The mechanism of ultrasonic irradiation effect on viscosity variations of heavy crude oil

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A B S T R A C T

Viscosity reduction of heavy oil has economic benefits and application value in the field. Applying viscosity reduction technology of heavy oil with ultrasound, this paper examines the influence of ultrasonic irradiation time and the addition of tetralin and ethylene glycol on viscosity reduction of heavy oil. Fourier Transform Infrared Spectrometer (FTIR) and Gas Chromatograph (GC) were used to analyze the chemical compositions and structural changes of oil samples before and after ultrasonic irradiation, and the effects of ultrasound on viscosity changes are presented. The viscosity reduction rate (VRR) was best at an irradiation time of 6 mins under the current experimental conditions, with VRR reaching more than 80%. When the irradiation time reached 12 mins, the viscosity of oil samples increased conversely. Chemical analysis reveals that the light components in the oil samples increased after the ultrasonic irradiation, indicating that the caviation, thermal, and mechanical effects of ultrasound could play a role in breaking long chains of carbon, thus reducing the viscosity. The increase in viscosity may have been caused by a reassociation between molecules and/or the coking of heavy oil.

1. Introduction

To date, fossil energy, such as oil and natural gas, has not been and cannot be completely replaced by new energy sources, such as wind and solar energy. However, when the exploitation of conventional petroleum and gas resources reaches a bottleneck, the exploration and effective utilization of unconventional energy such as heavy crude oil, become essential [1–3]. Even though the resource of heavy oil is rich, its characteristics of high viscosity, high density, and high impurity atom content make exploitation and transport difficult [4,5]. Therefore, reducing the viscosity of heavy oil is of great significance.

Compared with conventional methods of viscosity reduction including heating [6,7], dilution [8,9], and emulsification of oil in water [10–12], ultrasonic technology to reduce the viscosity of heavy oil is environment friendly, more economical, and energy saving [13–15]. Therefore, this technology has wide prospects in application, but the mechanism of ultrasonic effect on viscosity change of heavy oil is still unclear.

Jabber et al. [16] examined the influence of ultrasonic irradiation time on the changes of viscosity and the structure of heavy oil, and reported that acoustic cavitation power could break large asphaltene molecular particles into much smaller ones, thus disintegrating resin intermolecular bonds and causing variations in viscosity. Shi et al. [17] hypothesized that ultrasonic irradiation destroyed the ‘cage effect’ of heavy oil samples through the severe cavitation and mechanical vibration, and also damaged the C—C bond, leading to a reduction in viscosity. Huang et al. [18] applied Fourier Transform Infrared Spectrometer (herein referred to as ‘FTIR’) to analyze the residual oil samples and reported the largest VRR after ultrasonic treatment. Their researches indicated that the increase in the content of light component alkanes was caused by the high temperature, high pressure, and high speed microjet generated by ultrasonic cavitation.

Some scholars [16–18] have claimed that ultrasound can break the carbon chains, one of the main reasons for the reduction in viscosity. However, limited research has applied relevant chemical analysis methodologies to prove that the change in the structure of oil samples was caused by a break in the carbon chains. Recently, several

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researchers [19–21] have also applied chemical analysis methods to analyze oil samples, however, most have given experimental results of ultrasonic viscosity reduction. This paper shows that ultrasound can not only reduce viscosity, but also increase it under different ultrasonic irradiation time. The carbon number distributions and chromatographic distillation ranges of the heavy oil samples before and after ultrasonic irradiation were obtained with the Gas Chromatograph (herein referred to as 'GC'). FTIR was also applied to analyze the mechanism by reporting the changes in the functional groups. Furthermore, tetralin and ethylene glycol mixed with heavy oil were used to study the differences in the principles and mechanism of ultrasonic irradiation.

2. Methodology

2.1. Materials

The raw material, heavy crude oil, was extracted from an oil field in western China. The viscosity under different temperatures of the dehydrated heavy crude oil was measured by a rheometer (see section 2.2) and shown in Table 1. The element content was obtained with an Element Analyzer and is provided in Table 2.

The tetralin was purchased from Sinopharm Chemical Reagent Co., Ltd., China, with a molecular formula of C10H12, a density at 20 °C of 0.968–0.970 g/ml, and a boiling point of 205–209 °C. For this paper, C10H12 was used to dilute the heavy oil and stabilize the liquid system after ultrasonic irradiation.

The ethylene glycol was also purchased from Sinopharm Chemical Reagent Co., Ltd., with a chemical formula, density (at 20 °C), boiling point of (CH2OH)2, 1.111–1.115 g/ml, 196–198 °C, respectively. Ethylene glycol was used to increase the intensity of ultrasonic cavitation in segments of the experiments.

2.2. Instruments

A counterbalance was applied to weigh the quality of various liquids used in the experiments. A volumetric cylinder and dropping pipette were used to measure the volume of C10H12 and (CH2OH)2. A thermostatic water bath was used to heat the oil samples held in beakers.

The ultrasonic generator utilized is composed of ultrasonic power amplifier and ultrasonic transducer produced in the Institute of Acoustics, Chinese Academy of Sciences, and whose frequency and electric power are adjustable. The RV-DV-III type rheometer system was obtained from BROOKFIELD Company and used to measure the viscosity of oil samples with changes in temperature before and after ultrasonic irradiation.

NICOLET6700 type FTIR (American ThermoFisher Scientific Company) and Agilent7890B type GC (American Agilent Technologies Company) were applied to analyze the chemical compositions and structural changes of the oil samples before and after ultrasonic irradiation.

2.3. Process

2.3.1. Preparation of oil samples

The experiment procedure is shown in Fig. 1. Three kinds of oil samples were prepared: heavy crude oil, heavy oil with C10H12 mixed at a mass ratio of 3:1, and heavy oil with C10H12 and (CH2OH)2 intermixed at a mass ratio of 6:1:1. Each oil sample (60 ml) was placed in a separate beaker.

2.3.2. Comparison of the dissolution rate

While the heavy oil, C10H12 and (CH2OH)2 were mixed in different proportions, they were dissolved in three different ways: (1) kept at room temperature (about 28 °C); (2) heated in a 90 °C water bath; (3) irradiated with ultrasound. The dissolution rates could be compared by the time the liquids had completely dissolved.

2.3.3. Ultrasonic irradiation

The center frequency and electric power of the transducer were approximately 18 kHz and 250 W, respectively. The ultrasound horn was inserted into the center of each beaker and held at approximately 1/2 the depth of the oil samples. Ultrasonic irradiation time was set at 6 mins and 12 mins. The oil samples were first irradiated for 2 mins, then cooled to about 90 °C at room temperature, continued to be irradiated for 1 min, and then cooled to 90 °C at room temperature. This process was repeated for 6 mins or 12 mins. Details of the experimental procedures are shown in Table 3.

2.3.4. Measure of viscosity variation

Firstly, the original viscosities of the heavy crude oil, the heavy oil with C10H12, and the heavy oil with C10H12 and (CH2OH)2 were measured with a rheometer in Experiments 1, 4, 8, respectively. The changes in viscosity with temperature were also given, with an accuracy of ± 0.1 °C. It is worth nothing that the No. 21 rotor of the rheometer was applied in all experiments, and the torque was controlled between 30% and 70%. The viscosity was repeatedly measured until the measurement results were approximately equivalent, within an accuracy of ± 1% of the measurement value. Secondly, the viscosities of the oil samples after the ultrasonic irradiation were measured at different temperatures. Finally, the viscosity reduction rates were calculated with the following formula:

$$VRR = \frac{\mu_0 - \mu}{\mu_0} \times 100\%$$

where VRR (%) is the viscosity reduction rate, $\mu_0$ (Pa·s) is the original viscosity of the oil samples, and $\mu$ (Pa·s) is the viscosity at the end of the ultrasonic irradiation.

2.3.5. Chemical analysis

Chemical analysis can characterize the internal structure variations of oil samples before and after the ultrasonic treatment, which is an essential step in revealing the mechanism of ultrasound effects on viscosity changes. In the experiments for this study, different types of oil samples before and after ultrasonic irradiation are analyzed by FTIR and GC. FTIR can determine changes in category and content of oil samples. In addition, the chromatographic distillation range and carbon number distribution of different oil samples could be obtained through GC, and the test method ASTM D7169 is used here. Finally, the mechanism could be revealed by comparing the disparate experimental principles and chemical analysis results.

### Table 1
Viscosity of the original heavy crude oil under varied temperatures.

| Temperature/°C | 65 | 70 | 75 | 80 | 85 | 90 | 95 |
|----------------|----|----|----|----|----|----|----|
| Viscosity/μ Pas | 625.00 | 312.50 | 166.67 | 83.33 | 43.10 | 25.77 | 14.21 |

### Table 2
Elemental content of original heavy crude oil.

| Element | C | H | O | N | S |
|---------|---|---|---|---|---|
| Content/% | 86.03 | 10.09 | 0.42 | 0.49 | 2.28 |
3. Results and discussion

3.1. Viscosity reduction results

3.1.1. Comparison of dissolution rates

The contrastive results of dissolution rates for different compositions of samples are shown in Table 4. “Reaction time” represents the time of heating in a water bath, placing at room temperature, or irradiating by ultrasound. “Time of dissolving completely” represents the time required for two or three reagents to completely dissolve.

When the heavy oil and C_{10}H_{12} were mixed at a mass ratio of 3:1, the oil sample at room temperature only partially dissolved. By comparison, it took the oil sample 3 h to dissolve completely in the 90 °C water bath, and 1 min to dissolve completely with ultrasonic irradiation.

When the heavy oil, C_{10}H_{12} and (CH_{2}OH)_{2} were mixed at a mass ratio of 6:1:1, the oil sample at room temperature dissolved a little, and it took the oil sample 4 h to fully dissolve in the 90 °C water bath.

However, it took the mixed sample for only 1 min to completely dissolve with ultrasonic irradiation.

These results suggest that strong mechanical effect, thermal effect, and acoustic cavitation effect of ultrasound can possibly accelerate the dissolution of heavy oil and other solvents.

3.1.2. Ultrasonic irradiation on heavy oil

The viscosity changes with the increasing temperature are shown in Fig. 2. “Time of dissolving completely” represents the time required for two or three reagents to completely dissolve. In order to clearly demonstrate the difference in viscosity from 80 °C to 90 °C, a histogram is given in the upper right corner of Fig. 2(a). The blue color represents the viscosity of the original heavy crude oil; the gray color represents the viscosity of the heavy crude oil after 6 mins of ultrasonic (US) irradiation; and the yellow color marks the viscosity of the heavy crude oil after 12 mins of ultrasonic reaction. The changes of VRR are shown in Fig. 2 (b), with the solid and virtual sphere describing the VRR after irradiating for 6 mins and 12 mins, respectively.
As can be seen, the viscosity of heavy oil samples decreased with an increase in temperature with or without ultrasonic irradiation. The oil sample’s viscosity decreased by approximately 20% when irradiated with ultrasound for 6 mins. Conversely, when the ultrasonic irradiation time was increased to 12 mins, the viscosity increased, and it may even exceed the original viscosity of the heavy crude oil.

3.1.3. Ultrasonic irradiation on heavy oil & \( \text{C}_{10}\text{H}_{12} \)

Fig. 3(a) presents the changes in viscosity of heavy oil and \( \text{C}_{10}\text{H}_{12} \) mixed at a mass ratio of 3:1. While the viscosity of both oil samples decreased, whether irradiated for 6 or 12 min, the sample irradiated with ultrasound for 6 min exhibited a more rapid decline in viscosity. Fig. 3(b) shows that the VRR follows a decreasing trend along with an increase in temperature, with the curve gap tightening between 6 and 12 mins.

The VRR rate for the mixed sample was much higher than that of the heavy oil sample, thus suggesting that the addition of \( \text{C}_{10}\text{H}_{12} \) was helpful in improving the VRR.

3.1.4. Ultrasonic irradiation on heavy oil & \( \text{C}_{10}\text{H}_{12} \) & \( (\text{CH}_2\text{OH})_2 \)

The viscosity variations of heavy oil, \( \text{C}_{10}\text{H}_{12} \) and \( (\text{CH}_2\text{OH})_2 \) mixed at a mass ratio of 6:1:1 are given in Fig. 4(a). The effect on the irradiated oil samples for 6 mins was greater than those for 12 mins, indicating that the VRR was much higher in conjunction with the change in temperature.

Taken together, the above analyses show that the descending order of the ultrasonic reaction on the overall viscosity reduction effect of different oil samples was as follows: the mixture of heavy oil & \( \text{C}_{10}\text{H}_{12} \), the mixture of heavy oil & \( \text{C}_{10}\text{H}_{12} \) & \( (\text{CH}_2\text{OH})_2 \), and the heavy oil.

3.2. Chemical analysis results

3.2.1. FTIR

FTIR results of ultrasonic irradiation on heavy oil

FTIR spectra results are shown in Fig. 5. The red, green, blue curve represent the results of heavy oil, heavy oil with ultrasonic (US) irradiation for 6 mins and 12 mins, respectively. Fig. 5(a) presents the results for each sample, while Fig. 5(b) compares the data across the three groups. Figs. 1 and 2 enclosed in Fig. 5(b) are the partial amplification of the results.
As revealed in the divergent curves, it can be concluded that angular vibrations of methyl and methylene existed at about 1378 and 1458 cm\(^{-1}\). The characteristic peak of the carbon–carbon double bond appeared at 1599 cm\(^{-1}\). The stretching vibration of methylene appeared at about 2854 and 2924 cm\(^{-1}\), as did the methyl at about 2952 cm\(^{-1}\).

When the heavy oil was irradiated for 6 mins by ultrasound and had about 20\% VRR under different temperatures, the intensity of each peak increased. This strongly indicates that the contents of the functional groups such as methyl increased. There may have been broken chains of some heavy components, thus causing the changes in functional group contents and viscosity reduction.

However, when the viscosity increased after 12 mins of reaction, the peak intensity decreased. Due to the increase of ultrasonic irradiation time, more long chains would be broken into short chains in theory, but in reality, the temperature threshold which short chain molecules could withstand was reduced, with possible volatilization of some light components, thus resulting in increased viscosity.

FTIR results of ultrasonic irradiation on heavy oil & \(\text{C}_{10}\text{H}_{12}\)

Fig. 6(a) shows the FTIR spectra results of heavy oil with \(\text{C}_{10}\text{H}_{12}\) when the samples were irradiated for 6 mins and 12 mins. In addition to the characteristic peaks previously mentioned, Fig. 6(a) shows a stretching vibration of \(=\text{C–H}\) on aromatics at about 3016 and 3059 cm\(^{-1}\).
Fig. 6. FTIR spectra of different heavy oil & C_{10}H_{12} samples.

Fig. 7. FTIR spectra of different heavy oil & C_{10}H_{12} & (CH_{2}OH)_{2} samples.
There was a characteristic peak of the benzene ring at the position of 1602 cm\(^{-1}\). The results also display an out-of-plane bending vibration of \(=\text{CH}\) at about 741 cm\(^{-1}\).

The intensity of each peak decreased when heavy oil \& \(\text{C}_{10}\text{H}_{12}\) were irradiated with ultrasound. When the ultrasound was applied for 6 mins, FTIR showed that compared with Fig. 6(a)-(1), the contents of functional groups decreased, and the light components may increase due to bond breaking reaction [18,22], resulting in a decrease in viscosity.

When the reaction lasted for 12 mins, a repolymerization between molecules may have also occurred. However, the presence of \(\text{C}_{10}\text{H}_{12}\) maintained a low degree of polymerization. As a result, compared with Experiment 3, the degree of increase in viscosity was not high.

FTIR results of ultrasonic irradiation on heavy oil \& \(\text{C}_{10}\text{H}_{12}\) \& \((\text{CH}_{2}\text{OH})_2\)

Fig. 7(a) presents the FTIR spectra results of different mixture samples of heavy oil with \(\text{C}_{10}\text{H}_{12}\) and \((\text{CH}_{2}\text{OH})_2\). Apart from the previously mentioned functional group characteristic peaks, the hydroxyl characteristic peak and C-OH stretching vibration of alcohol appeared at about 3370 and 1040 cm\(^{-1}\) were observed.

The intensity of each peak decreased when heavy oil with \(\text{C}_{10}\text{H}_{12}\) and \((\text{CH}_{2}\text{OH})_2\) were irradiated for 6 mins with ultrasound, reaching about 60\% VRR under different temperatures. The difference in peak height indicated that the contents of different functional groups had changed greatly, and the fracture of the chemical bonds led to a decrease in viscosity. However, different from Experiment 6, the decrease of VRR may have been caused by a decrease in the volume of \(\text{C}_{10}\text{H}_{12}\), leading to a decrease in the stability of the system after ultrasonic irradiation.

However, when the viscosity increased after 12 mins of reaction time, the intensity of the peaks increased probably due to other factors that had also affected the viscosity, such as the reassociation of molecules and/or the coking of oil samples.

As the viscosity of the oil samples under various conditions altered, the intensity of peaks changed as well, indicating that the changes in functional group contents may have been an influential factor in the viscosity changes on the micro level.

3.2.2. GC

Analysis of chromatographic distillation range

The flame diagram made through the analysis results of chromatographic distillation range is shown in Fig. 8. The deeper the color in the diagram, the higher the temperature. The corresponding temperatures of the distillates of five varieties are given in Table 5, where IBP represents the initial boiling point.

For the heavy oil samples which involved distillate oil and heavy component oil, the contents of the two components decreased with the increase of ultrasonic irradiation time. However, the viscosity of oil samples was higher in Experiment 3, which can possibly be explained by the evaporation of some light components with the passage of time.

In Experiments 4 and 8, the heavy components increased and the light components lessened compared to Experiment 1. Moreover, the contents of gasoline, kerosene, and diesel oil rose, but those of distillate oil and heavy component oil decreased under ultrasonic irradiation on the whole (there may have been volatilization of some light components in Experiment 10). Therefore, the increase of light components and the decrease of heavy components may have been one of the factors affecting VRR of the oil samples.

Table 5

| Variety | Gasoline | Kerosene | Diesel oil | Distillate oil | Heavy component oil |
|---------|----------|----------|------------|---------------|---------------------|
| Temperature range (°C) | IBP-180 | 140-240 | 180-350 | 350-500 | >500 |

Fig. 8. Analysis results of chromatographic distillation range on different oil samples.
Carbon number distribution results

The carbon number distribution results of the different oil samples including less than carbon 10, from carbon 11 to 20, from carbon 21 to 40, and greater than carbon 40 are shown in Fig. 9.

No more than carbon 20 was present in the heavy oil samples. The total carbon contents, called long chains, gradually decreased with the increase of ultrasonic irradiation time. Viscosity in Experiment 3 increased, possibly due to the volatilization of light components.

As for Experiments 4, 6, and 7, the carbon chains became shorter than the experiments with heavy oil. The contents of short chains below carbon 20 increased little, while those of long chains reacted conversely. The factor that affected Experiment 7 in obtaining a higher viscosity may have been similar to that in Experiment 3.

The mixture of heavy oil with C_{10}H_{12} and (CH_{2}OH)_{2} exhibited a similar change in principles, except in Experiment 10. This may possibly be explained as follows: the long chains of macromolecules were interrupted first and then reassociated [16]. Higher viscosity obtained in Experiment 11 probably resulted from the volatilization of light components.

3.3. Mechanism

When ultrasound of a certain intensity irradiates oil samples, it usually forms severe cavitation in the liquid. Acoustic cavitation is generally believed to have three effects: mechanical effect, chemical effect, and heat effect. The mechanical effect usually produces a strong stirring effect on the mixed oil samples, and the thermal effect produces high temperature and high pressure in the local solution, thus promoting the mutual dissolution of different liquids.

For the three heavy oil experiments, viscosity first decreased and then increased. The contents of the functional groups increased, which indicates that there may have been a breaking-chain effect. The contents of heavy components decreased, which indicates that ultrasound could have decomposed the heavy components. This paper suggests that the decrease in viscosity caused by ultrasound may have been due to the interruption of long chains and the decrease of heavy components due to the acoustic cavitation. When the irradiation time continuously increased, there may have been volatilization of some light components because of the effects of local high temperature and high pressure.

For the heavy oil with C_{10}H_{12} experiments, which resulted in the highest VRR, the viscosity decreased when irradiated for 6 mins and increased again after 12 mins. FTIR results show that the acoustic cavitation had caused the reduced contents of the functional groups. The analysis of chromatographic distillation range show light components increased and heavy components decreased with the increase of ultrasonic irradiation time. In addition, the contents of less than carbon 20 received more, while the ones of more than carbon 20 received less. This indicates that acoustic cavitation produced by ultrasonic irradiation could have changed the chemical structure of the oil samples, and compared with Experiments 1–3, the experiments with heavy oil with C_{10}H_{12} displayed more obvious results. Thus, it can be argued that the C_{10}H_{12} can stabilize the reaction system and effectively reduce the recovery of the system after ultrasound [23].

As for the experiments with heavy oil with C_{10}H_{12} and (CH_{2}OH)_{2}, the viscosity first decreased and then increased, and the VRR was lower than Experiments 6–7. Besides for the increase of light components, the increase in heavy components and carbon numbers greater than carbon 21 may have been due to the volatilization of some light components. This may be explained by the following two factors: (1) The acoustic cavitation has produced the local high temperature and pressure, which reached the boiling point of the mixed liquid, thus possibly causing the volatilization of some light components; (2) The decrease of C_{10}H_{12} content may have reduced the stability of the system, thus causing the reunion effect of broken bonds. However, the increase of acoustic cavitation effect resulted from the addition of (CH_{2}OH)_{2} and made the system more complex. The specific reasons for this require further research.

![Fig. 9. Analysis results of carbon number distribution on different oil samples.](image-url)
In summary, the cavitation effect, mechanical effect, and thermal effect generated by ultrasonic action had a strong impact on the mixture system of heavy oil [24–26]. However, due to these effects and the complexity of heavy oil itself, though experimental results of this study can support the fact that the chemical structure could be changed with ultrasound, the mechanism by which that chemical structure had changed cannot be easily explained by only one or two effects caused by the ultrasound; thus, more methods need to be introduced to reveal the mechanism of ultrasonic effects on the variation of the heavy oil samples viscosity.

4. Conclusion and prospects

This study reveals that the viscosity of different oil samples significantly decreased after ultrasonic treatment for 6 mins (VRR was greater than 20% or so), but when ultrasound continued for 12 min, the viscosity increased, with the viscosity of some oil samples higher than the initial viscosity. Moreover, this study confirms that ultrasonic action can break the inherent balance of the heavy oil system, increase the contents of light components, and thus reduce viscosity. However, the excessive action time may lead to a reassociation between molecules, the volatilization of light components, and/or the coking of oil samples, resulting in an increase in viscosity. In addition, due to the complexity of heavy oil itself, and the influence of principles, the mechanism of different ultrasonic irradiation time, and intensity of acoustic cavitation on the viscosity of heavy oil still needs further investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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