



# Ultrasound-assisted desalination of crude oil: The influence of mixing extent, crude oil species, chemical demulsifier and operation variables

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## ABSTRACT

Coalescence of water droplets in crude oil has been effectively promoted by chemical demulsifiers integrated with ultrasound. Temporary images of water droplets in W/O emulsions were directly monitored using a metallurgical microscope. Water droplets achieved expansion of 118% at 40 min ultrasonic irradiation time under well mixing conditions. However, water droplets in heavy crude oil undergo less aggregation than those in light crude oil, due to resistance of mobility in highly viscous fluid. In the absence of chemical demulsifiers, water droplets enveloped by native surfactants appeared to aggregate arduously because of occurrence of interfacial tension gradients. Influential significance analyses have been executed by a factorial design method on operation variables, including acoustic power intensity, operation temperature, ultrasonic irradiation time and chemical demulsifier dosages. In this work, the outcomes indicate that the optimal operating conditions for desalination of crude oil assisted by ultrasound were as follows: acoustic power intensity = 300 W, operation temperature = 90°C, ultrasonic irradiation time = 75 min and chemical demulsifier dosages = 54 mg/L. Besides, it was found that the most influential importance of operation parameter was temperature, followed with acoustic power intensity, ultrasonic irradiation time and chemical demulsifier dosages.

## 1. Introduction

Desalination of crude oil is an important process for refineries, due to sodium/potassium chlorides contained would cause severe corrosion for sequential distillation columns and fouling on heat exchangers. Commercially, the desalting process involves injection of 5–15 vol% of wash water into crude oil, mixing violently to form a W/O emulsion and then undergoing electrostatic demulsification. Under the electrostatic fields the tiny dispersed water droplets polarize, coalesce and settle, leading to demulsification of W/O emulsion [1–3]. It has been recognized that asphaltenes and resins in crude oil would tend to accumulate at the interface of W/O emulsions, resulting in a rigid film which prevents water droplets from aggregation [4–6]. Chemical demulsifiers are usually added into the crude oil for replacement of asphaltenes and resins in the rigid film to accelerate coalescence of water droplets. Nonetheless, most demulsifiers are synthesized chemical compounds, which are not recyclable and partially enter into wastewater effluents [7–9].

In the last few decades, ultrasonic field has been extensively applied for desalination of crude oil [10–19]. The ultrasonic standing waves

could push the water droplets in the crude oil to the wave nodes or antinodes and bring about coalescence, due to the density difference between water and crude oil. Thus, the settling time for separation of water droplets and crude oil would be significantly reduced. Khajehe-samedini et al. claims that an increase of acoustic power intensity shortens the time required for migration of water droplets to bonding zone. Therefore, the desalting rate would increase with an increase of acoustic power intensity [13]. Antes et al. executed crude oil demulsification at low frequency ultrasonic baths (25–45 KHz), wherein microjets and turbulence were presumed to be responsible for breaking emulsion and allowing coalescence of water droplets [15]. Initial water content exhibited positive effects on removal of water droplets, resulting from a greater number of water droplets dispersed in crude oil, which facilitate flocculation and coalescence [10]. Several publications reported that desalting efficiency was enhanced with an increase of ultrasonic irradiation time. Besides, chemical demulsifiers coupled with ultrasonic irradiation offered better desalting performance than those of single operation alone [11,19]. The interfacial viscosity of water–oil would decrease upon increasing operation temperature, wherein the film surrounding water droplets was thinning. Consequently, the rate of

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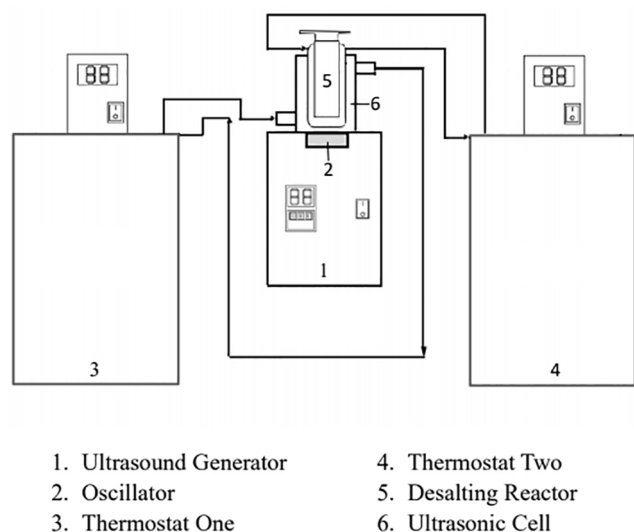
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**Fig. 1.** Experimental system composed of main apparatus for ultrasound-assisted desalination of crude oil.

**Table 1**  
Characterizations of the crude oils.

Crude oil	AL (Arabian Light)	KC (Kuwait)
Density API, 15.6°C	32.3	30.5
Kinematic viscosity, 50°C(cSt)	4.76	7.86
Carbon residue (wt%)	4.23	6.52
Pour point (°C)	< -30	< -30
Total nitrogen (ppm)	1124.0	1567.0
Sulfur by D-4294 (wt%)	2.109	2.810
Salt & Water (vol%)	0.05	0.05
Salt (chloride ions, mg/L)	30.0	90.0

coalescence assisted by ultrasound would be intensified [17,18].

The influences of operating parameters in an ultrasound-assisted desalting process on the behaviors of desalination have been broadly investigated, including acoustic power intensity, temperature, ultrasonic irradiation time, wash water added and chemical demulsifier dosages. Nonetheless, little research has focused on the influential importance of above operation variables on the desalting performance, which is essential for the industrial process design. Therefore, this study is aimed at elucidation of operating variable significances by the ANOVA analyses using the Design-Expert software. The water droplets size expansion caused by ultrasound would be examined in situ. Moreover, the effects of greater water droplet numbers arisen from violent agitation and crude oil species on the desalting efficiency would be explored simultaneously.

## 2. Experimental methods

### 2.1. Ultrasound-assisted desalination test of crude oil

Fig. 1 illustrates the scheme of the main apparatus in experiments. Ultrasonic irradiation was continuously emitted by an ultrasonic oscillator at a constant frequency of 160 kHz (300 W with adjustable power output), controlled via a mosfet circuit located under the bottom of the ultrasonic cell (PIIN JIA Technol.). The desalting reactor was a double jacket cylinder made of Pyrex glass. Prior to tests, proportionate volumes of deionized water (10 mL) were mixed vigorously with crude oil (100 mL) for one hour at the designed temperature to make W/O emulsion in the reactor. All experiments were carried out batch-wisely at atmospheric pressure among the temperature ranges of 45°C–90°C, wherein the temperature of W/O emulsion was maintained at the set point using a circulating water bath (VWR Scientific Co.). Within the duration of tests, samples (1 mL) were withdrawn from the reactor at constant time intervals (20 min) for examination of water droplet diameters using a metallurgical microscope. In another group of experiments with different ultrasonic irradiation times (30 min to 75 min), the water layer separated from W/O emulsions underwent ion chromatograph analyses for evaluation of chloride ion concentrations and aqueous solution volume measurement.

To make clear the influence of acoustic power intensity on the desalting behavior of crude oil, four tests with a variety of power intensity (180 W to 300 W) were carried out. For the purpose of evaluating the effect of chemical demulsifier dosages on the desalting performance, a series of experiments with various chemical demulsifier (CPC DS-1) concentrations of 27 mg/L to 54 mg/L were conducted. Moreover, experiments for W/O emulsions formed at ordinary agitation speed (400 rpm) or violent agitation speed (1000 rpm) were executed to investigate the influence of water–oil mixing situations on desalting efficiency. In another respect, desalting tests related to diverse species of crude oils were performed for assessment of crude oil viscosities on desalting efficiency. In this research, all experimental tests were executed at least in duplicate to make the data reliable.

### 2.2. Reagents

The crude oil tested was supplied by CPC Corporation (shown in Table 1). The reagents of  $\text{Na}_2\text{CO}_3$  ( $\geq 99.9\%$ , Merck),  $\text{NaHCO}_3$  ( $\geq 99.7\%$ , Merck) and NaCl standard solution ( $\geq 99.99\%$ , Merck) were used for ion chromatograph analysis. Purified water from a Milli-Q system (Milipore, Billerica, USA) was employed for preparation of water–oil emulsions. The chemical demulsifier (CPC DS-1) was provided from CPC Corporation (Taiwan).

### 2.3. Ion chromatograph (IC) analysis

Desalting efficiency of crude oil is calculated on the basis of removal of chloride ions, due to most components involved in water droplets to be chloride [20,21]. With regard to desalination tests of crude oil, the

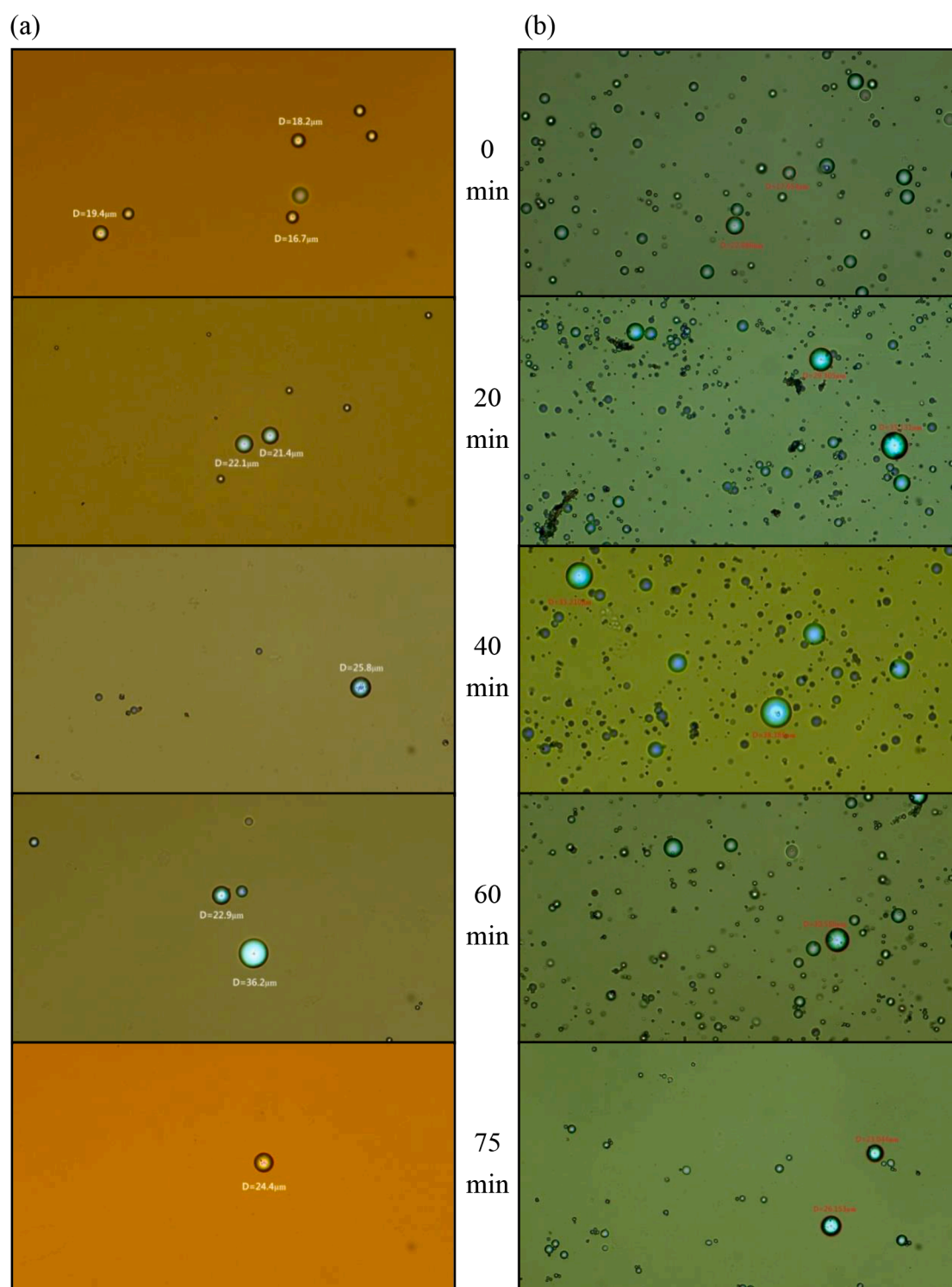
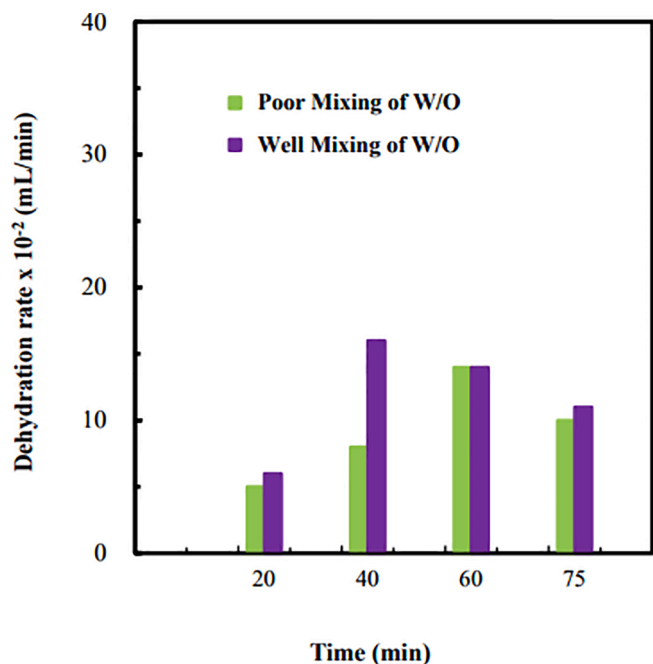


Fig. 2. (a) The time dependent shapes and diameters of water droplets formed at poor mixing, (b) The time dependent shapes and diameters of water droplets formed at well mixing (W/O = 10/100) under the conditions of acoustic power intensity = 260 W, T = 75°C, chemical demulsifier concentration = 54 mg/L.

**Table 2**

Diameter of water droplets observed under ultrasonic irradiation with poor water/crude oil mixing beforehand. (tested on AL crude oil, W/O agitated with 400 rpm)

Ultrasonic irradiation time (min)	Water droplet diameter ( $\mu\text{m}$ )
0	16.7, 18.2, 19.4
20	21.4, 22.1
40	25.8
60	36.2
75	24.4

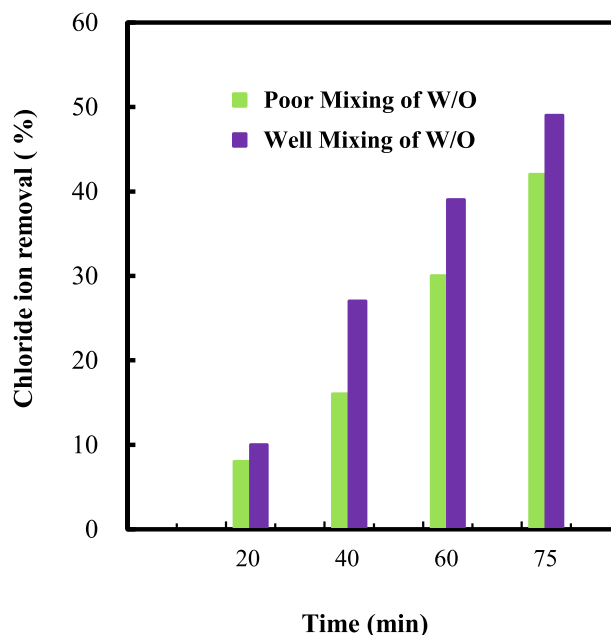


**Fig. 3.** The time dependent dehydration rate of emulsions (W/O = 10/100) at poor mixing or well mixing under the conditions of acoustic power intensity = 260 W, T = 75°C, chemical demulsifier concentration = 54 mg/L.

**Table 3**

Diameter of water droplets observed under ultrasonic irradiation with well water/crude oil mixing beforehand. (tested on AL crude oil, W/O agitated with 1000 rpm)

Ultrasonic irradiation time (min)	Water droplet diameter ( $\mu\text{m}$ )
0	17.6
20	29.3
40	38.4
60	30.3
75	26.1

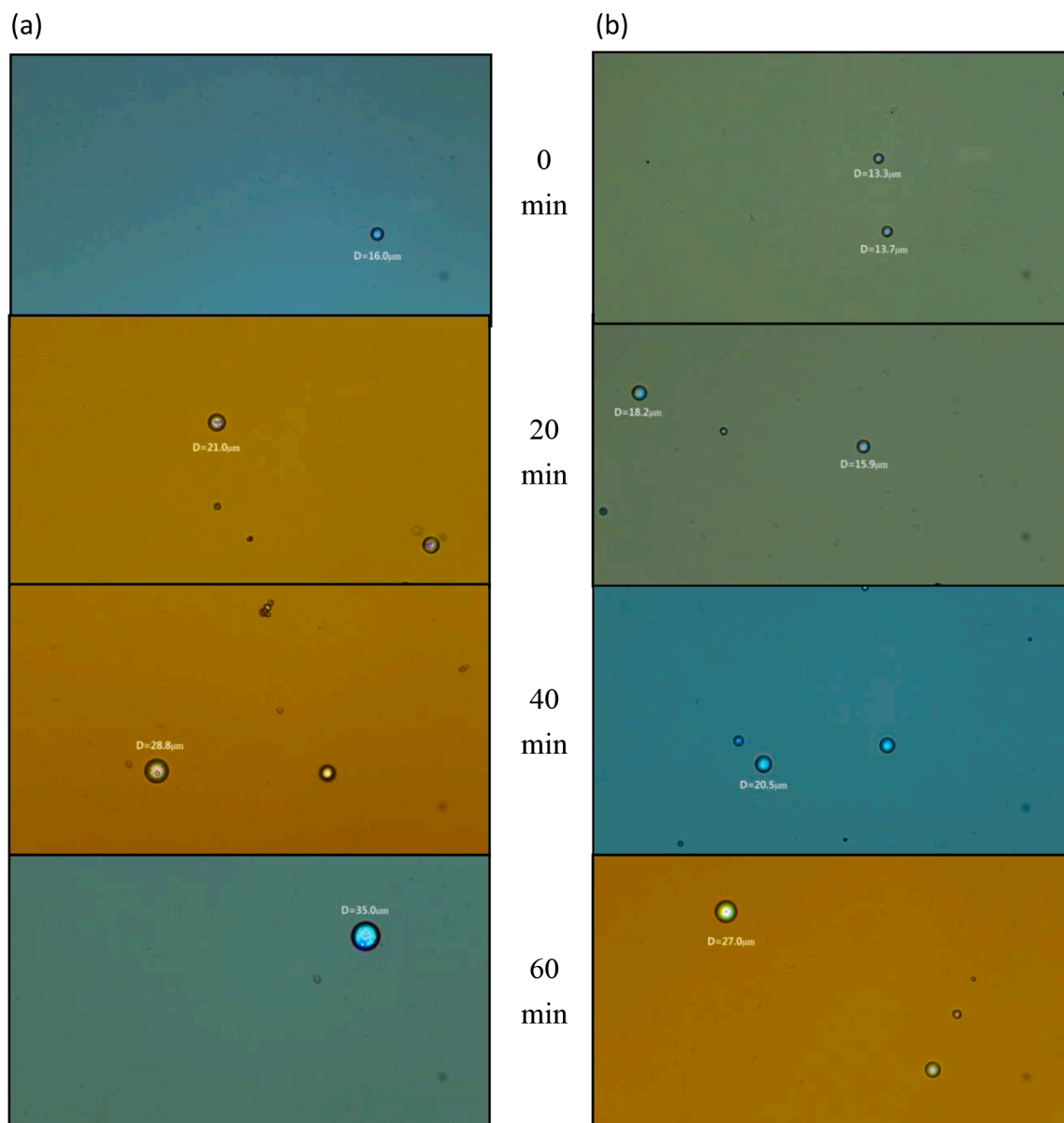


**Fig. 4.** The time dependent desalting efficiency of crude oil at poor mixing or well mixing (W/O = 10/100) under the conditions of acoustic power intensity = 260 W, T = 75°C, chemical demulsifier concentration = 54 mg/L.

water layer obtained was directly analyzed for chloride ion concentrations utilizing an ion chromatograph (IC) instrument (Metrohm, 883 Compact IC plus) equipped with an isocratic pump and a dialysis system. The aqueous solutions of  $\text{Na}_2\text{CO}_3$  (3.2 mM) and  $\text{NaHCO}_3$  (1.0 mM) served as the eluent, which had been degassed by ultrasound for 30 min. The sample injection volume was 20  $\mu\text{L}$  and a column packed with polymeric substrates functionalized by quaternary ammonium groups (Metrosep A Supp 5, 4 mm  $\times$  250 mm) was used. The data acquired from the IC analyzer was corrected to the calibration curve, which had been cautiously established at the range of 0 mg/L to 500 mg/L by the sodium chloride standard solutions.

#### 2.4. Metallurgical microscope analysis

In the course of experiments, samples were picked from the desalting reactor periodically for determination of instant water droplet diameters by means of a metallurgical microscope (Olympus, BX60). The maximum diameter of water droplets observed would be indicated and labeled for comparison within the experiments. As far as two crude oil species are concerned, both their water droplet diameters would be examined under identical operating conditions. Additionally, the water droplet diameters would be monitored to distinguish between gentle mixing and severe mixing of water/crude-oil emulsions. During the testing with or without dosages of chemical demulsifier, the water



**Fig. 5.** (a) The time dependent shapes and diameters of water droplets in emulsions for AL crude oil (b) The time dependent shapes and diameters of water droplets in emulsions (W/O = 10/100) for KC crude oil under the conditions of acoustic power intensity = 260 W, T = 75°C, chemical demulsifier concentration = 54 mg/L.

**Table 4**

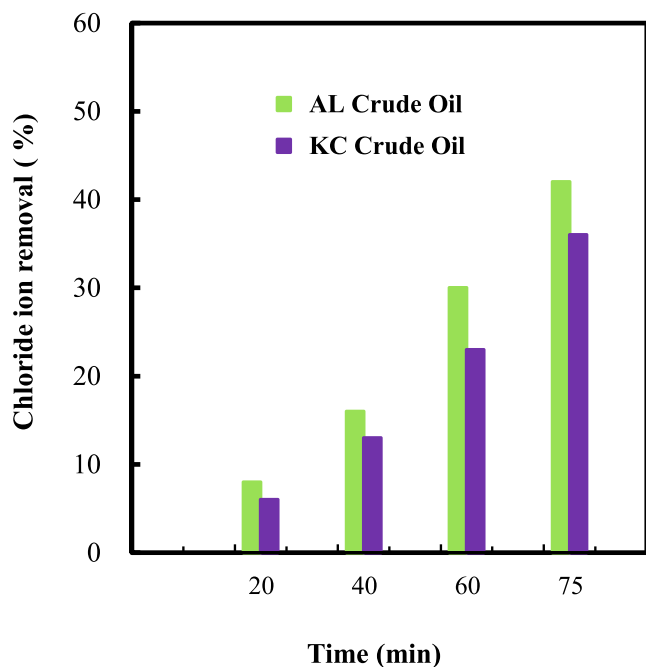
Diameter of water droplets observed under ultrasonic irradiation for different species of crude oils.

Ultrasonic irradiation time (min)	AL, Water droplet diameter ( $\mu\text{m}$ )	KC, Water droplet diameter ( $\mu\text{m}$ )
0	16.0	13.3, 13.7
20	21.0	15.9, 18.2
40	28.8	20.5
60	35.0	27.0

droplet diameters would be recorded for comparison as well.

#### 2.5. Influential importance analysis of operation variables

Experimental data was analyzed statistically using a factorial design method, wherein the operation parameters including acoustic power intensity, operation temperature, ultrasonic irradiation time and chemical demulsifier dosages were taken into consideration. The statistical ANOVA analyses were performed by means of a Design-Expert package, in which the primary criterion for discrimination of statistically significant operation variables was Model P-value < 0.0005.



**Fig. 6.** The time dependent desalting efficiency of crude oil for AL or KC crude oils under the conditions of acoustic power intensity = 260 W, T = 75°C, chemical demulsifier concentration = 54 mg/L.

Further, the high level of Model F-value indicates operation variables to be more significant.

### 3. Results and discussion

#### 3.1. Effect of mixing extent on ultrasound-assisted desalination of crude oil

It has been well recognized that acoustic waves would drive water droplets in the crude oil to the wave nodes or antinodes on account of density difference, leading to coalescence of water droplets [22]. Fig. 2 (a) illustrates water droplets observed at constant time intervals in W/O emulsions, descended from poor mixing, irradiated by ultrasound. The time dependent water droplet diameters are summarized in Table 2. It clearly indicates that water droplets coalesce and their diameters expand about 14.0% to 32.0% within 20 min, consistent with the literatures [23,24]. After ultrasonic irradiation time of 60 min, tiny water droplets coalesced would reach a maximum expansion of 87%. Nonetheless, water droplet diameters would reduce from 36.2  $\mu\text{m}$  to 24.4  $\mu\text{m}$  for further ultrasonic irradiation (75 min). This phenomenon may be interpreted with settlement of larger water droplets ( $\geq 40 \mu\text{m}$ ), resulting in small water droplets residual. It could be essentially supported with time dependent dehydration rate of W/O emulsions (shown in Fig. 3), wherein the dehydration rate achieved a maximum speed at the

operation time of 60 min.

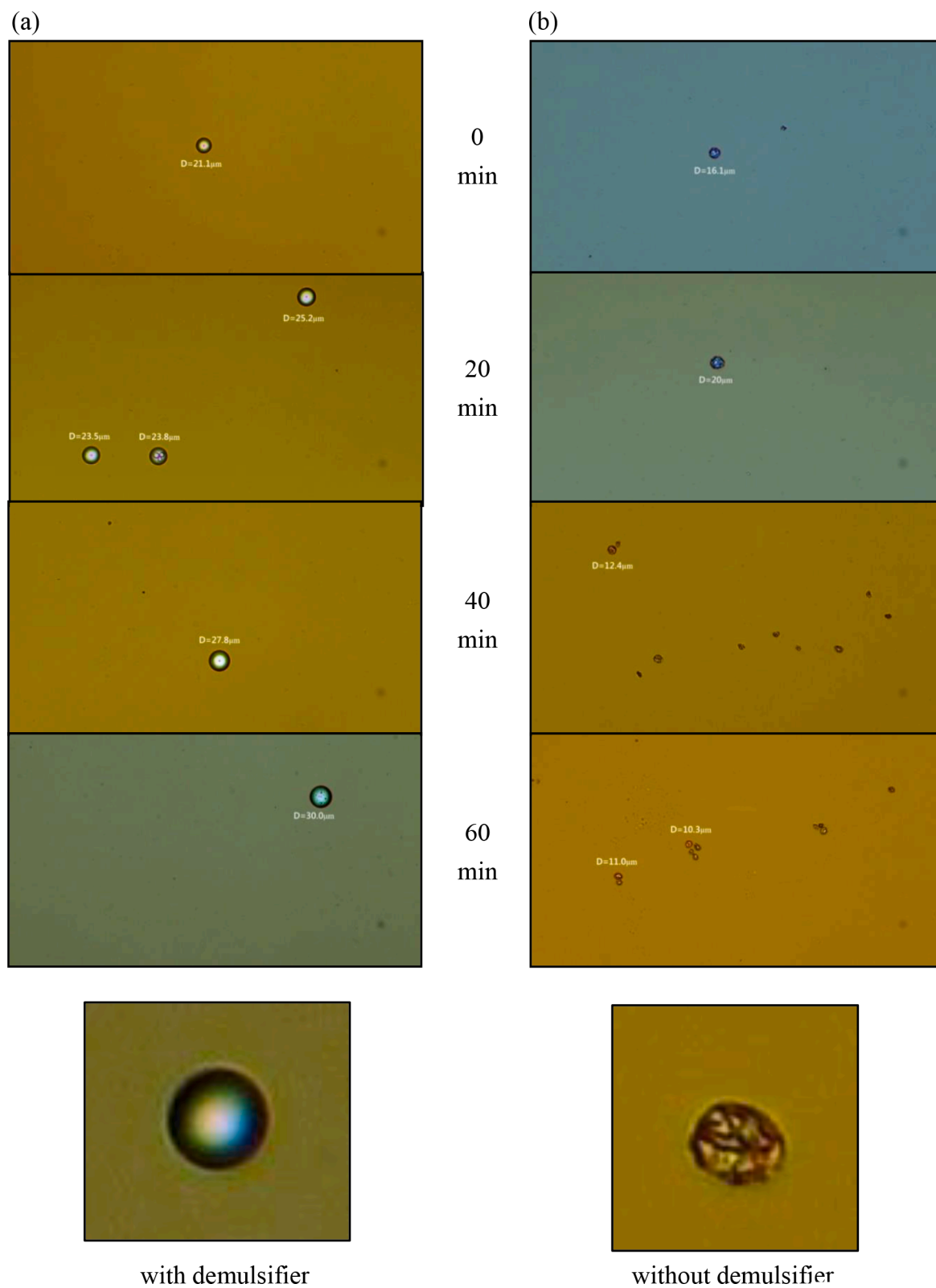
In contrast, Fig. 2(b) demonstrates water droplets observed periodically in W/O emulsions under well mixing conditions and ultrasonic irradiation. The time dependent water droplet diameters are summarized in Table 3. Apparently, water droplet numbers increased significantly under well mixing, caused by violent agitation. It brings about coalescence of water droplets to be accelerated. The larger water droplet occurred at the operation time of 40 min with a maximum expansion of 118%, whereas under poor mixing situations maximum diameters appeared until an operation period of 60 min. With reference to time dependent dehydration rate of W/O emulsions (shown in Fig. 3), it also achieved maximum speed at the operation time of 40 min. It reveals that well mixing is favorable to dehydration of crude oil. According to the results (Fig. 2), it indicates that water droplets coalesce gradually and reach to maximum diameter of 40  $\mu\text{m}$  approximately. Then, the large diameter water droplets would settle to the bottom of the reactor. That is, coalescence of water droplets irradiated by ultrasound depends on both water droplet diameters and amounts. Further, desalting efficiency of crude oil under either poor mixing or well mixing conditions was determined upon chloride ion concentrations of water layer analyzed (refer to Fig. 4). Obviously, well mixing conditions are beneficial to desalination of crude oil as well on account of existence of more water droplets and magnification of acoustic effects. Besides, the sharp expansion on water droplet is thought to facilitate the sequential electrostatic demulsification [25].

#### 3.2. Effect of crude oil species on ultrasound-assisted desalination of crude oil

From the viewpoint of process design, it is essential to elucidate the influences of crude oil species on desalting performance assisted by ultrasound. Fig. 5 illustrates water droplets observed periodically in W/O emulsions irradiated by ultrasound. The time dependent water droplet diameters are summarized in Table 4. The original water droplet diameters for AL and KC crude oils observed were at the range of 13.3 to 16.0  $\mu\text{m}$ , corresponding to reports by Bresciani et al. [26]. After 60 min treatment of ultrasonic irradiation, water droplets coalesced apparently for both AL and KC crude oils, wherein the coalescing rate of the former (35  $\mu\text{m}$ ) is higher than that of the latter (27  $\mu\text{m}$ ). It may be ascribed to resistant mobility of water droplets by the viscous drag force in heavy crude oil (KC), which depends on the ratio of viscosity of water to crude oil and the surface active species on water droplets [27–29]. Accordingly, all of water droplets in AL crude oil are larger than those in KC crude oil at the same time. Fig. 6 presents the time dependent desalting efficiency in W/O emulsions for both AL and KC crude oils. Transparently, desalting rate for AL crude oil is higher than that of KC crude oil. The outcomes convince us that desalination of crude oil assisted by ultrasound would be more suitable for crude oils with low viscosity.

#### 3.3. Effect of chemical demulsifier on ultrasound-assisted desalination of crude oil

It has been well understood that chemical demulsifiers could

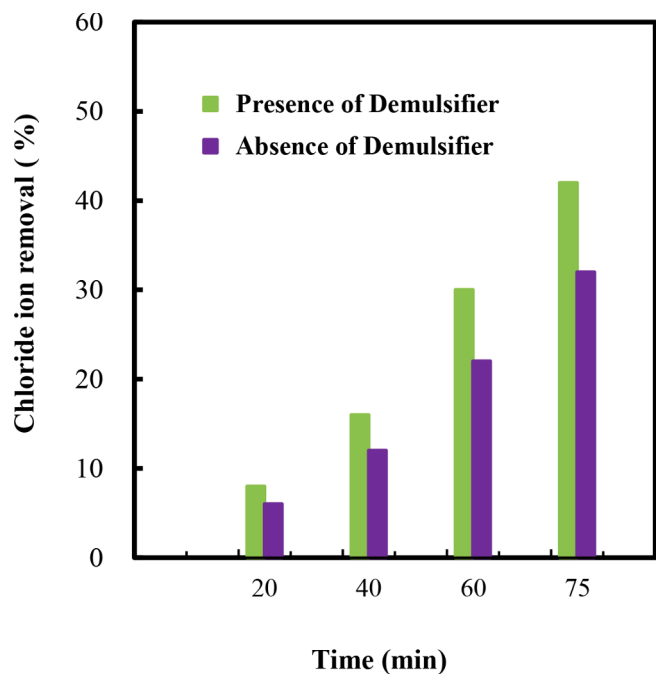


**Fig. 7.** (a) The time dependent shapes and diameters of water droplets in emulsions in the presence chemical demulsifiers (b) The time dependent shapes and diameters of water droplets in emulsions (W/O = 10/100) in the absence of chemical demulsifiers under the conditions of acoustic power intensity = 260 W, T = 75°C, chemical demulsifier concentration = 54 mg/L.

**Table 5**

Diameter of water droplets observed under ultrasonic irradiation in the presence or absence of chemical demulsifier.

Ultrasonic irradiation time (min)	Water droplet diameter with demulsifier ( $\mu\text{m}$ )	Water droplet diameter without demulsifier ( $\mu\text{m}$ )
0	21.1	16.1
20	23.5, 23.8, 25.2	20.0
40	27.8	12.4
60	30.0	10.3, 11.0



**Fig. 8.** The time dependent desalting efficiency of crude oil in the presence or absence of chemical demulsifiers under the conditions of acoustic power intensity = 260 W, T = 75°C, chemical demulsifier concentration = 54 mg/L.

effectively enhance desalting efficiency of crude oil. Effects of chemical demulsifier dosages on the desalting performance assisted by ultrasound are demonstrated in Fig. 7. The time dependent water droplet diameters are summarized in Table 5. It is evident that in the absence of chemical demulsifiers water droplets could not coalesce successfully even with assistance of ultrasound. This phenomenon could be attributed to accumulation of asphaltenes and resins at the interface of W/O and inhibition of coalescence of water droplets [30,31]. On the contrary, asphaltenes and resins were replaced with chemical demulsifiers, leading to intense coalescence of water droplets with the aid of ultrasound [32]. As expected, desalting efficiency of crude oil in the existence of demulsifiers would be superior to that with lack of demulsifiers (refer to Fig. 8). As far as the water droplet shape is concerned, it is likely in the

form of spheres in the presence of chemical demulsifiers. Nonetheless, water droplets look like coffee beans in the short of demulsifiers. The observation could be ascribed to interfacial tension gradient, induced by native surfactants, such as asphaltenes and resins [33,34]. The results imply that chemical demulsifiers coupled with ultrasonic irradiation is a promising pretreatment method for desalination of crude oil.

### 3.4. Test of the influential significance of operation variables on ultrasound-assisted desalination of crude oil

It is essential to investigate the influential importance of operation parameters for the purpose of developing a novel technique. Therefore, a statistically factorial design analysis (ANOVA Model, Design-Expert) was executed in dealing with four factors, including acoustic power intensity, operation temperature, ultrasonic irradiation time and chemical demulsifier dosages using four levels (shown in Fig. 9). Apparently, the optimal operating conditions for desalination of crude oil with avail of ultrasound were as follows: acoustic power intensity = 300 W, operation temperature = 90°C, ultrasonic irradiation time = 75 min and chemical demulsifier dosages = 54 mg/L. Based on experimental data analyzed statistically, it was found that the most influential significance of operation parameter was temperature, followed with acoustic power intensity, ultrasonic irradiation time and chemical demulsifier dosages.

## 4. Conclusions

On the basis of above discussion, it is evident that coalescence of water droplets in crude oil was significantly accelerated via ultrasonic irradiation, leading to enhancement on desalting efficiency. The time dependent images of water droplets indicate that water droplet coalesced achieved a maximum expansion of 118% in emulsions within 40 min under well mixing conditions. Nonetheless, water droplets in heavy crude oil undergo less aggregation than those in light crude oil, wherein desalting efficiency of the former is lower than that of the latter. In the lack of chemical demulsifiers, water droplets surrounded by native surfactants coalesce hard on account of occurrence of interfacial tension gradient. Further, influential significance analyses have been performed statistically on operation variables by a factorial design method. In this work, the results present that the optimal operating conditions for desalination of crude oil assisted by ultrasound were as follows: acoustic power intensity = 300 W, operation temperature = 90°C, ultrasonic irradiation time = 75 min and chemical demulsifier dosages = 54 mg/L. The most influential importance of operation parameter was established to be temperature, followed sequentially with acoustic power intensity, ultrasonic irradiation time and chemical demulsifier dosages.

## 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.



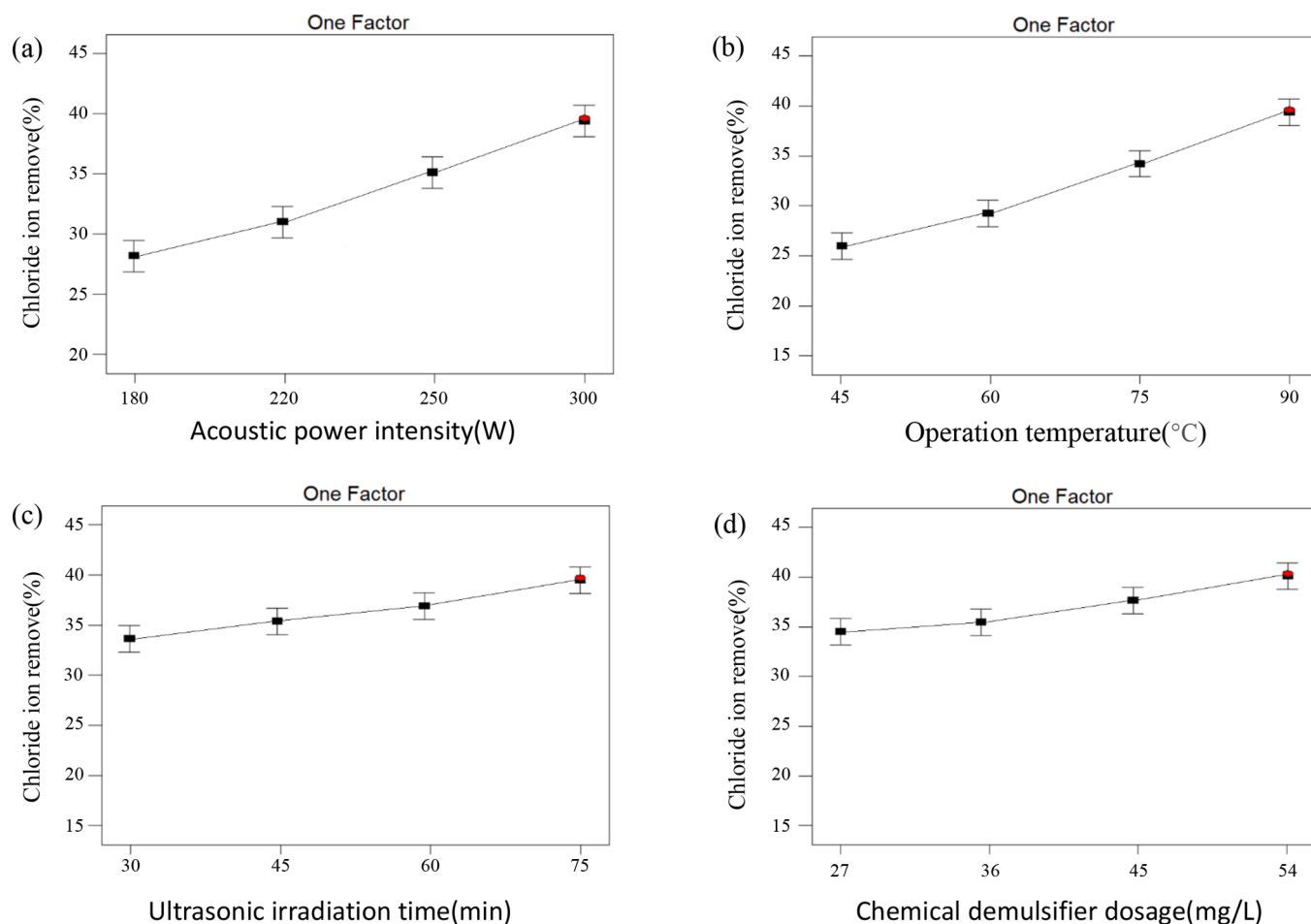


Fig. 9. The statistical ANOVA analyses on operation variables, including (a) acoustic power intensity, (b) operation temperature, (c) ultrasonic irradiation time and (d) chemical demulsifier dosages.

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