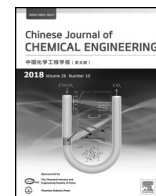




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Article

De-emulsification of 2-ethyl-1-hexanol/water emulsion using oil-wet narrow channel combined with low-speed rotation

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ABSTRACT

Low-speed rotation of disc in an internal circulation of a novel de-emulsification with rotation-disk horizontal contactor (RHC-D) realized de-emulsification for O/W emulsions due to repeated coalescence in oil-wet narrow channels at a low rotation speed. For three emulsions included ethanol/water/2-ethyl-1-hexanol, ethanol/water/2-ethyl-1-hexanol/SDS (Sodium Dodecyl Sulfonate) and 2-ethyl-1-hexanol/water/SDS emulsion, de-emulsification ratios of oil phase could reach 1, 1 and 0.67 respectively at $170 \text{ r} \cdot \text{min}^{-1}$, and de-emulsification ratios increased obviously after agitating 10 min. De-emulsification experiment in the seam indicated that oil droplet sizes in O/W emulsion became larger after de-emulsification. The main de-emulsification mechanism in RHC-D was the coalescence of oil droplets in oil-wet narrow channels. With increase of the rotation speed, oil droplets dispersed better in the aqueous phase. However, de-emulsification effect enhanced due to the increase of the coalescence rate at a bit higher rotation speed. In addition, internal circulation made those O/W emulsions to be broken repeatedly, consequently de-emulsification ratio increased. Repeated de-emulsification through internal circulation might make continuous extraction of ethanol come true at a low rotation speed.

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1. Introduction

Ethanol is used for bio-fuel which is expected to replace fossil fuels [1–3]. Biological fermentation was one of main methods for produce ethanol [4]. The mixture prepared from biomass fermentation was dilute aqueous solution usually contained 3 %–8% ethanol. In the United States, azeotropic distillation was the predominant method for recovering ethanol from dilute aqueous fermentation broths, but it consumed about 14000–17000 Btu/gallon ethanol [5]. Emulsion liquid membrane (ELM) is a cheap and competitive method to separate various solutes from aqueous solutions because both extraction and stripping steps are combined in one stage [6–8]. However, the major problem of emulsion liquid membrane is the stability of the emulsion globules in the application [9]. Small droplets have better stability and high surface-to-volume ratio, but if the droplet diameter is too small, the emulsion is very difficult to be broken by any mechanical means [10].

Solvent extraction using low cost extractants may provide a more energy-saving method to recover ethanol [11–13]. 2-Ethyl-1-hexanol could be used as a kind of ideal extraction agent to extract ethanol

from dilute aqueous solution because of better distribution coefficient and separation factor, non-toxic for fermented liquid, the cheap price and high boiling point [3,5,14]. However, water, ethanol and 2-ethyl-1-hexanol could form O/W emulsion easily under the stirring condition during the extraction process. Critical conditions, such as centrifugation of 276 G, should be applied for complete separation. It is difficult to realize continuous separation in the ethanol extraction process [5]. Therefore, high efficient and fast de-emulsification is necessary for emulsion of ethanol, water and 2-ethyl-1-hexanol.

De-emulsification of typical O/W emulsion could be realized via physical methods such as filtration [15–18], magnetic nanoparticles [19], centrifugal force [20], gravity sedimentation [21,22], electric field [23], micro-channel [24], hydrocyclone [25] etc. De-emulsification in the micro-channel possessed high efficiency and easy operation. For example, dynamic filtration with rotating disk in the narrow mixing space forced emulsion flow through membrane channels to realize ultrafiltration of O/W emulsion with droplets radius of 50–3000 nm [18,26–30]. Chen reported that de-emulsification efficiency of O/W emulsions with different droplets diameters of 5–10 μm could reach 30% when emulsions passed through stainless steel micro-channels of 100 to 200 μm at the flow rate less than $40 \text{ ml} \cdot \text{min}^{-1}$ [24]. Coalescence of the dispersed droplets in O/W emulsion also occurred by passing through the PTFE micro-channel at the flow rate less than $30 \text{ ml} \cdot \text{min}^{-1}$ [31]. The O/W emulsion produced in the phosphoric acid extraction process

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was demulsified at $180 \text{ r} \cdot \text{min}^{-1}$ with silk fishing net stirrer and de-emulsification ratio reached as high as 98% [32]. However, O/W emulsion was demulsified through internal circulation pathway under the low-speed mixing which emulsion pass through oil-wet narrow channels repeatedly has not been reported.

In this work, a novel de-emulsification device RHC-D was made where O/W emulsion flowed through oil-wet seams repeatedly by internal circulation at the low rotating speed. Oil droplets coalesced repeatedly in oil-wet narrow seams and then could be separated from the emulsion. De-emulsification rates under different stirring time and settling time with different rotational speeds for ethanol/water/2-ethyl-1-hexanol emulsion, ethanol/water/2-ethyl-1-hexanol/SDS emulsion and 2-ethyl-1-hexanol/water/SDS emulsion were discussed. Experimental results showed that the design of internal circulation enhanced de-emulsification rate due to repeated coalescence. This work put forward an inner circulation method at a low agitating speed to make the O/W emulsion separate. It might become possible to realize the continuous extraction for ethanol from water.

2. De-Emulsification Equipment and Experiment Preparation

2.1. De-emulsification equipment

In this work, a novel de-emulsification equipment RHC-D with an internal circulation was made to realize de-emulsification repeatedly in the middle chamber with low-speed mixing and narrow oil-wet seams between chambers. Stirring in the middle chamber generated the differential pressure and realized the inner circulation of emulsion. The emulsion could flow into middle chamber through orifices and then flow through narrow seams repeatedly. Oil droplets in the emulsion might be collided by low shear-force in the narrow mixing space, and then coalesced in oil-wet narrow seams between chambers, finally could be separated from the emulsion in the settler chambers.

Fig. 1 showed the diagram of de-emulsification process inside the RHC-D. De-emulsification equipment was cylindrical in shape, and it had three chambers including both side chambers and the middle one where rotational solid disc installed. A stainless steel disc of 10 cm diameter and thickness of 2 mm in the middle chamber could be drove by a horizontal axis connected by a motor. The thickness of the wall between chambers was 7 mm. The wall was made of stainless steel, and the opening ratio of orifices on the same circle near the center of each wall respectively was 0.16%. The opening ratio of seams located at the edge of cross wall was 0.8%. Chambers on both sides were cylindrical cavity of 10 mm height where two phases could be separated. The distance between disc and cross wall was 1 mm in the middle chamber so enough centrifugal force produced by agitating disc made emulsion

flow through narrow oil-wet seams at a low rotation speed. The pressure difference between the center and the edge forced liquids flow from the intermediate orifices into the middle chamber and then to be thrown out from the edge repeatedly. Consequently, an inner circulation formed, which realized de-emulsification repeatedly in seams. Oil phase could be separated from emulsion, and the interface of oil phase and emulsion phase could be seen clearly in two side chambers.

2.2. Material

All chemical reagents used in the experiment were of pure analysis and without further purification. Chemical reagents included: ethanol, 2-ethyl-1-hexanol, SDS and deionized water. Diameters of oil droplets in the emulsion were detected by a laser particle analyzer.

2.3. Preparation of the stable emulsion

To study the de-emulsification effects of O/W emulsions in RHC-D, ethanol/water/2-ethyl-1-hexanol emulsion was prepared firstly. 50 ml ethanol aqueous solution (20 wt%) and 50 ml 2-ethyl-1-hexanol were put into a 250 ml volumetric flask, then shocked violently for 2 min and formed O/W emulsion which could dissolve in water but not in 2-ethyl-1-hexanol. After 5 h, there were traces of oil phase separated from emulsion and a small amount of water presented on the bottom of flask. To improve the stability of the emulsion, 0.1 wt% SDS was added to 20 wt% ethanol aqueous solution and stirred them until dissolved completely. Same volume of 2-ethyl-1-hexanol and 20 wt% ethanol aqueous solution were mixed intensively to form stable white emulsion. As it was shown in Fig. 2, oil phase could not be separated from the emulsion after 10 d but some water could be separated because emulsion droplets floated and gathered. SDS could adsorb on the surface of the oil droplets, and prevent the coalescence of oil droplets. The results showed that the ethanol/water/2-ethyl-1-hexanol/SDS system was easy to form emulsion where oil phase was stable. To study the effect of ethanol on emulsion, 2-ethyl-1-hexanol/water/SDS emulsion was also prepared.

2.4. De-emulsification experiment

Firstly, effects of the stirring time and settling time at different rotating speeds in the internal circulation pathway of RHC-D were discussed. There were three emulsions included ethanol/water/2-ethyl-1-hexanol system, ethanol/water/2-ethyl-1-hexanol/SDS system and 2-ethyl-1-hexanol/water/SDS system. After the emulsion formed, let the emulsion fill fully in RHC-D at once, then start the motor at an adjusted rotating speed. The phenomenon of de-emulsification was visible at a low-speed of rotation in two side chambers of equipment. We could see the upper oil phase being separated from the lower emulsion layer; two phases interface declined, emulsion volume decreased and the emulsion color became shallow. To quantitatively measure separated volumes of oil phase and aqueous phase, all liquids were released in the measuring cylinder for different settling times after stopping stirring. The separation efficiency of oil phase and water phase was calculated by measuring the volume of oil phase and water phase within different settling times. De-emulsification ratios were calculated as follows:

$$\eta_{(\text{oil})} = \frac{V_{\text{oil,d}}}{V_{\text{oil,i}}} \times 100\% \quad (1)$$

$$\eta_{(\text{water})} = \frac{V_{\text{water,d}}}{V_{\text{water,i}}} \times 100\% \quad (2)$$

where, $\eta_{(\text{oil})}$ and $\eta_{(\text{water})}$ were the de-emulsification ratios of oil phase and aqueous phase respectively. $V_{\text{oil,d}}$ and $V_{\text{water,d}}$ were the volumes of oil phase and aqueous phase separated from emulsion.

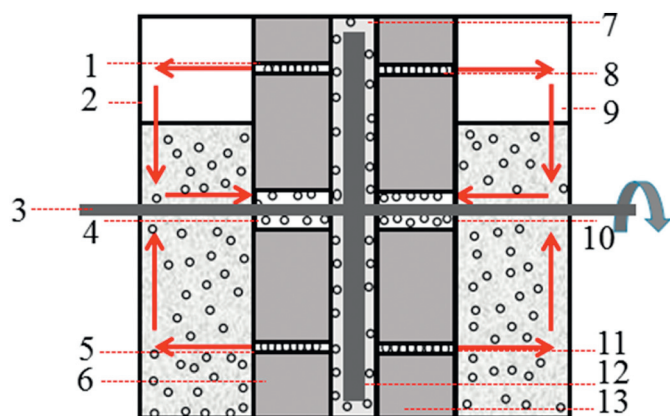


Fig. 1. The diagram of RHC-D: 1, 5, 8, 11—seam; 2—inlet chamber; 3—rotating shaft; 4, 10— orifice; 7—middle chamber; 9—outlet chamber; 12—solid disc; 6, 13—cross wall. Thickness of rotation disc: 2 mm; Solid disc diameter: 10 cm; Rotor-stator distance: 1 mm; Cross wall thickness: 7 mm; Settler chamber distance: 10 mm.



Fig. 2. Stability of ethanol/water/2-ethyl-1-hexanol/SDS system.

$V_{oil,i}$ and $V_{water,i}$ were the volumes of oil phase and aqueous phase in initial emulsion respectively.

To analyze the de-emulsification phenomenon, the de-emulsification effect through seams was respectively detected without stirring. Fig. 3 showed the process of de-emulsification only through seams. The O/W emulsion was filled in the one side chamber then forced through seams and orifices from the one side chamber to another side chamber at the definite flow rate by the peristaltic pump. Fluid filled fully in the whole de-emulsification device then flowed out of the export. The export fluid directly flowed into the entrance on the inlet chamber through a buffer bottle and formed an outer cycle. When inlet chamber was filled fully, the circle time was equal to 0. With the increase of time, aqueous phase could not be separated from the emulsion, but oil phase could be separated from emulsion. The volume of oil phase increased in the outlet chamber. De-emulsification ratio of oil phase could be estimated through calculating the oil phase volume in the outlet chamber within different times.

3. Results and Discussions

In this work, de-emulsification effects of three O/W emulsions in RHC-D were discussed. Three emulsions included ethanol/water/2-ethyl-1-hexanol system, ethanol/water/2-ethyl-1-hexanol/SDS system and 2-ethyl-1-hexanol/water/SDS system.

3.1. De-emulsification experiments in the internal circulation pathway

First, de-emulsification experiment of ethanol/water/2-ethyl-1-hexanol emulsion in the internal circulation pathway combined low speed rotating space with oil-wet narrow channels was discussed. Let equal volume of 20 wt% ethanol aqueous solution and 2-ethyl-1-hexanol form emulsion, then filled it fully in RHC-D. After agitating for 30 min at a fixed rotation speed, all liquids in the de-emulsification

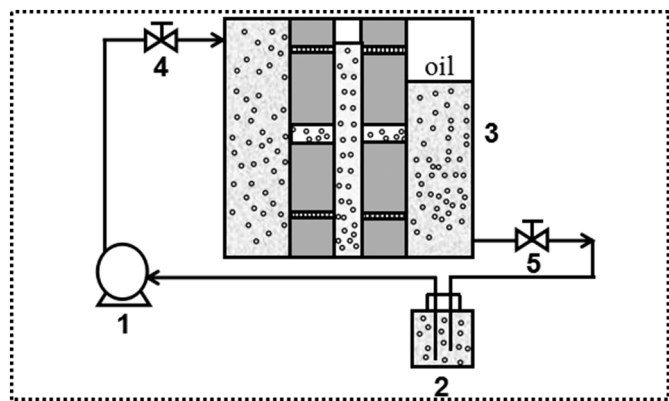


Fig. 3. Diagram of de-emulsification process in the seam: 1—peristaltic pump; 2—surge flask; 3—RHC-D; 4, 5—valve.

device were put in a measuring cylinder and de-emulsification ratio was detected within different settling times. When all liquids were put into measuring cylinder, the settling time was 0. The color of emulsion became shallow in two sides chambers because oil droplets coalesced during the de-emulsification process. As it was shown in Fig. 4 (a), with the increase of the settling time, the de-emulsification ratio of oil phase increased. De-emulsification ratio increased quickly before 10 min and was stable after settling time surpassed about 20 min. Under the low speed, de-emulsification ratio increased then decreased with the increase of stirring speed. De-emulsification effect was best at $170 \text{ r} \cdot \text{min}^{-1}$ and de-emulsification ratio of oil phase was 0.98. From Fig. 4(b), with the increase of settling time, de-emulsification ratio of aqueous phase increased then reached 0.95 when the settling time up to 20 min.

The de-emulsification effect might be the result of a combination of low speed mixing and coalescence in the seam. Under low-speed rotating of disc, low shear force mainly caused a part of oil droplet collision and burst but not generating smaller droplets. High shear force mainly promoted smaller oil droplets dispersed in the aqueous phase, consequently the emulsification was more serious. With decrease of rotation speed, the flow rate of emulsion through the seam decreased so discontinuous droplets flowed through the seam. Continuous droplets flowed through the seam with increase of rotation speed. Weak coalescence effect at a lower rotation speed but excellent coalescence effect at a higher rotation speed in the seam because of increase of pressure and flow rate through the seam.

Both oil phase and aqueous phase could be separated from the unstable emulsion of ethanol/water/2-ethyl-1-hexanol under the gravity. SDS could enhance the stability of ethanol/water/2-ethyl-1-hexanol emulsion. This work also discussed the de-emulsification effect of water/2-ethyl-1-hexanol/SDS emulsion and ethanol/water/2-ethyl-1-hexanol/SDS emulsion. As it was shown in Fig. 5 and Fig. 6, emulsions of two systems added SDS were demulsified at the low rotation speed. De-emulsification ratios increased quickly within 10 min and then reached stable after settling about 20 min. For water/2-ethyl-1-hexanol/SDS emulsion, the de-emulsification ratio of oil phase reached 0.76 in Fig. 5(a) and that of aqueous phase reached 0.94 in Fig. 5(b) at $170 \text{ r} \cdot \text{min}^{-1}$. For ethanol/water/2-ethyl-1-hexanol/SDS emulsion, the de-emulsification ratio of oil phase reached 0.63 in Fig. 6(a) and that of aqueous phase reached 0.24 in Fig. 6(b) at $170 \text{ r} \cdot \text{min}^{-1}$.

The experimental results showed that the low speed rotating in the narrow space combined oil-wet seams formed circulation flow which had excellent de-emulsification effect for O/W emulsion. It became more difficult to realize the separation of oil phase after adding ethanol or SDS might due to the increase of emulsion stability.

Fig. 7 showed the influence of agitating time on de-emulsification ratios of three kinds of emulsions including ethanol/water/2-ethyl-1-hexanol system, water/2-ethyl-1-hexanol/SDS system and ethanol/water/2-ethyl-1-hexanol/SDS system. It has been proven that the best rotating speed for de-emulsification was $170 \text{ r} \cdot \text{min}^{-1}$. So, all de-emulsification experiments within different agitating times were carried out at $170 \text{ r} \cdot \text{min}^{-1}$ and the same settling time of 30 min. From

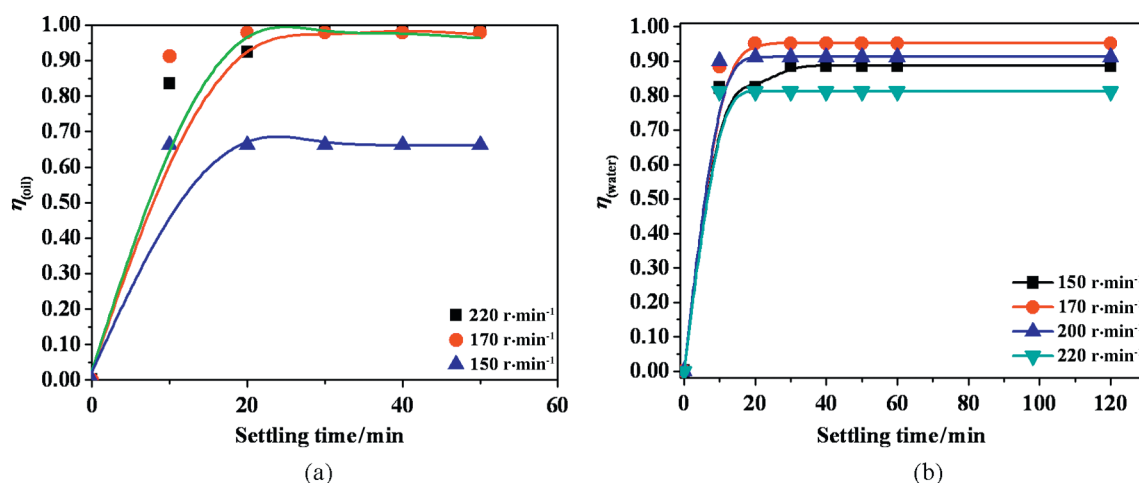


Fig. 4. Influence of rotation speed on ethanol/water/2-ethyl-1-hexanol after agitating 30 min then settling different time.

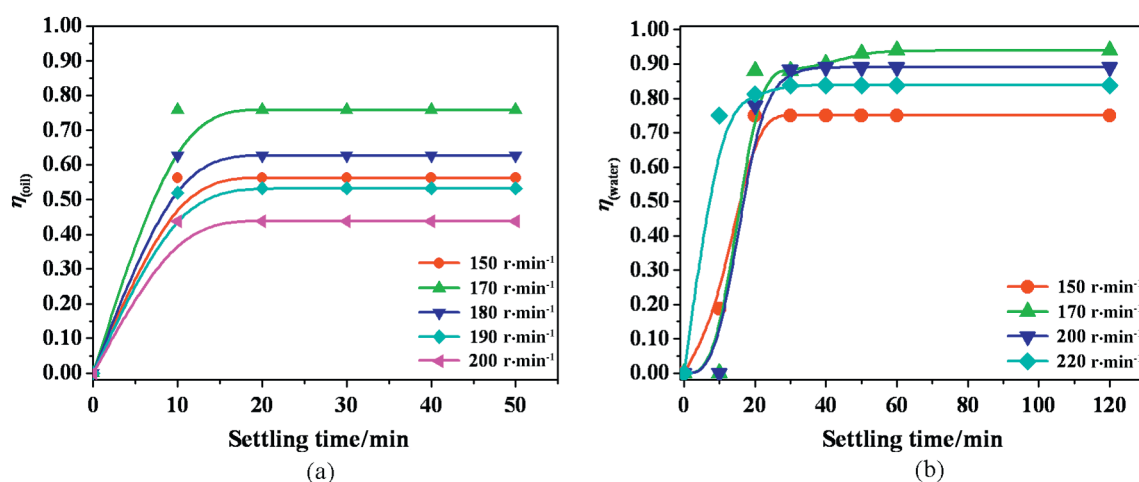


Fig. 5. De-emulsification ratios of oil phase and water phase. Agitating 30 min then settling different time for water/2-ethyl-1-hexanol/SDS emulsion.

this figure, de-emulsification ratio of ethanol/water/2-ethyl-1-hexanol emulsion improved obviously after agitating 10 min, and then reached stability gradually. The max values of de-emulsification ratio of both oil phase and water phase were nearly 1. Water/2-ethyl-1-hexanol/SDS emulsion was also broken at the different agitating times. The emulsion included equal volume of the 0.1% SDS aqueous solution and

2-ethyl-1-hexanol. De-emulsification ratios of two phases increased quickly within agitating 20 min. The de-emulsification ratio of oil phase was close to 1 and that of aqueous phase was 0.97 when emulsion reached stability. For ethanol/water/2-ethyl-1-hexanol/SDS emulsion, maximum values of de-emulsification ratios of oil phase and aqueous phase were 0.67 and 0.24 respectively. Under the same conditions, the

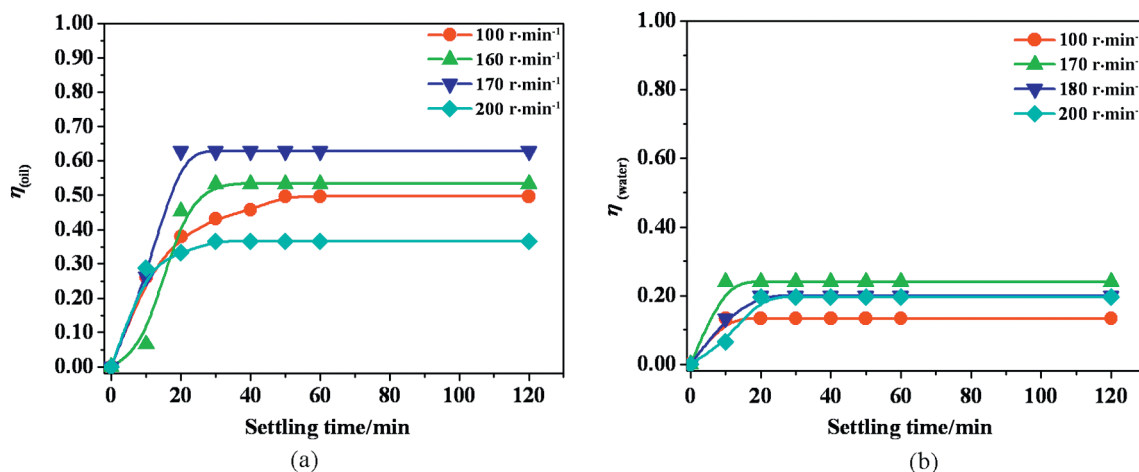


Fig. 6. De-emulsification ratios of oil phase and water phase. Agitating 30 min then settling different time for ethanol/water/2-ethyl-1-hexanol/SDS emulsion.

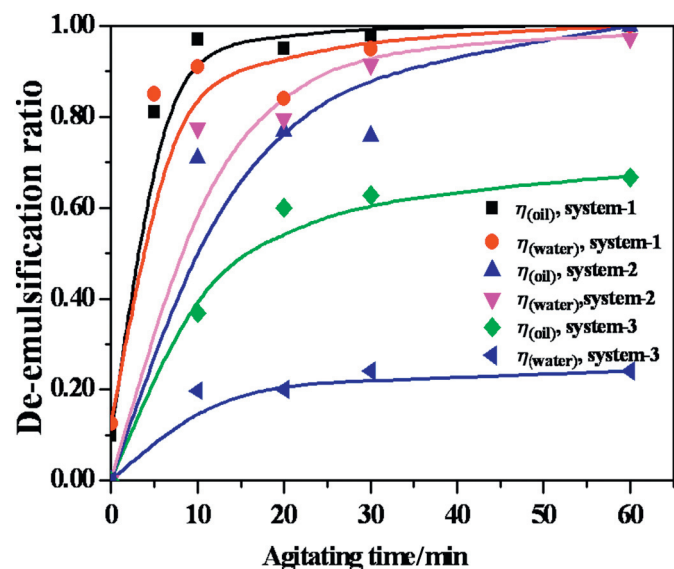


Fig. 7. Influence of agitating time on three emulsions at 170 rpm. System-1 was ethanol/water/2-ethyl-1-hexanol emulsion, system-2 was water/2-ethyl-1-hexanol/SDS emulsion, system-3 was ethanol/water/2-ethyl-1-hexanol/SDS emulsion.

sequence of the de-emulsification ratio of three emulsions: ethanol/water/2-ethyl-1-hexanol > water/2-ethyl-1-hexanol/SDS > ethanol/water/2-ethyl-1-hexanol/SDS.

Experimental results showed that de-emulsification effects of those three emulsions were obvious in a short agitating time at a low agitating speed. With the increase of agitating time, the de-emulsification ratio increased then achieved stability. After adding ethanol and SDS, the emulsion became more stable so that de-emulsification became more difficult, the consequence of time for achieving stable de-emulsification ratio was longer and the de-emulsification ratio was lower. Finding a better oil-wet material instead of stainless steel might result in a better de-emulsification effect for O/W emulsion added SDS.

3.2. De-emulsification experiment in the seam

Emulsion flow through internal circulation included low rotating speed space and oil-wet seams. To further discuss the reason of separation in the de-emulsification equipment, the de-emulsification effect only in the seam was detected. During the de-emulsification process, oil phase could be separated from emulsion in the outlet chamber but aqueous phase could not. De-emulsification ratio of oil phase could be calculated through detecting the position of two phase interface in the outlet chamber.

Fig. 8 showed the de-emulsification effect of ethanol/water/2-ethyl-1-hexanol/SDS emulsion in the seam. From this figure, the de-emulsification ratio of oil phase was proportional to circle time. The de-emulsification ratio of oil phase was 2.4% at the flow rate of $50 \text{ ml} \cdot \text{min}^{-1}$ for 2 min. The results indicated that oil droplets in the emulsion could coalesce obviously in a short time. Fixing the flow rate of $50 \text{ ml} \cdot \text{min}^{-1}$ and the circle time of 10 min, the de-emulsification ratio of oil phase was 5.4%. Adjusting the flow rate of $70 \text{ ml} \cdot \text{min}^{-1}$ and the circle time of 10 min, the de-emulsification ratio of oil phase reached 14.5%. With the increase of the flow rate of emulsion, the de-emulsification ratio of oil phase increased. The oil phase separated probable main attributed to the narrow oil-wet channel. With increase of flow rate, the pressure of emulsion through the seam increased and frequency through the seam increased, consequently the de-emulsification effect in the oil-wet seam enhanced.

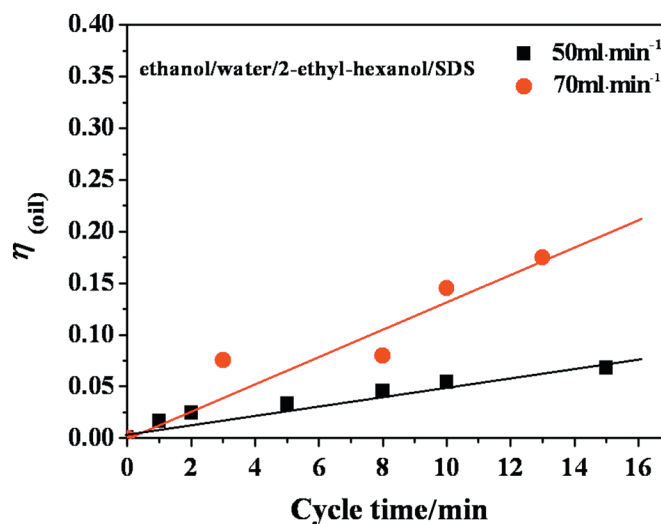


Fig. 8. Influence of the seam on de-emulsification ratio of ethanol/water/2-ethyl-1-hexanol/SDS emulsion at different flow rates. 0.1% SDS was added to ethanol water solution.

3.3. De-emulsification mechanism

To analyze the de-emulsification mechanism, the droplet size distribution of ethanol/water/2-ethyl-hexanol/SDS emulsion was detected by a laser particle analyzer. Fig. 9(a) showed the droplet size distribution after repeated de-emulsification through narrow mixing space combined narrow oil-wet seams at a low rotating speed of $170 \text{ r} \cdot \text{min}^{-1}$. From this figure, the average droplet diameter was $1.6 \mu\text{m}$ in the initial emulsion. After 10 min, part of oil phase was separated and the average droplet diameter in emulsion was $37 \mu\text{m}$. The results indicated that a part of small oil droplets coalesced and became bigger droplets which resulted in the separation of oil phase from emulsion. To detect the coalesce phenomenon in the seam and confirm the main de-emulsification mechanism, droplet size distribution of ethanol/water/2-ethyl-hexanol/SDS emulsion before and after de-emulsification only in oil-wet seams was measured. From Fig. 9(b), the average diameter of oil droplets in the initial emulsion was $2 \mu\text{m}$. After de-emulsification, the average diameter of oil droplets was $88 \mu\text{m}$ under the flow rate of $50 \text{ ml} \cdot \text{min}^{-1}$ and $99 \mu\text{m}$ under the flow rate of $70 \text{ ml} \cdot \text{min}^{-1}$. It indicated that small oil droplets coalesced in seams and became larger droplets after through oil-wet seams. With the increase of flow rate of emulsion, de-emulsification ratio increased because pressure and frequency of coalescence through the seam increased. In other words, de-emulsification ratio in seams enhanced with the increase of rotation speed.

Comparing Fig. 9(a) with (b), the large droplet size after de-emulsification through two methods included only in the seam and in the seam combined narrow rotation space was near. The average droplet size after de-emulsification only in the seam was larger than that in the internal recycle pathway. It indicated that oil phase separated from O/W emulsion mainly due to de-emulsification through oil-wet seams repeatedly.

4. Conclusions

In this work, a novel de-emulsification device RHC-D was designed to enhance de-emulsification effect of O/W emulsion through internal recycle. The de-emulsification ratio of oil phase and aqueous phase for three O/W emulsions which included ethanol/water/2-ethyl-1-hexanol system, water/2-ethyl-1-hexanol/SDS system and ethanol/

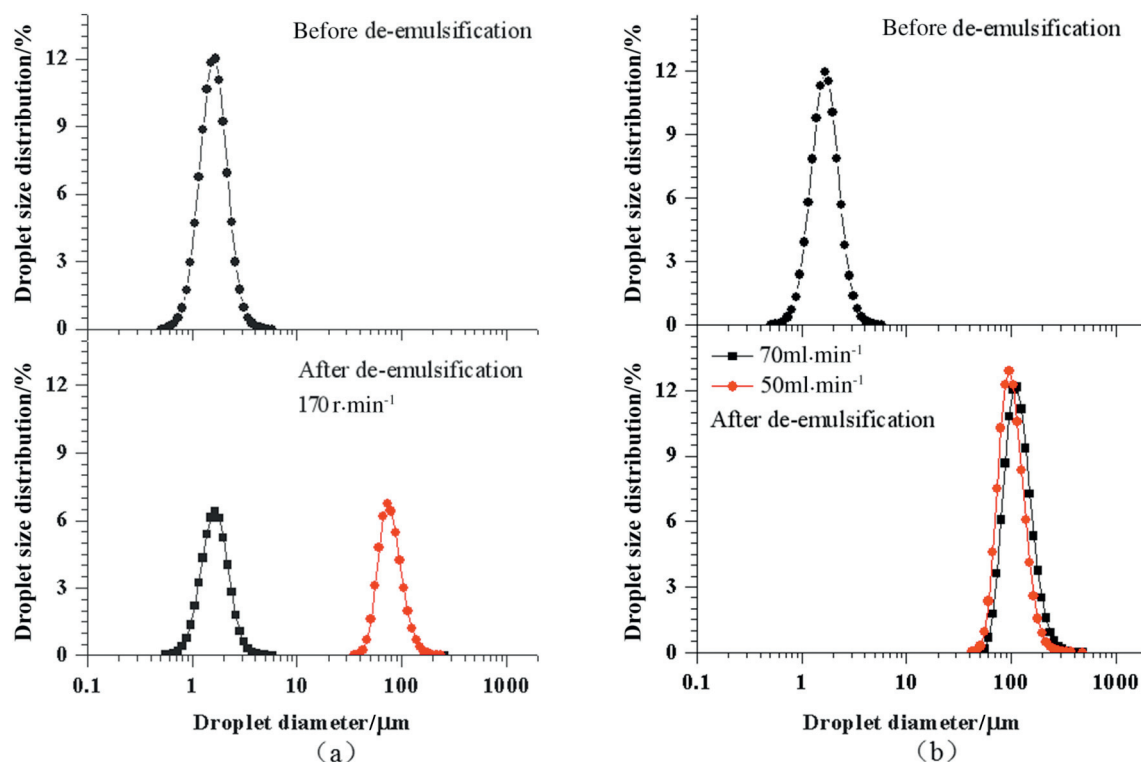


Fig. 9. Droplet size distributions of ethanol/water/2-ethyl-hexanol/SDS before and after de-emulsification in the narrow mixing space combined narrow oil-wet seams repeatedly at the rotation speed of 170 r·min⁻¹ (a), and droplet size distributions of emulsion before and after de-emulsification through narrow oil-wet seams (b).

water/2-ethyl-1-hexanol/SDS system were discussed. Experimental results indicated that de-emulsification effect was obvious in RHC-D at a low rotation speed. For those three emulsion systems, de-emulsification ratio of ethanol/water/2-ethyl-1-hexanol system was best because of worst stability. After de-emulsification, the average droplet size through the seam was larger than that through the seam combined narrow mixing space, which indicated that de-emulsification through the seam was the main reason for the oil phase separated from O/W emulsion, and rotating of disc in the narrow space mainly produced internal circulation. With increase of flow rate of emulsion through the seam, de-emulsification ratio increased due to the increase of pressure and coalescence frequency in the seam. De-emulsification in the seam repeatedly through internal recycle might make continuous extraction for those systems emulsified come true.

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