

Acoustically aided coalescence of water droplets and dehydration of crude oil emulsion

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Abstract—We studied the use of acoustics for coalescence of water droplets and dehydration of crude oil emulsion. Experimental studies were conducted using acoustic standing waves in a resonant cavity to trap water droplets and enhance oil separation. The focus was on the effect of ultrasound irradiation on crude oil emulsion properties, such as viscosity, water drop radius, shear strength of oil-water interfacial films, and flocculation size of asphaltene. These properties are important to the coalescence of water drops in water-oil (W/O) emulsion in the process of oil separation with ultrasound. Ultrasound irradiation is able to decrease the emulsion stability, which provides a new insight into the acoustics-aided demulsification mechanism. It can be considered as a supplement of traditional acoustics-aided demulsification mechanism (ultrasound-induced motion of water droplets). Furthermore, the effects of ultrasonic parameters such as the type of ultrasonic field, irradiation time, frequency, and acoustic intensity on dewatering the W/O emulsion are discussed. These results provide guidance for setting the optimum conditions for the separation of W/O emulsion with ultrasound. Under the optimum conditions, water content in crude oil emulsion can be decreased from 40% to 3.8%, which satisfies the requirement of dehydration for refinery.

Keywords: Acoustics, Ultrasonic Parameters, Dehydration

INTRODUCTION

Separation and recovery of the oil phase from W/O emulsions has practical applications in many situations, including environmental remediation, food processing, and petroleum refining. Chemical methods involve the addition of demulsifier to enhance the phase separation efficiency by changing the interfacial tension between oil and water. Although these methods are fast, they are expensive and have the disadvantages of having to remove the additives in subsequent processing steps. Physical methods often include gravity settlers and application of external centrifugal or electric fields [1], which can enhance phase separation by helping migration, collisions, and subsequent coalescence of dispersed phase droplets. However, large residence times or large physical spaces are required in these methods. In addition, difficulties in scale-up and maintaining high efficiencies on a large scale over longer periods may pose problems in using these processes for practical applications.

In the recent years, acoustic technology has been applied to liquid-liquid systems. This method is a potent technology for nucleation, coagulation, and coalescence, which is easy, simple, and more efficient to implement. Moreover, devices for irradiation can be easily installed into common equipment and the irradiation conditions can be adjusted to satisfy the objectives.

Check [2] used two periods of ultrasonication for the dehydration and desalting of heavy crude oil. Zhang [3] applied ultrasound to enhance the industrial sludge settling ability and dewatering ability. Carney [4] separated a stable water-oil (W/O) emulsion of

wastewater from washing raw wool using 20 kHz, of ultrasound, followed by electrolysis. Pangu and Feke [5] applied a low-intensity, resonant ultrasonic field to assist the coalescence of oil droplets and recover oil from an aqueous emulsion in a cavity with porous material. Crum [6] combined ultrasound and electrolysis techniques to flocculate and coalesce micelles for oil recovery. Jiao [7] found that cavitation bubbles migrate to pressure antinodes and coalesce when subjected to an ultrasonic standing-wave field. Browne [8] applied an ultrasound field (213 kHz) to assist the coalescence of bubbles in aqueous electrolyte solutions. Zhang [9] applied ultrasound to recover oil from oily sludge and achieve satisfactory oil recovery rate and total petroleum hydrocarbon (TPH) concentrations. Jin [10] recovered oil from oil sludge through combined ultrasound and thermochemical cleaning treatment. Check [11] dehydrated crude oil by ultrasonic irradiation in a novel batch of standing-wave resonator reactor. Nii [12] irradiated 2 MHz of ultrasound to the emulsion prepared from canola oil and water, and found that flocculation of oil droplets occurred immediately.

We focused on the effect of ultrasound on the properties of W/O emulsion, which is important for oil separation with ultrasound. Furthermore, the effects of ultrasonic parameters on the separation degree were also investigated. Results would provide guidance for setting the optimum conditions for the separation W/O emulsion with ultrasound.

THEORY

1. Acoustic Effect on Emulsion Stability

Developing rational and systematic approaches for demulsification is difficult because of the lack of full range of factors that govern emulsion stability. However, recent studies have found that com-

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ponents, such as asphaltenes and resins, accumulate at the oil/water interface because of their surface-active and structure-forming properties, and hence, have pronounced effects on emulsion stabilization and breaking [13-16]. These surface-active constituents stabilize water-in-oil emulsions only when they are near or above the point of incipient flocculation, suggesting that their mode of action is to collect at the interface in the form of finely divided solid particles or aggregates [17,18]. In addition, strong indications have demonstrated that asphaltene molecules slightly affect the stabilization of emulsion when dissolved in the oil phase in the form of a molecular solution. By contrast, colloiddally dispersed asphaltenes significantly stabilize emulsion [19].

To date, ultrasonic technology has been regarded as a potent method for reducing asphaltene flocculation rates because it can change the kinetics of aggregation and remove deposits. Asphaltene flocs in sonicated samples are smaller than those in non-sonicated samples. Therefore, generation of macrostructure flocs and interfacial film toughness can be remarkably reduced when the emulsion is irradiated by ultrasound.

2. Acoustic Forces Acting on a Drop

Ultrasound irradiating can produce a net force on the drops in the emulsions because the density and compressibility are different between the dispersed phase and the continuous phase drops.

2-1. The Primary Acoustic Force

When the distance between individual drops is long enough, the motion of individual drops is determined by body forces, including gravity, buoyancy, and primary acoustic force.

For drops smaller than the half-wavelength of sound, the application of a 1D resonant acoustic field results in a time-averaged force in a direction parallel to the sound field propagation direction. This primary acoustic force is given by [20-25]

$$F_{1,ac} = 4\pi R^3 \kappa E_{ac} F \sin(2\kappa y) \quad (1)$$

where R is the drop radius (m), E_{ac} is the average energy density of the acoustic field (J/m^3), y is the position relative to a pressure antinode, and F is the acoustic contrast factor that quantifies the density and compressibility difference between the drop and the continuous phase. The acoustic contrast F for the particle is given by [26]

$$F = \frac{A + 2(A-1)/3}{1 + 2A} - \frac{1}{3\sigma^2 A} \quad (2)$$

where A is the ratio of particle density to the fluid density and σ is the ratio of longitudinal sound speed in a particle to that in the fluid.

After simple stability analysis of Eq. (2), the value of F for W/O emulsion is positive. The primary acoustic force drives the drops toward the pressure nodes of the acoustic field [20]. Given that the primary acoustic force can be as strong as several hundred times that of gravity, the motions of drops can be relatively accelerated.

2-2. The Secondary Acoustic Force

When two drops are in close proximity (located at the pressure node or antinode), the secondary acoustic forces may become significant [20-25]. The secondary radiation force can be expressed as follows:

$$F_{2,ac} = \frac{\kappa E_{ac}}{2\pi} \left(1 - \frac{\gamma_d}{\gamma_f}\right)^2 \frac{V_2 V_1}{r^2} \quad (3)$$

where V_i is the volume of drop, r is the center-to-center distance between drops, γ_d is the compressibility of the fluid, and γ_f is the compressibility of the drop.

The secondary acoustic force acts along the line of the centers and can contribute to the coalescence of the two droplets. The secondary acoustic force at the start of ultrasound irradiation is much smaller than the primary acoustic force. However, agglomeration is induced and smaller droplets become larger ones when droplets are gathered at the antinodes (or nodes). Acoustic force can grow rapidly because it is proportional to particle volume.

Pangu [5] also summarized that the trajectory model of the agglomeration of droplets can be divided into two regimes. (a) Droplets are driven to equilibrium positions near the pressure node (or antinodes). This process is fast because of the gravity and the primary acoustic force. (b) The droplets subsequently approach collision slowly because of the combination of the secondary acoustic forces and van der Waals forces.

MATERIAL AND METHODS

The experiments were conducted in a rectangular constant-temperature chamber. Fig. 1 shows the schematic of the chamber, which consists of a PZT transducer (a rectangular flat), an ultrasound generator (20/40/80 kHz), a hydrophone, and an oscillograph. The PZT transducer was fixed on top of the chamber and a screw bolt was used to adjust the distance between the transducer surface and reflector to ensure that the chamber stayed at a standing wave field condition with high fraction of the power delivered to the emulsion. The distance must be kept constant at an odd number of the acoustic half wavelengths: $h = n\lambda/2$. After the standing wave field has been established, the ultrasound intensity at the antinodes may be four times that of the incident wave [27,28]. Fig. 2 demonstrates the sound intensity distribution of 20 kHz of acoustic standing-wave field in the rectangular chamber. The ultrasonic field was produced by energizing the transducer at various frequencies (20/40/

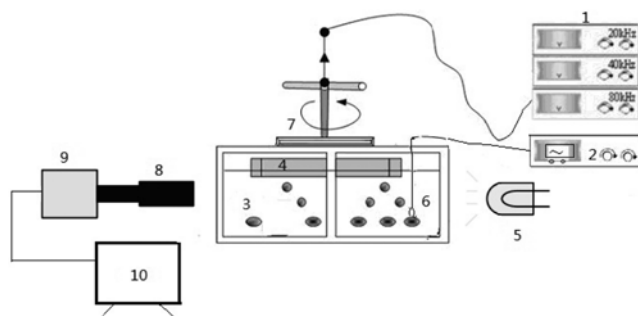


Fig. 1. Experimental device of ultrasonic dehydration.

- | | |
|--|----------------------|
| 1. Ultrasonic generator (20/40/80 kHz) | 6. Hydrophone |
| 2. Oscillograph | 7. Screw bolt |
| 3. Acoustic chamber | 8. Macro lens |
| 4. Ultrasound transducer | 9. High-speed camera |
| 5. Light | 10. Computer |

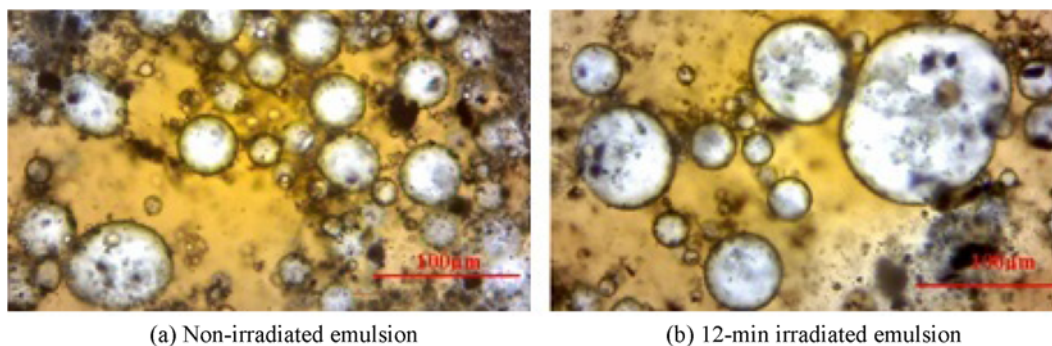


Fig. 2. Comparison of the confocal image of crude oil emulsion (Frequency: 20 kHz; Acoustic intensity: 0.4 W/cm²; Temperature: 60 °C; Standing-wave field).

Table 1. Characteristics of LU-NING pipeline oil

| Species | Value | Species | Value |
|--|-------|---------------------------------|-------|
| API° | 22 | Salt content (mg NaCl /L) | 31.93 |
| Density (20 °C) (g/cm ³) | 0.92 | Water content (% v.) | 0.25 |
| Kinematical viscosity (50 °C) (mm ² /s) | 108 | Carbon residue (% v.) | 6.41 |
| Pour point (°C) | 19 | Ash (% v.) | 0.02 |
| Acid value (mg KOH/g) | 1.92 | Characteristic factor (K value) | 11.75 |

80 kHz).

The chamber was filled with emulsions prepared from an RU-NING crude oil (provided by Sinopec Yangzi Petrochemical Company and Characteristics showed in Table 1) and deionized water to obtain 40 volume percentage of water in the mixture. The whole emulsion was mixed thoroughly with a high-speed paint mixer for 30 min, and then ultrasonicated (using a home-built Sonicator) for 10 min to ensure homogeneity of the oil samples.

The number, size, and size distribution of the droplets formed were accomplished during and after ultrasonication using an optical microscope fitted with a high-performance computer-controlled digital camera. Viscosity and shear strength of oil-water interfacial films of the W/O emulsion were measured immediately with an automatic viscometer (miniQV-X) and an interfacial shear viscometer (SVR-S). After W/O emulsion was irradiated by ultrasound in the rectangular acoustic chamber, and gravity settled for 10 min at 60 °C, the upper layer of the emulsion was removed with a pipette, and then the water content of the upper layer of the emulsion was analyzed by the method of distillation (GB/T8929-1998 measurement of water content in crude oil). To get reproducibility, we repeated every experiment four times and the final experimental data was a mean of three results, whose errors were not more than 5%.

RESULTS AND DISCUSSION

1. Effect of Ultrasound on the Properties of W/O Emulsion

1-1. Radius of Water Droplets in the Emulsion

Fig. 2 demonstrates the confocal images of non-irradiated crude oil emulsion and 12 min irradiated crude oil emulsion. The size of

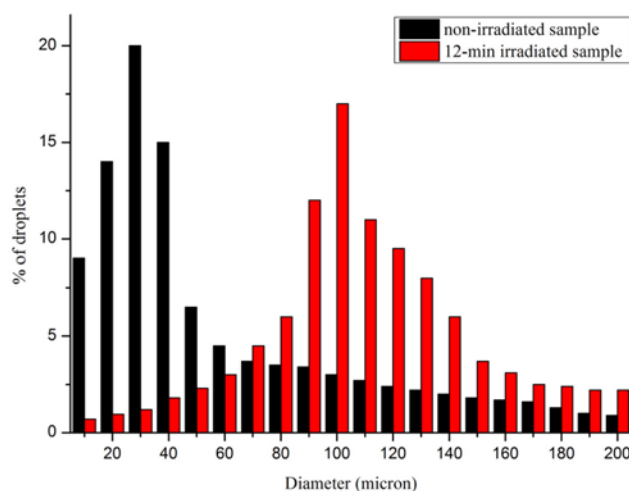


Fig. 3. Evolution of water droplet size distribution (Frequency: 20 kHz; Acoustic intensity: 0.4 W/cm²; Temperature: 60 °C; Standing-wave field; Irradiation time: 12 min).

water droplets in the crude oil emulsion significantly increased and the number of small water droplets decreased.

The result can also be reported in terms of the water droplet size distribution of non-irradiated crude oil emulsion (analyzed before the start of the experiment) and 12 min irradiated crude oil emulsion (a sample obtained at 12 min after the activation of the acoustic field). Fig. 3 also shows that about 64% of all water droplets in non-irradiated sample have a size in the range of 10 μm to 50 μm. However, after 12 min of activation of the acoustic field, about 58% of all water droplets exhibited a size in the range of 90 μm to 130 μm and only 6% of all water droplets have a size less than 50 μm. This finding indicates that ultrasonic irradiation can help small water droplets coalesce and become bigger, which is favorable to water droplet settling down caused by an external force such as gravity. After the irradiated emulsion settles for a reasonable period, the water content of the emulsion will decrease.

1-2. Flocculation of Asphaltene in the Emulsion

Fig. 4 shows the size of asphaltene flocs in crude oil was significantly reduced after crude oil was irradiated by ultrasound. The average radius of asphaltene particles in the sample was 4.2 μm. However, for three-minute irradiated sample and for the six-min-

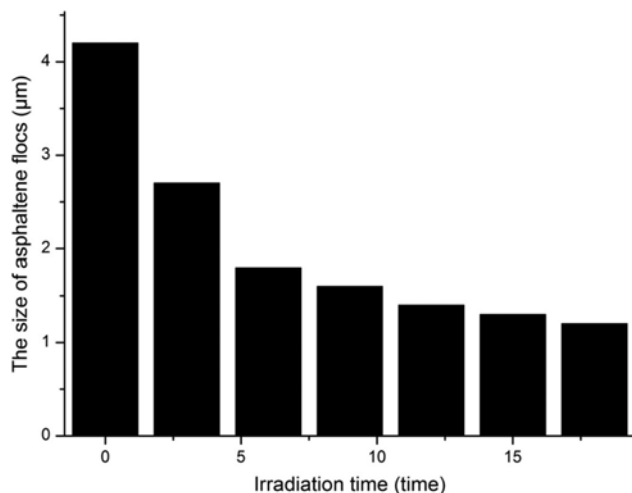


Fig. 4. Effect of ultrasound on the size of asphaltene flocs (Frequency: 20 kHz; Acoustic intensity: 0.4 W/cm²; Temperature: 60 °C; Standing-wave field).

ute irradiated sample, the average radius of the flocs decreased from 2.7 μm and even 1.8 μm. Ultrasonic irradiation can help reduce the average radius of flocs and prevent the formation of large particles. This finding indicates that ultrasonic technology can reduce asphaltene flocculation rates because ultrasonication can change the kinetics of asphaltene aggregation and remove deposits.

Colloidally dispersed asphaltene accumulating at the oil/water interface has surface-active and structure-forming properties, so reduction of the size of asphaltene reduces the interfacial film toughness. Moreover, ultrasonic irradiation can reduce the emulsion stabilization and break the emulsion, which is beneficial to the coalescence of water drops in water-oil (W/O) emulsion and dehydration of crude oil emulsion.

1-3. Viscosity of the W/O Emulsion

Fig. 5 shows that ultrasound can effectively change the viscosities of the W/O emulsion samples. After the W/O emulsion was

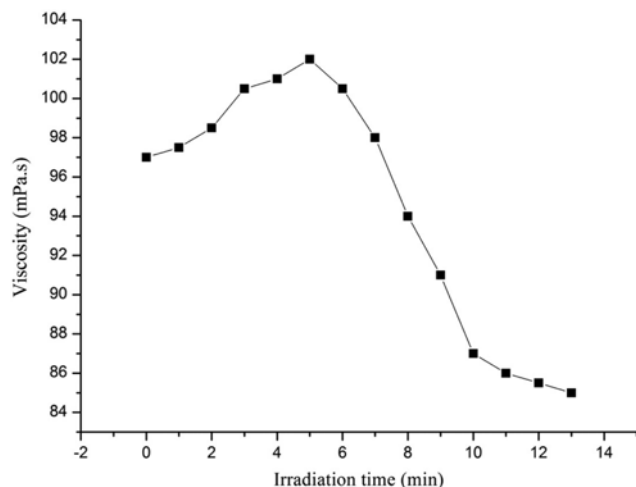


Fig. 5. Effect of ultrasound on the viscosity of W/O emulsion (Frequency: 20 kHz; Acoustic intensity: 0.4 W/cm²; Temperature: 60 °C; Standing-wave field).

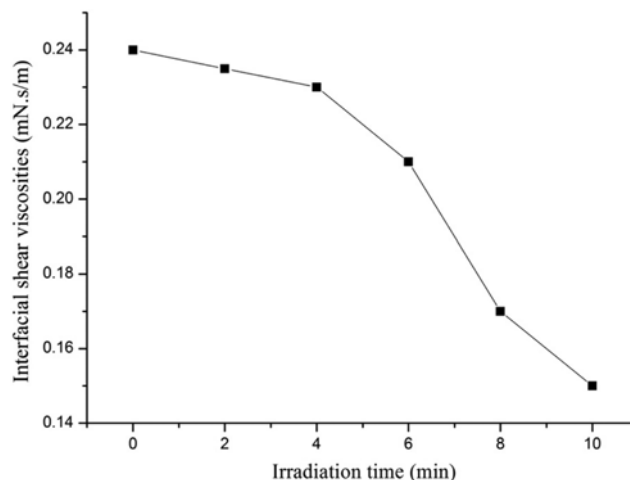


Fig. 6. Effect of ultrasound on shear strength of oil-water interfacial films (Frequency: 20 kHz Acoustic intensity: 0.4 W/cm²; Temperature: 60 °C; Standing-wave field; shear rate: 0.2 rad/s).

irradiated by ultrasound, the viscosity of samples was relatively increased within the first minute because the crude oil consists of heavy components, such as asphaltene and wax, which exist in the suspension. After being irradiated by ultrasound, the heavy components dissolved in the crude oil, thereby increasing the viscosity of W/O emulsion. However, the viscosity of crude oil decreased after 5 min of ultrasonic irradiation because of the breakdown of asphaltene molecules to lighter molecules, as shown in Fig. 6. Although remaining asphaltene and wax still dissolved into the crude oil after 5 min, the breakdown of asphaltene molecules to lighter molecules dominated the decrease of the viscosity of crude oil.

Therefore, ultrasonic technology obviously reduced the viscosity of crude oil after a reasonable time, which reduced the hydrodynamic drag force and helped water drops to collide and coalesce.

1-4. Shear Strength of Oil-water Interfacial Films

Fig. 6 shows that decrease in interfacial shear viscosities can be achieved after the ultrasound irradiation, which means a decrease in shear strength of oil-water interfacial films. The ultrasonic technology can reduce asphaltene flocculation rates and help dissolve asphaltene and wax into the oil as mentioned earlier, thereby decreasing the shear strength of oil-water interfacial films. The surface-active constituents, such as asphaltene and wax, stabilize water-in-oil emulsions only when they are collected at the interface in the form of finely divided solid particles or aggregates [17,18]. They formed hard barriers at the O/W interface, which held back small water droplets from coalescing. In addition, asphaltene and wax molecules slightly affected the stabilization of emulsion when dissolved in the oil phase in the form of a molecular solution [19]. Therefore, ultrasound irradiation can decrease stability of water-in-oil emulsions and help coalescence of the water droplets.

2. Effect of Ultrasonic Parameters on Dehydration of the W/O Emulsion

2-1. Type of Ultrasonic Field

Figs. 7 and 8 demonstrate the sound intensity distribution (20 kHz) of the rectangular chamber in the acoustic standing-wave field and acoustic irregular field, respectively. The comparison of the

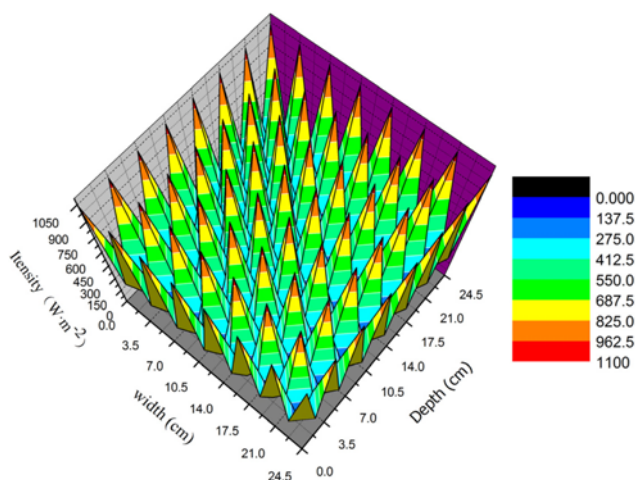


Fig. 7. Sound intensity distribution of 20 kHz acoustic standing-wave field in the rectangular chamber.

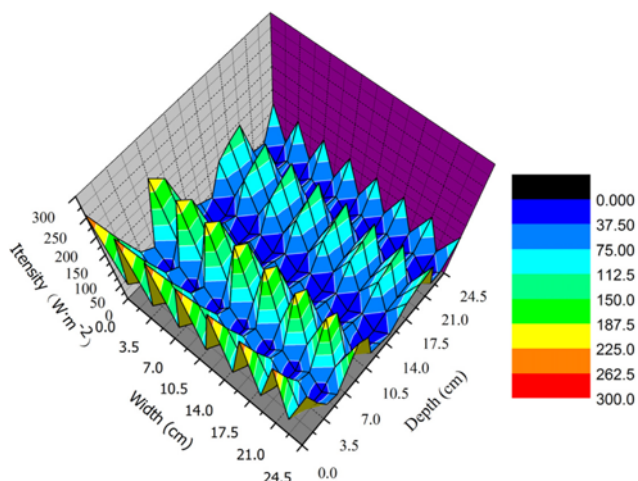


Fig. 8. Sound intensity distribution of 20 kHz acoustic incident wave field in the rectangular chamber.

sound intensity distribution of Fig. 7 with Fig. 8 showed that sound intensity at the antinodes of the standing-wave field may be approximately four-times that of the incident wave field.

Fig. 9 shows the effect of different ultrasonic fields on the average radius of water droplet in the emulsion. The water droplet coalescence effect of the standing-wave field is superior to that of the incident wave field. The incident wave field takes more time to achieve the best coalescence effect than the standing-wave field, which is attributed to the fact that sound intensity at the antinodes of the standing-wave field may be much larger than that of the incident wave field. This condition results in a larger acoustic force on the water drops in the standing-wave field, thereby accelerating the movement of water drops to the pressure antinodes or nodes.

Fig. 10 shows the effect of the type of ultrasonic field on water content of emulsion. It proves the results of Fig. 9 from another aspect. After the W/O emulsion was irradiated by standing-wave ultrasound at 20 kHz for 12 min, water content in crude oil emulsion could be decreased from 40% to 3.8%, which satisfies the re-

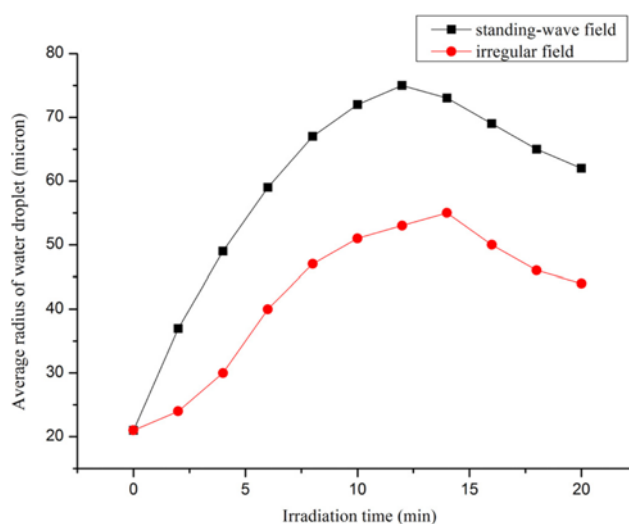


Fig. 9. Effect of the type of ultrasonic field on the average radius of water droplet in the emulsion (Frequency: 20 kHz; Acoustic intensity: 0.4 W/cm^2 ; Temperature: 60°C).

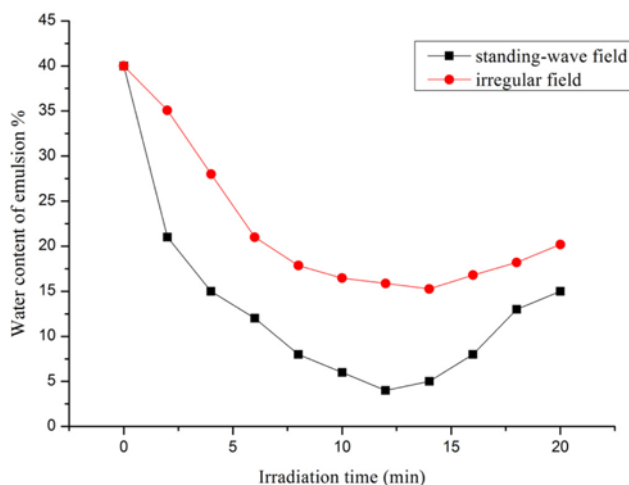


Fig. 10. Effect of the type of ultrasonic field on water content of emulsion (Frequency: 20 kHz; Acoustic intensity: 0.4 W/cm^2 ; Temperature: 60°C).

quirement of dehydration for refinery.

2-2. Ultrasonic Irradiation Time

Fig. 11 shows the average radius of the water droplets in the W/O emulsion increased within the first minute and reached the minimum value at the 12th min. However, at later times, the average radius of the water droplets decreased slightly because the crude oil was emulsified again after long period of ultrasound irradiation.

Moreover, comparison of the evolution of the average radius of water droplets under acoustic fields showed that the average radius increased more quickly at 20 kHz than at 40 kHz within the first minutes, indicating that the rate of coalescence at the lower frequency was significantly higher than that at higher frequency at earlier times. However, as time progressed, the average radius at 20 kHz increased relatively more slowly and the rate of coalescence decreased more rapidly.

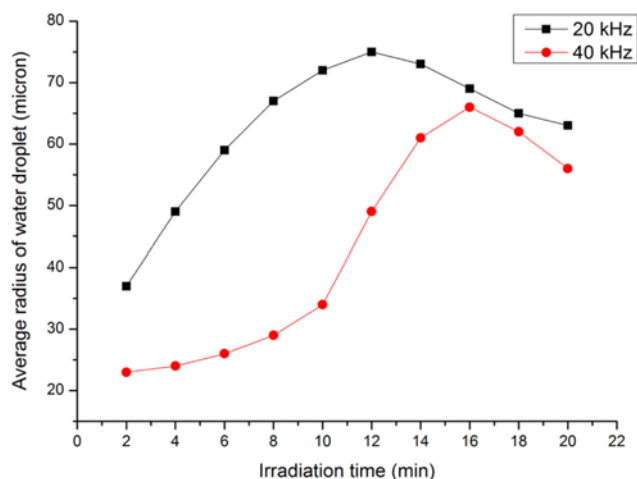


Fig. 11. Effect of ultrasonic irradiation time on the average radius of water droplets (Acoustic intensity: 0.4 W/cm^2 ; Temperature: 60°C ; Standing-wave field).

When the droplets moved close to the pressure antinodes, their coalescence was mainly due to secondary acoustic force. According to Eq. (3), the secondary acoustic force is proportional to the particle volume and the square of the frequency. However, lower frequency corresponds to higher wavelength, which will result in larger rate of coalescence for a given droplet pair at earlier times when the droplets are small.

However, as time progresses and at later times, many large droplets are formed, the secondary acoustic force becomes more important, and a shift in behavior occurs with the overall coalescence rate for larger droplets at lower frequencies being significantly lower than that the rate at higher frequencies.

2-3. Acoustic Intensity

Figs. 12 and 13 show the effect of acoustic intensity on the average radius of water droplets and on water content of emulsion, respectively. Given that water droplets disperse in water and are initially

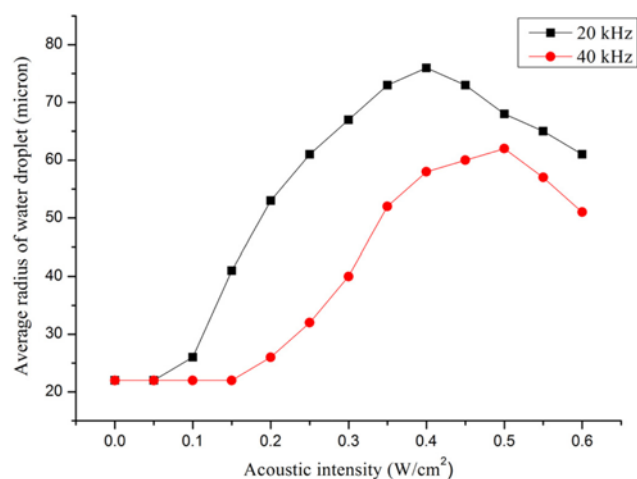


Fig. 12. Effect of acoustic intensity on the average radius of water droplets (Temperature: 60°C ; Irradiation time: 12 min; Standing-wave field).

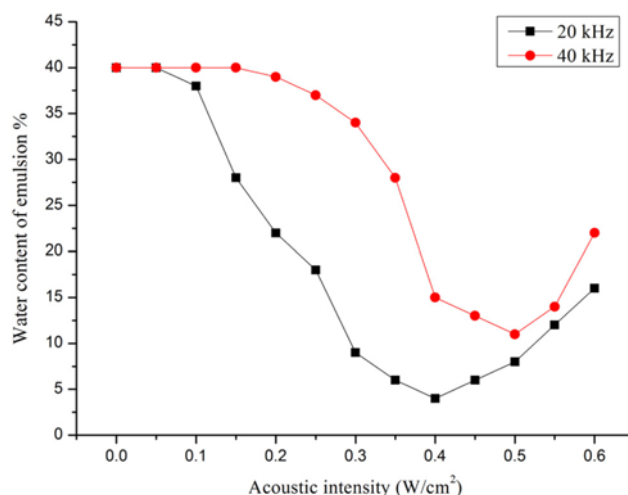


Fig. 13. Effect of acoustic intensity on water content of emulsion (Temperature: 60°C ; Irradiation time: 12 min; Standing-wave field).

in resting condition, they will be driven toward the pressure antinodes of the acoustic field by the resultant forces (gravity, buoyancy, acoustic force, and hydrodynamic drag force). Therefore, a minimal acoustic intensity is needed to drive droplets to move. Fig. 13 shows that the water content of the emulsion (irradiated by ultrasound of 20 kHz) starts to decrease at an acoustic intensity of approximately 0.05 W/cm^2 . However, the water content of the emulsion (irradiated by ultrasound of 40 kHz) starts to decrease at an acoustic intensity of approximately 0.15 W/cm^2 , indicating that the needed minimal acoustic intensity is comparatively higher for 40 kHz. This result is attributed to the fact that the primary acoustic force of 40 kHz at the same driving voltage is smaller according to Eq. (1).

Fig. 13 also demonstrates that acoustic intensity has an optimum value (cavitation threshold) to obtain the most effective dehydration after the emulsion is irradiated by ultrasound for 12 min, and then allowed to settle down for 10 min. This result indicates that cavitation was induced after the acoustic intensity exceeded the optimum value. When the intensity of the sound is increased, such that its energy is greater than that associated with the attractive forces between the liquid molecules, cavitation is observed. In the W/O system, the emulsification is initiated when the cavitation threshold is attained. Comparison of the cavitation threshold of 20 kHz with that of 40 kHz shows that the higher the frequency used, the higher the acoustic intensity one can apply without inducing the onset of cavitation.

CONCLUSIONS

We explored the use of ultrasound to help water drops to coalesce and recover oil from W/O emulsions. A series of experimental studies were conducted using acoustic standing waves in resonant cavities by adjusting the distance between the transducer surface and the reflector. This distance must be kept constant at an odd number of the acoustic half wavelengths: $h=n\lambda/2$. The experimental results showed that water droplet sizes in the crude oil significantly increased and the number of small water droplets decreased

after the W/O emulsion was irradiated by ultrasound. Moreover, ultrasound can effectively decrease the viscosity and shear strength of oil-water interfacial films and minify the size of asphaltene flocs in crude oil, which will be beneficial to coalescence of the water droplets in W/O emulsion in oil separation with ultrasound. Ultrasound irradiation can decrease the emulsion stability. The dehydration effect of the standing-wave field is superior to that of the incident wave field. In addition, the dehydration effect of 20 kHz is superior to that of 40 kHz. Acoustic intensity had an optimum value (0.4 W/cm^2) for obtaining the most effective dehydration. Ultrasonic irradiation time also had an optimum value (12 min) for obtaining the most effective dehydration. Cavitation was induced after ultrasonic irradiation time exceeded 12 min. Under the optimum conditions (frequency: 20 kHz; acoustic intensity: 0.4 W/cm^2 ; temperature: 60°C ; standing-wave field; ultrasonic irradiation time: 12 min), water content in crude oil emulsion can be decreased from 40% to 3.8%, which satisfies the requirement of dehydration for a refinery.

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