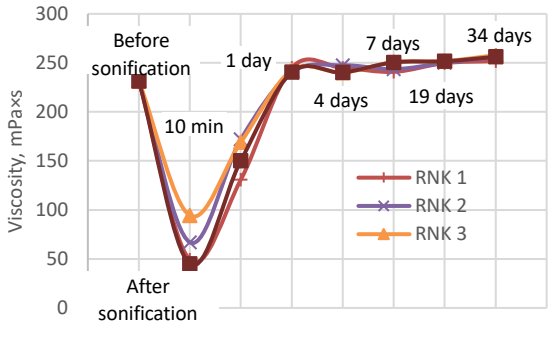
The viscosity measurements were performed before the treatment, after the treatment, 10 min after treatment and conditioning, and after 1, 4, 7, 19 and 34 days of relaxation at the temperature of 20 °C, 40 °C and 60 °C. During the relaxation samples were kept at ambient temperature of 20 °C. Measurement results are presented in Figure 4. Viscosity measurement temperature immediately after the treatment corresponds to the temperature in Table 1.

TABLE 1. Ultrasound treatment conditions.

Sample	Crude oil	Ultrasound power	Duration	Temperature at the end of tratment
RAsh 1	Ashal' chi	60 W	3 min	43.4 °C
RAsh 2		90 W	3 min	53.4 °C
RAsh 3		60 W	10 min with 1 min pause every minute	46.3 °C
RAsh 4		60 W	10 min	50.3 °C
RNK 1	North-Komsomol	60 W	3 min	43.1 °C
RNK 2		90 W	3 min	54.9 °C
RNK 3		60 W	10 min with 1 min pauses every minute	46.6 °C
RNK 4		60 W	10 min	52.3 °C



(a)



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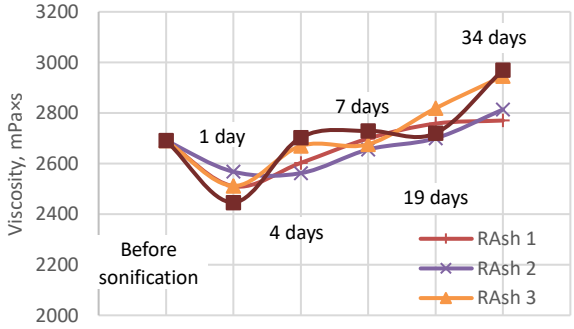

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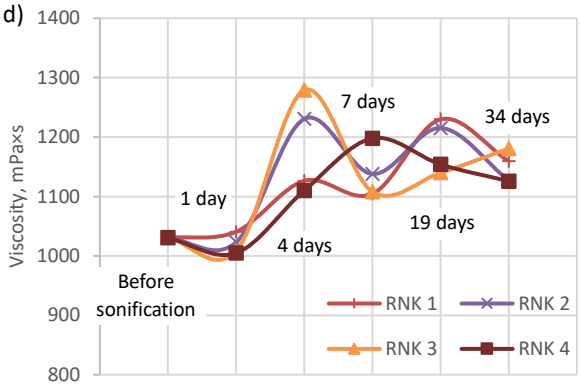

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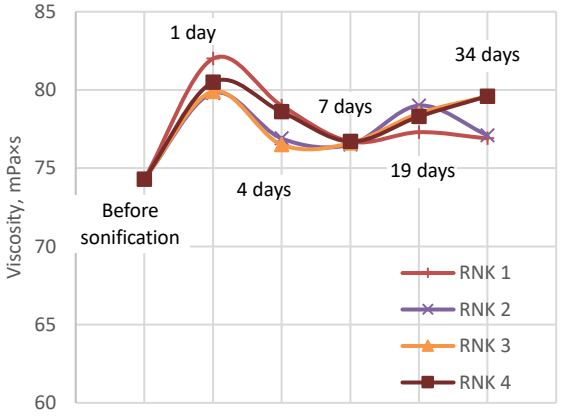

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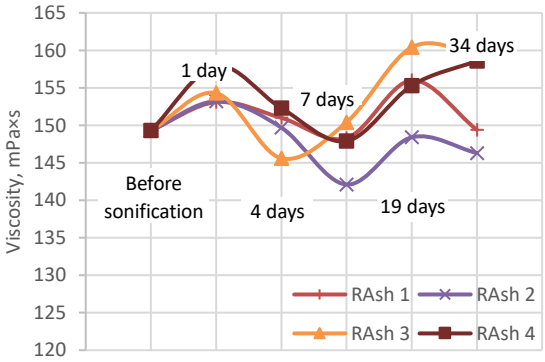


(f)

Figure 4. Crude oil viscosity after sonification: a) Ashal'chi 40 °C, shear rate 6.6 c-1, b) North-Komsomol 40 °C, shear rate 15.84 c-1, c) Ashal'chi 20 °C, shear rate 1.32 c-1, d) North-Komsomol 20 °C, shear rate 3.3 c-1, e) Ashal'cha 60 °C, shear rate 26.4 c-1, f) North-Komsomol 60 °C, shear rate 39.6 c-1

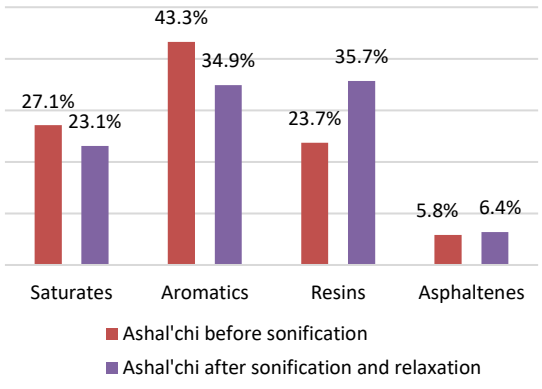
According to Figure 4, oil viscosity after 34 days increases to bigger level than before treatment, average increase is equal to 10% regardless of the treatment conditions. In the case of Figure 4, a, d, e, f an increase

of viscosity after 1-4 days can be seen. That effect can take place due to the formation of unstable temporary structure in the oil, which increases the oil viscosity. Ultrasound treatment itself can significantly reduce oil viscosity due to the oil heating and sonochemical destruction of the molecular structures and molecules, the viscosity of oil is significantly less after the treatment and conditioning than before the treatment. The viscosity reduction is equal to 80% after the treatment and 40% after the treatment and conditioning.

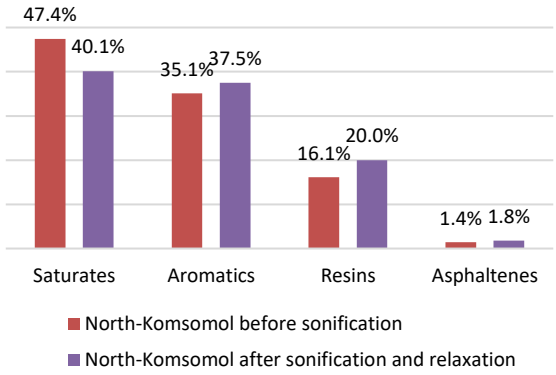
Measurement results indicate the possible molecular and aggregate structure destruction in crude oil after ultrasound treatment, which leads to the viscosity reduction. However, those changes are temporary, since molecular structure is tended to recover. During this process, temporary viscosity increase occurs, which indicates an unstable molecular structure appearance. After 7 days that temporary structures became more stable, making the oil less viscous, however viscosity is still higher than before the treatment due to the new molecular structure, which is formed under normal conditions. This new structure differs from an old structure, which was formed under the reservoir conditions.

The treatment conditions did not affect the viscosity changes significantly, that means that 3 minute and 60 W treatment is sufficient for the viscosity reduction of 100 mg of the crude oil.

After 34 days of relaxation SARA analysis of the samples exposed to 10 min of ultrasound without pauses was performed. The results are shown in Figure 5.
Ultrasound treatment and relaxation lead to the increase of the polar components of oil and the reduction of saturates (Figure 5). Aromatics fraction is decreased in the case of Ashal'cha crude oil and increased in the case North-Komsomol crude oil. That means, during sonification and relaxation different parallel processes in the aromatic compounds occur. The reduction of saturates mean its destruction and association with the polar compounds, resins and asphaltenes.



(a)



(b)

Figure 5. SARA results before and after sonification and relaxation

The chromatogram of Ashal'chi crude oil before and after the ultrasound treatment and relaxation is shown in Firgure 6. After the treatment and relaxation C25-C29 alkanes peaks are appeared on the chromatogram.

Figure 7 presents the chromatogram of alkanes of North-Komsomol crude oil before and after an ultrasound treatment and relaxation. After the treatment and relaxation naphtenes peaks are increased as well as C14H30 and C17H36 alkanes.

In Figure 8, spectra of aromatics fraction of Ashalcha heavy oil sample before and after ultrasonic treatment and relaxation are presented. New peaks were observed on the specter after ultrasonic treatment, which corresponds to the benzenes with C12-C13 alkyl substitutes. Moreover, the intensity of the peaks corresponding to the benzene-thiophenes with C10H10S and C11H12S alkyls and 1,1-diphenylethane C14H14 was reduced.

In Figure 9, we illustrated the spectra of aromatics fractions isolated from the heavy oil samples of North-Komsomol field before and after sonification. We

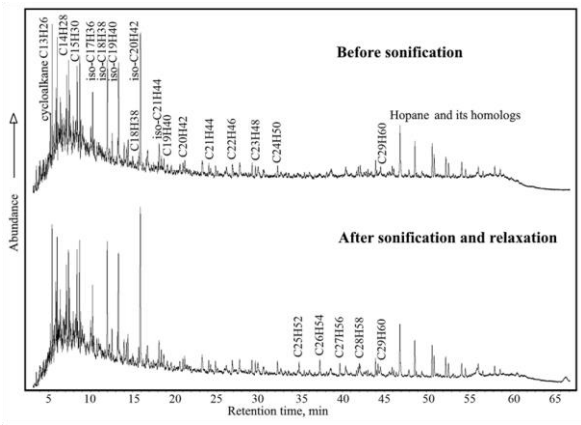


Figure 6. GC-MS spectra of saturates (m/z=57) isolated from Ashal'chi heavy oil samples

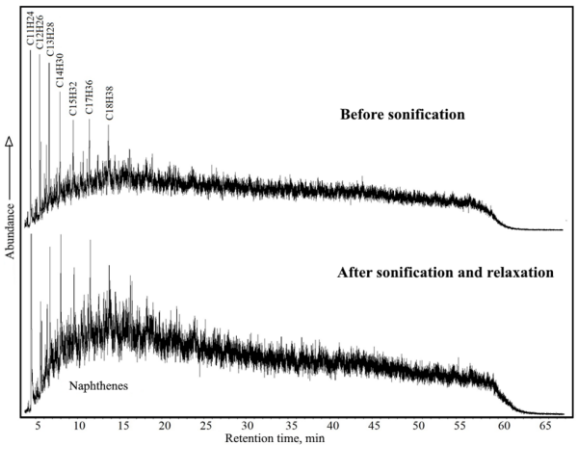


Figure 7. GC-MS spectra of saturates (m/z=57) isolated from North-Komsomol heavy oil samples

identified the new peaks corresponding to alkyl-tetra-hydro-naphthalenes and alkylbenzenes with the composition of C13H16.

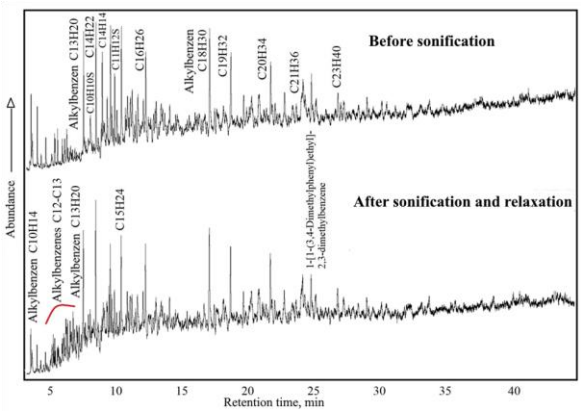


Figure 8. GC-MS spectra of aromatics isolated from Ashal'chi heavy oil samples

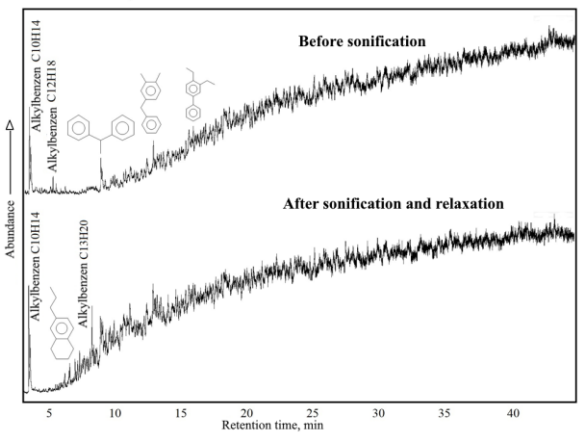


Figure 9. GC-MS spectra of aromatics isolated from North-Komsomol heavy oil samples

4. CONCLUSION

Ultrasonic treatment is a promising upgrading method to unlock the heavy oil production, transportation and refinery drawbacks. However, the long ultrasonic relaxation time can lead to the viscosity increase and decrease of light oil components. The following concluding remarks were achieved:

1. Ultrasonic treatment leads to instant heating of oil and a decrease in its viscosity, which is probably due to the destruction of its supramolecular structures.
2. A significant increase in viscosity after the relaxation period of 1-4 days, was observed, which may indicate the formation of large unstable supramolecular structures.
3. Stabilization of the structure and hence, viscosity was achieved after 7 days. However, the different power and sonication time under similar conditions did not have a noticeable effect on these processes.
4. Ultrasonic treatment of heavy oil with the long relaxation time contributed to the decrease in saturates and increase in heavy fragments such as resins and asphaltenes. The content of aromatics fraction was decreased in the composition of Ashal'cha heavy oil sample, while in case of North-Komsomol sample, it was increased. GC-MS results do not show significant changes of saturates and aromatics composition after ultrasonic treatment and relaxation.

In general, the results suggest that ultrasonic treatment can cause significant structural changes in oil and leads to a significant decrease in viscosity for the short period of time. This study expands the understanding of the behaviour of heavy oils with high resins and asphaltenes content after and during ultrasonic treatment in contrast to existing researches, where only short term effects were studied.

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Persian Abstract

چکیده

در این مقاله به بررسی تغییرات ویسکوزیته و ترکیب گروهی نمونه های نفت سنگین پس از فرآیند ارتقاء اولتراسونیک می‌پردازیم .نمونه‌های نفت سنگین مربوط به میادین نفتی مختلف) واقع در روسیه (تحت حالت‌های مختلف اولتراسونیک پردازش شدند که پیامد کاویتاسیون آن با روش آکوستیک کنترل شد .ویسکوزیته نمونه ها بلافاصله پس از درمان اولتراسونیک، 10 دقیقه و 34-1 روز اندازه گیری شد .تجزیه و تحلیل SARA پس از درمان اولتراسونیک با 34 روز آرامش انجام شد و نتایج به دست آمده با فراکسیون SARA نفت خام اصلی مقایسه شد .نتایج اندازه گیری ویسکوزیته کاهش ویسکوزیته را پس از درمان اولتراسونیک نشان داد .با این حال، ویسکوزیته پس از 4-1 روز آرامش با کاهش بیشتر در 7 روز پسرفت شد .درجه افزایش ویسکوزیته پس از 34 روز تنها 10 درصد در مقایسه با ویسکوزیته نفت خام اصلی بود .قدرت و زمان فراصوت بر روند آرامش تأثیری نداشت .با توجه به نتایج تجزیه و تحلیلSARA ، محتوای اشباع کاهش یافته و محتوای نسبی قطعات سنگین مانند رزین ها و آسفالتین ها افزایش یافته است که تعیین کننده درجه افزایش ویسکوزیته پس از دوره آرامش است.