

Study on demulsification and deoiling for O/W emulsion by microbubble pretreated resin

Xiaoping Wang, Wei Liu, Xueqian Liu and Jihang Luo

ABSTRACT

The microbubble pretreated resin was used for demulsification and deoiling of the simulated O/W emulsion. The demulsification and deoiling performance and the influencing factors were investigated systematically. Experimental results indicate that the microbubble pretreated resin reaches a 97% oil removal within 80 min; on the contrary, oil removals are 90% and 85% for NaOH solution soaked and un-pretreated resins respectively. After five repeated runs, the oil removal of microbubble pretreated resin can be maintained at over 70%. The demulsification mechanism was revealed by comparing zeta potential, surface tension, contact angle of the emulsion in treatment, and the characterization results of the resin before and after use. Three possible pathways of demulsification were concluded and the ranking contributions can be shown below. Pathway 1: Competitive trapping of surfactant. The cationic groups of the resin combine with the anionic groups of the surfactant and drag them away from the oil particle surface. Pathway 2: Distribution equilibrium of surfactant. Free surfactants in the emulsion are captured by resin and reduce the concentration of uncombined surfactant. This results in surfactants on the oil particle partly detaching from the oil surface to maintain the adsorption-desorption equilibrium of the surfactant. Pathway 3: Adsorption coalescence.

Key words | demulsification, microbubble, O/W emulsion, resin, surfactant

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INTRODUCTION

Oil-containing emulsified waste liquid (Yaghmur *et al.* 2006) is the waste that the oil dispersed stably in other mutually incompatible liquids (generally this is water). Usually, it is stabilized by various surfactants and forms a multiphase dispersion system (Hohl *et al.* 2016). The properties of oil in water are related to its particle size and the oil is classified as dissolved oil if $d < 2 \mu\text{m}$, emulsified oil if $2 \mu\text{m} < d < 10 \mu\text{m}$, dispersed oil if $10 \mu\text{m} < d < 100 \mu\text{m}$, or free oil droplets if $d > 100 \mu\text{m}$ (Lu *et al.* 2016). Demulsification of the oily waste liquid is to make the oil phase destabilize from its emulsified state and then agglomerate to big oil droplets (Li *et al.* 2016). After that, the oil droplets can be easily separated from water to achieve the purpose of deoiling.

Demulsification of emulsified wastewater by using resin has been studied from the pretreatment of resin, demulsification performance, interface properties, and hydrophobic or hydrophilic modification during the past decades (Zhou *et al.* 2009; Yan *et al.* 2010; Xie *et al.* 2012; Li *et al.* 2017). Wang Jun *et al.* (Jun *et al.* 2011) synthesized two phenol-

amine resins by using cardanol as raw materials, and obtained a better deoiling rate than commercial demulsifiers SP169, BP169 and TA1031. In the treatment of oil-in-water (O/W) emulsion, the various kinds of resins are usually used to purify the surfactant stabilized emulsion through the ion exchange effect. Actually, the resin is usually pretreated with strong acid or alkali solution to remove the impurities inside the resin in industrial applications (Kongdee *et al.* 2010). Resin, as a good demulsifier due to its porous and reticular structure, has created a huge ion exchange capacity (Okada *et al.* 2010). In addition, its naturally hydrophobic surface can effectively adsorb oil particles from O/W emulsion (Huang *et al.* 2017).

However, many studies on demulsification of O/W emulsion have been reported while there have been few researches on resin in recent years (Martínez-Palou *et al.* 2013; Zhang *et al.* 2014; Liang *et al.* 2015; Muto *et al.* 2016; Wang *et al.* 2016), since resin demulsifier has met many challenges with the developing of industrial activities if the

emulsion contains complex surfactants or contains no surfactant. In this way, the demulsification of complex emulsion by resin becomes more and more difficult, so the detailed demulsification mechanism must be found. Besides, commercial resin can be divided into gel and macroporous types and usually contains large amounts of impurities occupying the exchange pores or related groups, so it must be pretreated to obtain a good ability before use (Kaya *et al.* 2015). From previous reports, few researches have studied the multiple demulsification mechanisms and alternative regeneration methods of resin systematically, which has limited its application in demulsification of O/W emulsion. In view of this, the systematic demulsification mechanisms and effective pretreatment methods for commercial resin need to be investigated.

In this study, strong alkaline anion exchange resin was pretreated by microbubble and applied to the purification of O/W emulsion. The demulsification mechanism of the resin was summarized systematically and in detail basing on the experiment and characterization results. The demulsification influencing factors and the properties of the emulsion during the reaction were also investigated. In our literature search, pretreatment of resin by microbubble has not been reported yet.

MATERIALS AND METHODS

Chemicals and reagents

Edible peanut oil (Lu hua, China), anion surfactant SDBS (sodium dodecyl benzene sulfonate, AR, Kelong, China), NaOH (AR, Kelong, China), $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ (AR, Kelong, China), NaCl (AR, Kelong, China), strong alkaline anion exchange resin (201×7 (717), average particle size 0.400–0.700 mm, water content 42.00%–48.00%, functional group $\text{RCH}_2\text{N}(\text{CH}_3)_3^+$, Miao yang, China).

Pretreatment of resin

As shown in Figure 1, the microbubble pretreatment reactor was connected to an air pump, and a certain amount of resin and water were added in the reactor. Turn on the air pump and microbubbles generate continuously in water; after a set period, the impurities in the resin detached from its pores and then were dissolved or suspended in water. After that, the pretreated resin was separated from water and washed with distilled water. Importantly, the resin needed to have

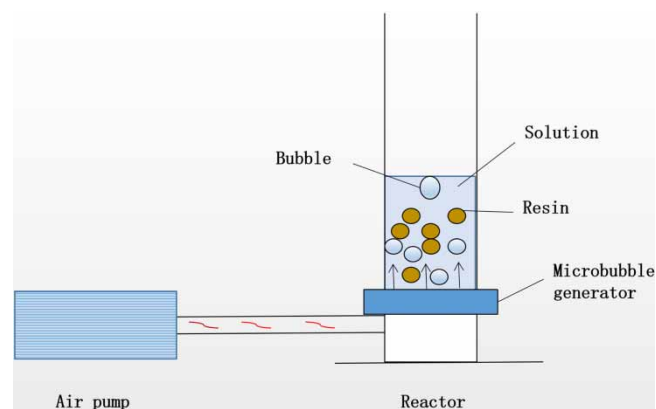


Figure 1 | The microbubble resin pretreatment reactor.

the excess water drained out so that it can be weighed accurately.

Preparation of emulsion, demulsification and deoiling experiment

The artificial surfactant stabilized emulsion used in this paper is prepared basing on a previous study and as shown in the following procedure (Zhang *et al.* 2016): 1 g edible peanut oil, 3 g SDBS and 1 L distilled water were added in a 2 L beaker. After full mixing, stirring for 20 min by an agitator (IKA RW 20 D S25, Germany) and sonication for 5 min by an ultrasonic cleaner (Shumei KQ3200DE, China), and then an emulsion containing 1,000 mg/L oil was obtained. The prepared emulsion was imaged by digital microscope and the size of oil droplets was estimated by nano measurer software.

Demulsification and deoiling experiment: 20 g resin and 50 ml simulated emulsion were placed in a 150 ml conical flask, and then put in a constant temperature culture oscillator (Zhicheng ZWYR-240, China). After shaking for 80 minutes (120 r/min, 30 °C) and settling down for 5–10 minutes at room temperature, the liquid in the middle layer was measured for absorbance at a wavelength of 400 nm. The oil content (C , mg/L) of the treated emulsion and the removal efficiency (R , %) of oil were calculated by Equations (1) and (2).

$$A = 0.000823 \times C - 0.00483 \quad (1)$$

$$R = (C_0 - C_t)/C_0 \times 100\% \quad (2)$$

where, A is the absorbance of the emulsion, C is the oil content of the emulsion (mg/L), C_0 (mg/L) is the initial concentration of the oil (1,000 mg/L) in the solution, C_t (mg/L) is the

concentration of oil after t minutes treatment. The oil particle concentration within 1–1,000 mg/L can be measured accurately, but the results will deviate from this rule gradually if the oil particle concentration is outside 1,000 mg/L.

Analytical approaches

Although the oil content measurement by spectrophotometer is influenced by the particle size distribution, this method can still be used to characterize the oil content in water when the oil particles are uniformly distributed and the particle size is less than 10 μm (Mao & Han 2013; Hanbing *et al.* 2018). In addition, the oil content of the sample to be tested should be in the range of 1–1,000 mg/L. Basing on the microscopic image shown in Figure 2(a) and the initial oil content setting, the oil concentration in this paper can be measured by UV spectrophotometer (Meipuda

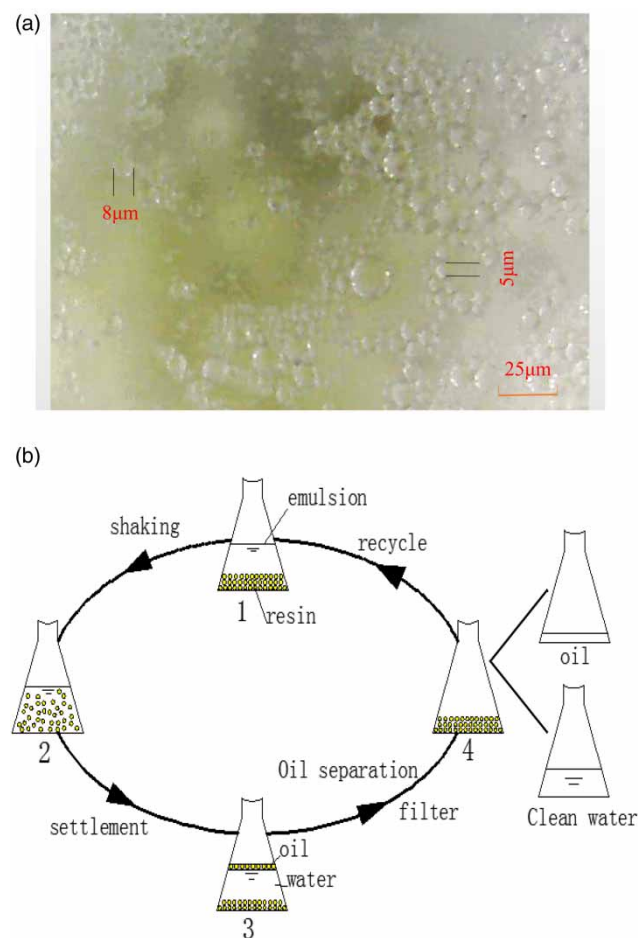


Figure 2 | Oil particles in the artificial emulsion (a) and the schematic diagram of the demulsification operation process (b).

UV-3100PC, China) and calculated according to Equation (1) shown above.

The zeta potential was measured by Zetasizer (Malvern Zetasizer Nano ZS90, UK). The changes in surface groups of resin were characterized by infrared spectrometer (Shimadzu IRPrestige-21, Japan). Briefly, grinding the resin into powder and drying at 60 °C for 2 hour, mixing the dry powder with infrared-quality KBr (1:100) and pressing into disks under a vacuum by a spectra lab pelletiser. The surface tension and contact angle were measured by Contact angle meter (Mingyu MYU-SPCAx3, China). In detail, the surface tension was measured by the hanging drop method, in which the hanging drop state was captured by a camera and then calculated automatically. The contact angle was measured by putting a liquid drop on the glass sheet and the results were obtained from the computer. The volumes of water droplet in the measurement are approximately 5 μL . The appearance of the resin was characterized by optical microscope (Shunyu DMXS, China) and the pH value of the artificial emulsion was measured by pH meter (Leici, PHSJ-4F, China).

RESULTS AND DISCUSSION

The demulsification results

The demulsification performances of resins pretreated by a traditional method and microbubbles were compared and are shown in Figure 3(a). It was evident that the removal rate always kept rising with the increase of resin dosage. Especially, the best demulsification and deoiling effect was obtained by the microbubble pretreated resin, which reached an oil removal of 97%. The 4% NaOH solution pretreated resin also achieved a good removal rate (90%), but the appearance of the resin pretreated by NaOH changed a lot and the eluents usually need further treatment. As to the untreated resin, the removal rate (85%) was lower than other two resins and the treated emulsion was also somewhat turbid.

The change of demulsification and deoiling effect with time is shown in Figure 3(b). In the initial stage of the reaction, the resin reacted rapidly with the emulsion, and a large content of oil was removed. One hour later, the removal rate stabilized gradually and peaked at 80 minutes. It can be observed that some big oil droplets floated on the surface of the liquid and the originally turbid emulsion became clear and transparent. A part of the resin located at the

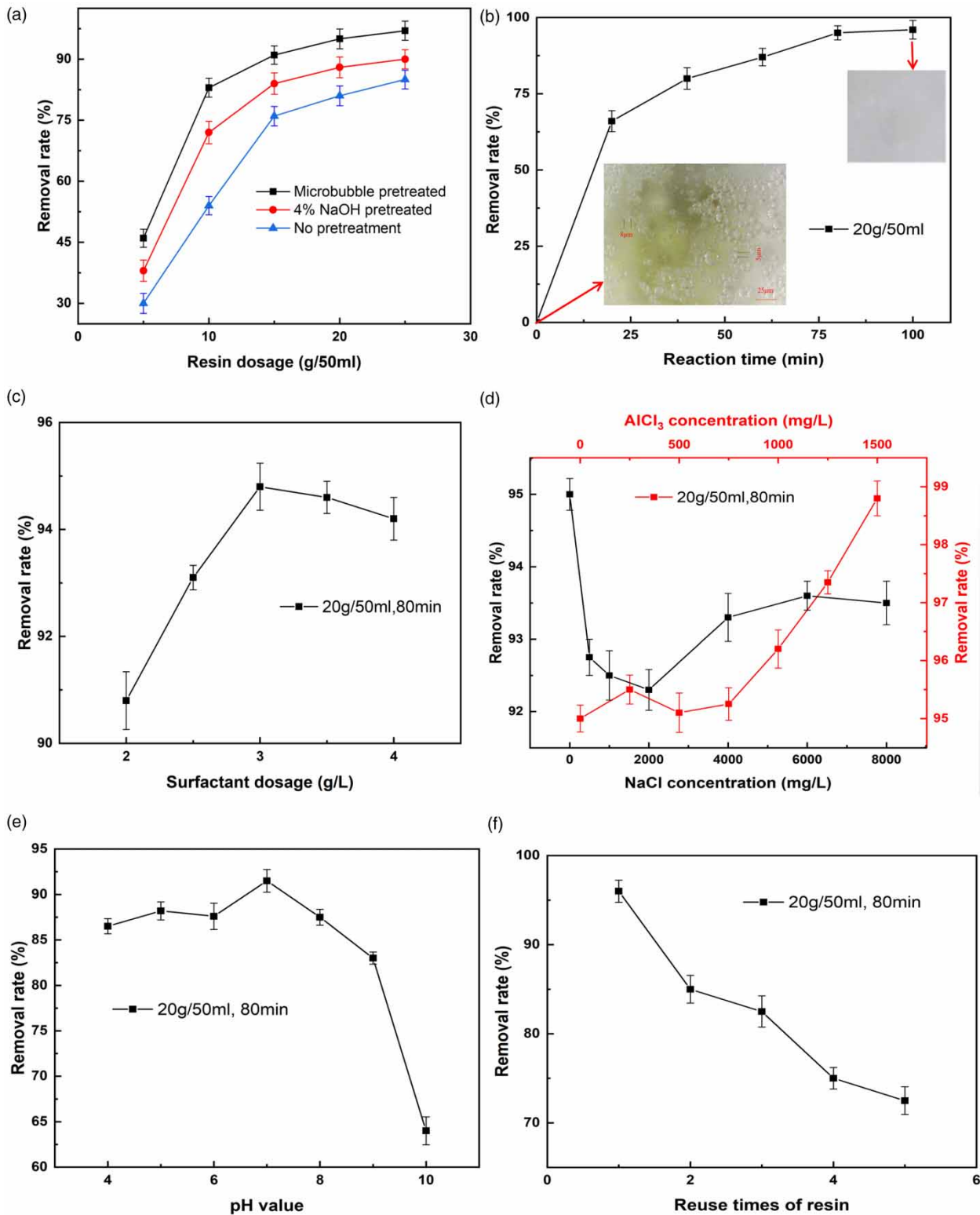


Figure 3 | The comparison of demulsification by different pretreatment methods within 80 min reaction, 20 g resin/50 ml emulsion (a); the effect of reaction time on demulsification, 20 g resin/50 ml emulsion (b); the influence of surfactant dosage on demulsification within 80 min reaction, 20 g resin/50 ml emulsion (c); the influence of salt concentration on demulsification within 80 min reaction, 20 g resin/50 ml emulsion (d); the effect of pH on demulsification within 80 min reaction, 20 g resin/50 ml emulsion (e); the effect of repeated use of resin on demulsification, within 80 min reaction, 20 g resin/50 ml emulsion (f).

bottom of the conical flask in the form of clusters, which can be attributed to the oil film coating on the resin.

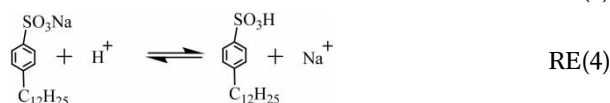
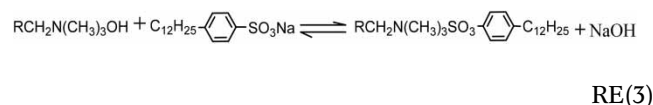
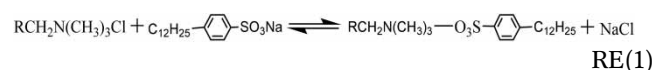
Surfactant played a major role in the preparation and purification of the emulsion. The influence of surfactant on demulsification is shown in Figure 3(c). Obviously, all the removal rates were above 90% with the surfactant in the range of 2.0 g/L–4.0 g/L. When a small amount of SDBS was added to prepare the emulsion, it was difficult to disperse the oil completely in water, which resulted in some free oil particles in the emulsion. The free oil particles were preferentially adsorbed by the resin and then the surfactants were rejected for contact with the resin. However, when the dosage of the surfactant was over 3.0 g/L, there was a large amount of free surfactant dissolved in the water and the exchange groups of the resin were occupied preferentially by the dissolved surfactant, which also caused a decreased oil removal.

The effect of salinity (NaCl and AlCl₃ concentration) on demulsification is shown in Figure 3(d). It can be seen that the oil removals with salt are lower than without salt, which indicates that the salt exhibits an adverse effect on demulsification performance of the resin. This phenomenon can be attributed to the inhibition effect of Na⁺ and Cl⁻ on the combination between resin and surfactant, as shown in Reaction 1. Adding NaCl into the emulsion is equivalent to regenerating resin that had adsorbed surfactant. However, the adverse effect of NaCl on demulsification performance is weak in the additional experimental range and the oil removal was still over 90%. It was presumed that the ion exchange capacity of the resin was much larger than the amount of chloride ion added.

Considering the basicity of the emulsion and resin, metal ions that can be hydrolyzed may have a different effect on demulsification. As can be seen in Figure 3(d), the oil removal rate increases when adding a small amount of aluminum chloride into the emulsion. The aluminum ions were hydrolyzed and dispersed colloiddally in the emulsion, and began to coalesce as the concentration of aluminum ions increased (Li *et al.* 2014). Aluminum hydroxide colloid and meta-aluminate formed in the emulsion can capture and cause the coalescence of oil particles, which results in the improvement of demulsification and deoiling efficiency.

The influence of initial emulsion pH on the demulsification effect was investigated in the pH range from 4 to 10 and the results are shown in Figure 3(e). When the initial emulsion pH < 7, it was known from Reactions (1)–(3) that it was advantageous to transfer RCH₂N(CH₃)₃OH to RCH₂N(CH₃)₃Cl, the ion exchange reaction between resin and surfactant was enhanced. However, as the hydrogen ion

concentration increased, that is, the pH decreased, the SDBS obtained hydrogen ions to form an acid, shown as Reaction (4). The molecular surfactant, of which the hydrophobic end had combined with oil particles, cannot be exchanged by the resin, which resulted in a slight reduction in demulsification. When the initial emulsion pH > 7, which mainly occurred in Reactions (2), and (3) and the exchange of the resin and surfactant was suppressed, the demulsification effect was lowered. There may exist some competitive relationship among Reactions (1)–(4). From the experimental results, it can be seen that Reaction (1) was dominant when the emulsion was neutral, and the demulsification effect was on the top level. When changing the pH value, Reactions (2)–(4) will reduce the probability of Reaction (1) and harm the demulsification performance.



The reusability of resin in demulsification was investigated and is shown in Figure 3(f). The resin was separated from water after each run, and then reacted with new emulsion again. It was found that the high demulsification and deoiling efficiency was retained after five repetitions. In other words, 1 cubic metre of resin was used to treat 10–20 cubic metres of emulsion with an oil content of 800–1,000 mg/L, and the removal rate can still be maintained at over 70%.

Discussion of demulsification mechanism

Zeta potential of emulsion

The zeta potential was an important parameter in O/W emulsion; usually, the oil particles combined with surfactant and formed colloids dispersed in water under electrostatic repulsion (Avranas *et al.* 1988). This electrostatic repulsion was dependent on the zeta potential between oil droplets. The higher the zeta potential, the stabler the emulsion, and vice versa. The zeta potential before and after

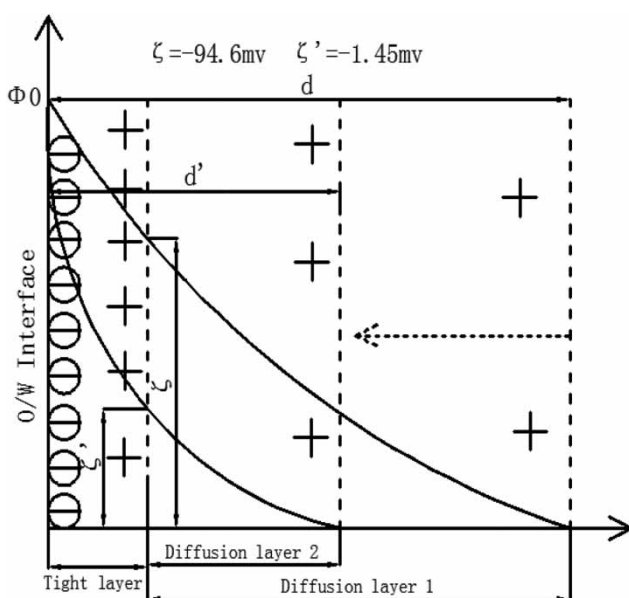


Figure 4 | The schematic diagram of zeta potential before and after demulsification (Smith *et al.* 2017). ζ : The zeta potential of initial emulsion; ζ' : The zeta potential of treated emulsion after 80 min reaction.

demulsification is shown in Figure 4 and this schematic diagram can refer to the literature (Smith *et al.* 2017). The zeta potential was reduced from -94.6 mV to -1.45 mV after demulsification, which meant that the final solution was not as stable as the initial emulsion, since a large content of oil and surfactant were separated during the demulsification. Table 1 summarizes the relationship between zeta potential and emulsion stability. The stability of the emulsion is directly related to the particle size of the fine oil particles dispersed in emulsion.

Change of surface tension and contact angle of emulsion

Figure 5 shows the suspended drop states and the spreading states on the glass sheet of the emulsions after 20, 40, 60, and 80 min treatment, respectively. Actually, the

size of suspended droplet and the contact angle of spreading drops are positively correlated with the surface tension of liquid. The surface tension and contact angle of water will decrease sharply when contained with organic matter like oil and surfactant, so the initial emulsion had a small surface tension and contact angle (Singh *et al.* 1984). It can be seen from Figure 5(a)–5(d) and 5(1)–5(4) that the size of the suspended droplet and the contact angle of the spreading drop changed during the demulsification. As summarized in Figure 5(5), the surface tension and contact angle of the emulsion kept increasing as the demulsification reaction progressed. As can be seen from this figure, after reacting for 80 min, the surface tension changed from 24.45 mN/m to 58.6 mN/m and the contact angle changed from 7.4° to 46.8° . The constantly increasing surface tension and contact angle indicated that a large amount of oil particles and surfactants were separated from the emulsion. However, the surface tension and the contact angle of natural water are 72.7 mN/m and 49.02° respectively at 20°C , which is much higher than that of the demulsified and deoiled emulsion. This may be caused mainly by the residual surfactant in water.

Infrared spectrum of resin

The change of surface groups of resin before and after demulsification was helpful in investigating the demulsification and deoiling mechanism. The Fourier transform infrared (FT-IR) spectrum is shown in Figure 6(a)–6(d). It can be clearly seen that the absorption peak of C-Cl, C-N, C-H, N-H stretching vibration occurred at about 680 cm^{-1} , $1,250\text{ cm}^{-1}$, $2,800\text{--}3,000\text{ cm}^{-1}$ and $3,500\text{ cm}^{-1}$ respectively in all spectrums. However, comparing with Figure 6(a), there are some differences in Figure 6(b), an absorption peak of outside bending vibration of C-H on the benzene ring appeared at about 678 cm^{-1} and the absorption peak of $-\text{SO}_3\text{H}$ appeared at $1,190\text{ cm}^{-1}$, which meant that SDBS may be transferred to the resin. In Figure 6(c), the absorption peak of the C=O absorption peak was obtained at $1,750\text{ cm}^{-1}$. Moreover, a C=C bond appeared in the range of $1,450\text{ cm}^{-1}\text{--}1,060\text{ cm}^{-1}$, which was the signal that the oil was adsorbed by the resin. It was worth noting that there is both outside bending vibration of C-H on the benzene ring, $-\text{SO}_3\text{H}$ and C=O bond in Figure 6(d). In summary, the FT-IR spectrums shown in Figure 6 indicate that both the adsorption of oil and the ion exchange of surfactant by the resin can happen in the demulsification process.

Table 1 | Zeta potential and its corresponding properties

Zeta potential (mv)	Property
0 to ± 10	Rapid condensation or condensation
± 10 to ± 30	Becoming unstable.
± 30 to ± 40	General stability
± 40 to ± 60	Better stability
Over ± 61	Excellent stability

Note: the table is quoted in zeta potential of 360 encyclopedia.

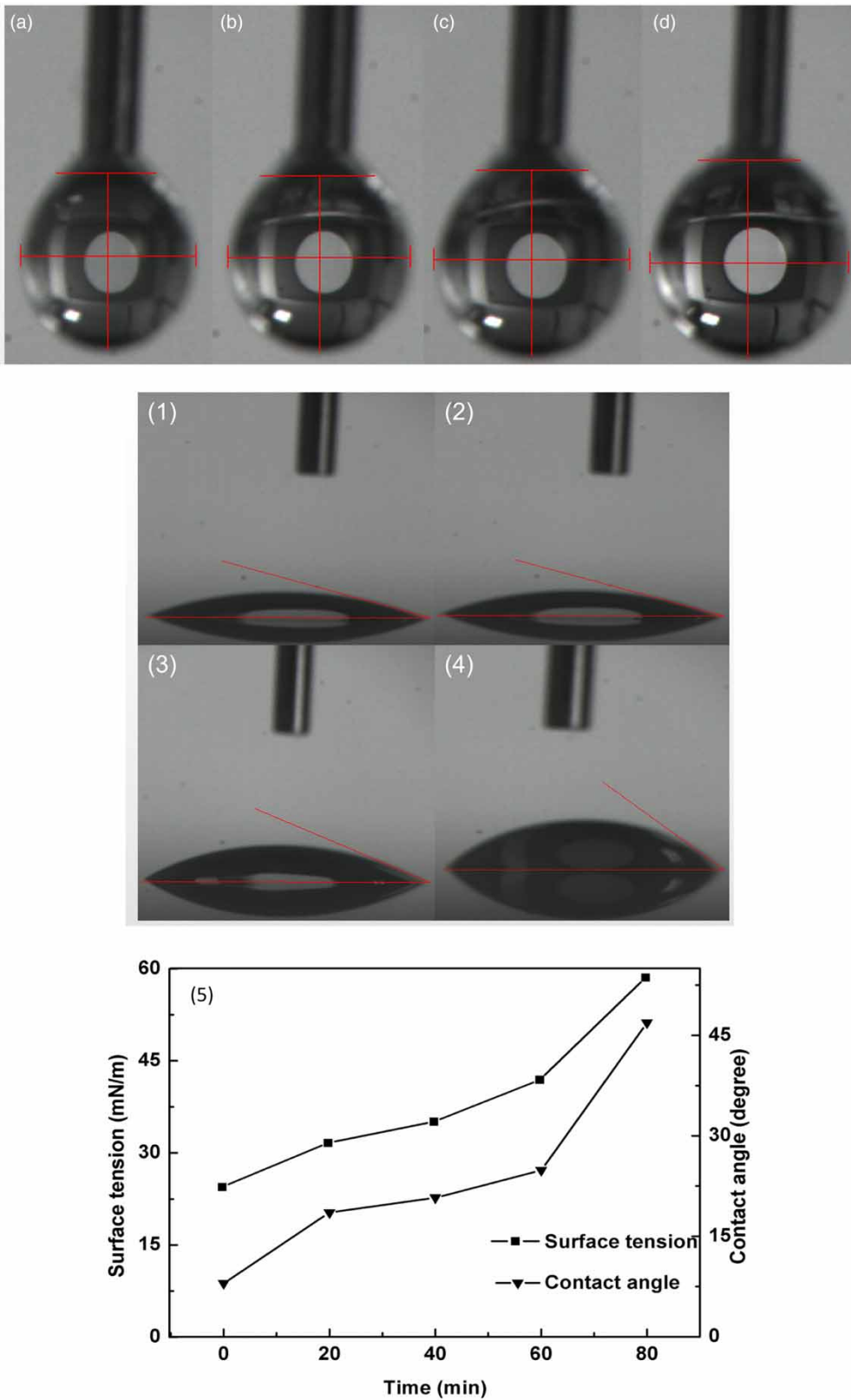


Figure 5 | Changes of emulsion droplets and properties in the demulsification process. Suspended drop at 20 min (a); 40 min (b); 60 min (c); 80 min (d) for the surface tension measurement. Spreading state of emulsion at 20 min (1); 40 min (2); 60 min (3); 80 min (4) for the contact angle measurement. The trend of emulsion surface tension and contact angle (5) within 80 min demulsification reaction.

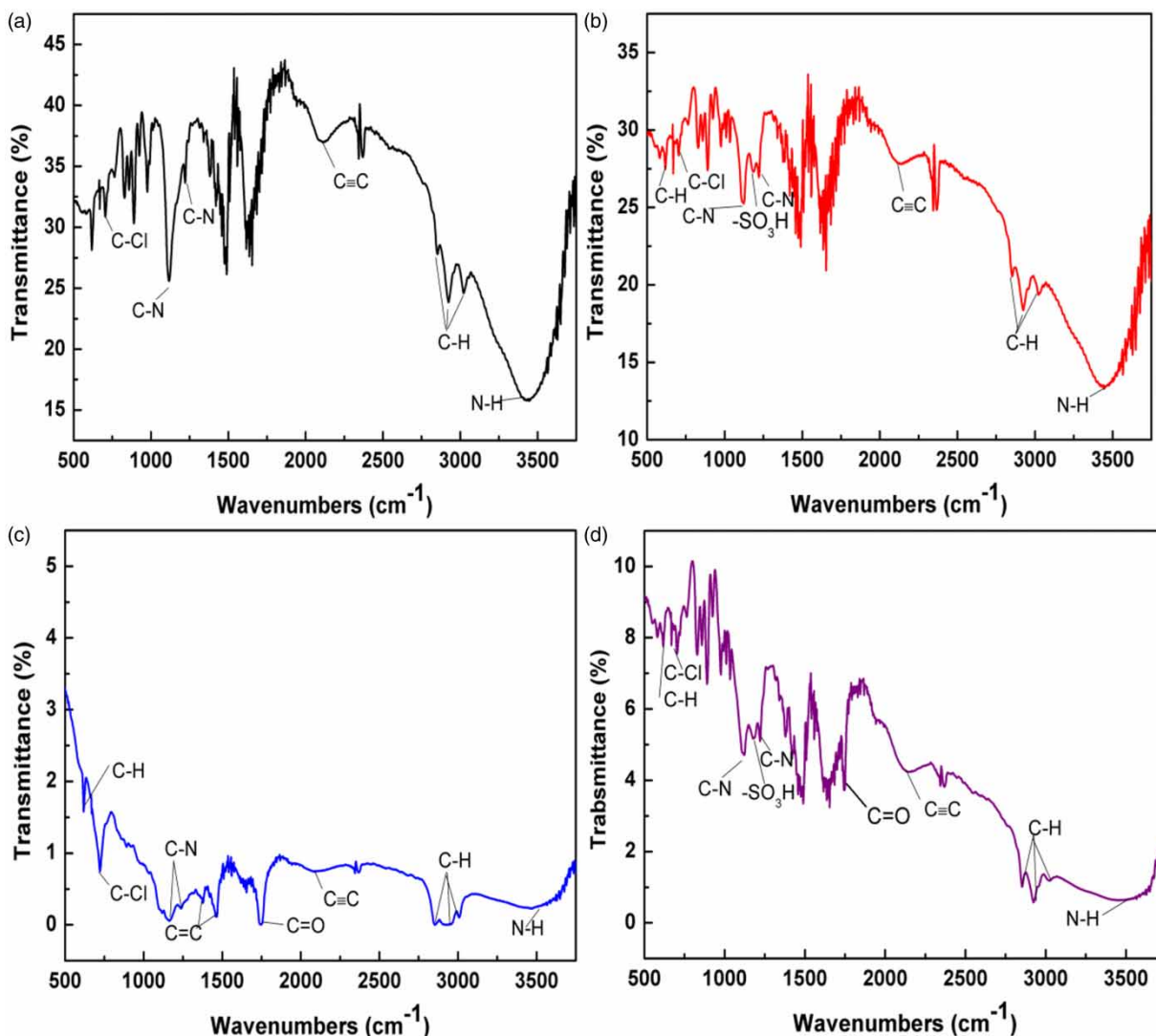


Figure 6 | Changes in surface groups of resin in demulsification process. The new resin (a); after soaking in SDBS for 2 h (b); after soaking in oil for 2 h (c); and the resin reacted with simulated emulsion in this experiment for 80 min (d).

Appearance of the resin

The change in appearance of the resin and its aggregation state after the demulsification reaction are presented in Figure 7(a) and 7(b). It is clearly shown that the surface of the raw resin is clean and free of impurities; however, the surface of the used resin is coated with an oil film and clustered together. Previous results have shown that a few oil droplets floated on the surface of the water.

Demulsification mechanism

The zeta potential, the surface tension and the contact angle of the emulsion, the appearance of the resin and the change

of surface groups were characterized in the experiments. Based on the experimental results and analyses, the comprehensive demulsification and deoiling mechanism can be proposed as in Figure 8. There are three possible pathways. Pathway 1: Competitive trapping of surfactant. The cationic groups of the resin combine with the anionic groups (the hydrophilic end) of the surfactant and drag them away from the oil particle surface. Pathway 2: Distribution equilibrium of surfactant. Free surfactant in the emulsion is captured by resin, which will reduce the concentration of uncombined surfactant. As a result, surfactants on the oil particle partly detach from the oil surface to maintain the adsorption-desorption equilibrium of the surfactant. Pathway 3: Adsorption coalescence. The oil-surfactant complexes are

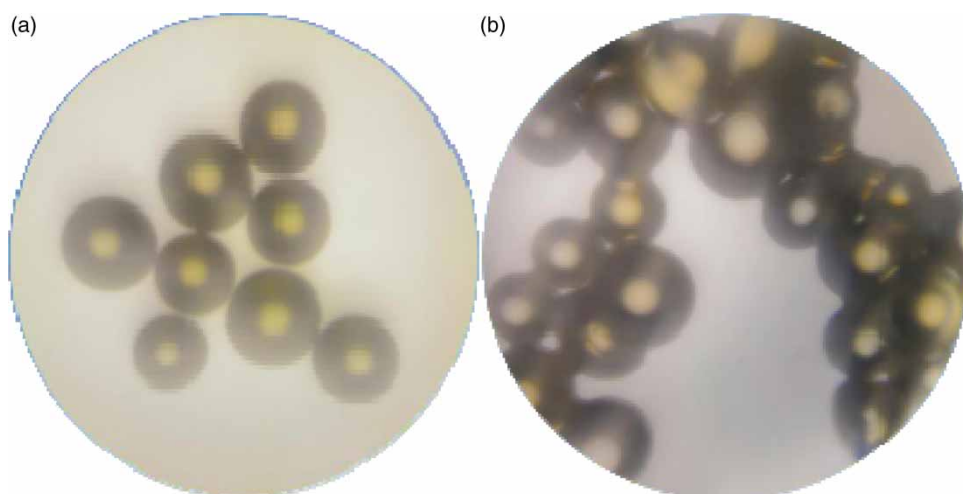


Figure 7 | Changes in appearance of resin during demulsification. New resin (a); after 80 minutes demulsification reaction (b).

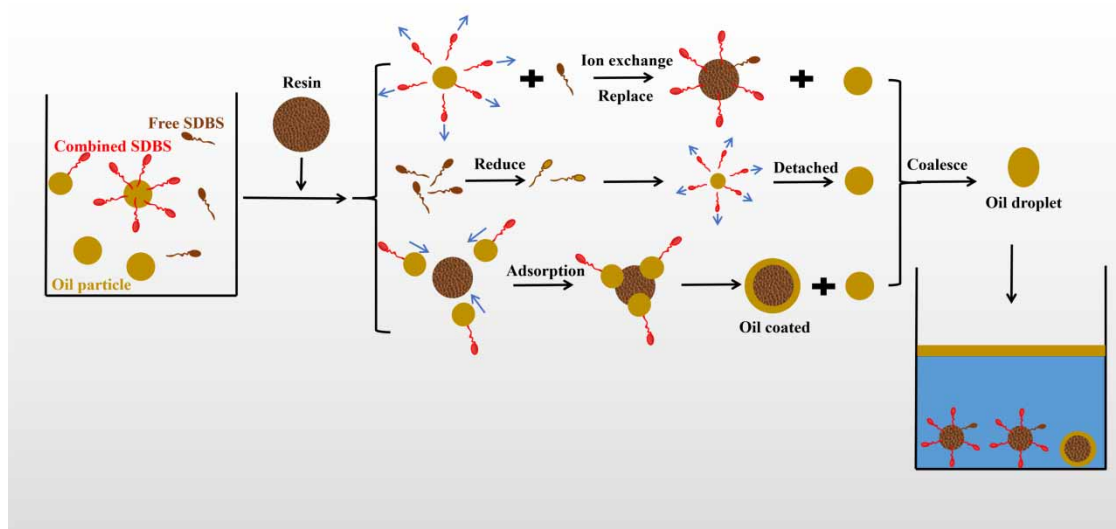


Figure 8 | The concluded demulsification mechanism.

coalesced onto the resin because of the adsorption effect of resin on oil. The oil will detach from the resin when excess oil particles enrich on the surface of the resin. When excess oil enriches on the surface of the resin, big oil droplets will form and detach from the resin surface.

Pathway 1 is the main direct way to demulsification and is proved by the characteristic functional groups of surfactant in the infrared spectrum of used resin. The surfactant on resin may include surfactant-oil complexes and free surfactants. Pathway 2 plays an important role in demulsification and can be proved by the zeta potential, the surface tension, the contact angle and the floating oil droplets. The direct result of pathway 2 is that the stability

of the oil particles decreases and they coalesce to big oil droplets, which can float onto the water surface. Pathway 3 helps to remove oil from water. This pathway was concluded from the comparison of resin appearances before and after use. Moreover, pathway 3 can also be proved by the characteristic functional groups of oil in the infrared spectrum of used resin.

CONCLUSIONS

In summary, a series of influencing factors such as the resin dosage, reaction time, surfactant dosage, salt concentration,

pH, recycle experiment for the demulsification and deoiling of O/W emulsion was systematically investigated. The changes of physical and chemical properties are characterized and mainly obtain the following contributions:

- (1) The resins used in demulsification by different pretreatment methods are compared, the microbubble pretreated resin obtains the best demulsification and deoiling effect (97%) followed by the 4% NaOH pretreated resin (90%) and the non-pretreated resin (85%).
- (2) The resin has a considerable demulsification and deoiling effect and the high demulsification and deoiling efficiency was still retained at about 70% or more after repeated use for five times. The new pretreatment method avoids producing elution waste liquor, is easy to operate and low cost.
- (3) Compared with the previous studies using resin, we reveal some more comprehensive demulsification mechanisms than that report of a certain mechanism.

Competitive trapping of surfactant

The cationic groups of the resin combine with the anionic groups (the hydrophilic end) of the surfactant and drag them away from the oil particle surface.

Distribution equilibrium of surfactant

Free surfactant in the emulsion is captured by resin, which will reduce the concentration of uncombined surfactant. As a result, surfactants on the oil particle partly detach from the oil surface to maintain the adsorption-desorption equilibrium of the surfactant.

Adsorption coalescence

The oil-surfactant complexes are coalesced onto the resin because of the adsorption effect of resin on oil. The oil will detach from the resin when excess oil particles are enriched on the surface of the resin. When excess oil enriches on the surface of resin, big oil droplets will form and detach from the resin surface.

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