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Synergistic effects of ultrasonic irradiation and PEG demulsifiers on oil-in-water emulsion separation

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ABSTRACT

A crude oil exporter like Malaysia continues to face challenges related to oil-in-water (O/W) emulsions. These emulsions are typically generated during extraction and transportation processes, leading to difficulties in effective separation. This study investigates the synergistic effects of ultrasonic irradiation and polyethylene glycol (PEG) 1500 demulsifiers on the separation efficiency of model diesel-based O/W emulsions. The emulsions were prepared using Tween 60 as a stabiliser, followed by treatments involving PEG 1500 and varying ultrasonic exposure for 5, 10, 30, and 60 s. A series of bottle tests and particle size analyses were conducted to evaluate demulsification performance and droplet size distribution. The findings revealed that the combination of ultrasonic treatment and PEG 1500 effectively enhanced emulsion destabilisation compared to chemical treatment alone. The optimal demulsification efficiency was achieved at 10 s of ultrasonic exposure (Sample D), which produced the highest oil separation percentage and the most balanced phase separation. However, prolonged irradiation beyond 30 s led to over-dispersion, droplet fragmentation, and re-emulsification thereby reducing separation efficiency. The present particle size distribution results supported these finding where excessive ultrasonic irradiation yielded smaller and overly stable droplets resistant to coalescence.

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1. INTRODUCTION

Malaysia is a net global exporter country and is recognised for high-quality crude oils. The latest export data from the Observatory of Economic Complexity (2024) revealed that Malaysia exported crude oil valued approximately \$27.8 billion to China, Australia, and Thailand. So far, the global demand for crude oil was recorded at 102.21 million barrels per day (mb/d) in 2023 (Statistica Research Department, 2024). However, this demand is projected to reach 105.7 mb/d by 2028 (U. S. Energy Information Administration, 2023). Hence, more than 25 wells are expected to be drilled per year in peninsular Malaysia, Sabah and Sarawak by Petronas to attain the target (Petronas, 2024). This fact implies the research related to crude oil remains necessary and is worth conducting.

In principle, crude oil naturally exists as a liquid in underground reservoirs. However, the water contacted in reservoir with intense agitation during extraction and transportation (Fuentes et al., 2022) prone to oil-in-water (O/W) emulsions formation (Abed et al., 2019; Mat-Shayuti et al., 2020). The O/W would disperse in fine droplets within the oil to create a stable emulsion (Abdulredha et al., 2020). Conceptually, an emulsion is a mixture of two immiscible liquid phases whereas one liquid appears as droplets within the other (Fajun et al., 2020). This droplet phenomenon is undesirable due to its extreme stability obstacle. As such, this stability leads to an increase in density from 800 kg/m^3 to as high as 1030 kg/m^3 and elevates its viscosity from 560 mPa·s to 1000 mPa·s (Fingas et al., 1993). This inevitably cause the mixture to be more challenging to handle, transport, and refine (Dong et al., 2022; Yonguep et al., 2022). The pipelines also more susceptible to corrode, poisoning and exposed to overhead of distillation column (Rajamanickam, 2021; Alao et al., 2021). Therefore, demulsification methods provide a potential solution to this issue.

Traditional demulsification technique such as mechanical separation (e.g.: centrifugal, membrane, filtration and gravity settling), biological (e.g: microbial metabolism) and thermal processing (e.g.: heating and microwave) are commonly employed (Topilnytskyy et al., 2024; Tang et al., 2024). It is used to disrupt the interfacial film stabilising the emulsified droplets and promote phase separation. However, those methods often face limitations when dealing with highly stable emulsions. So, chemical demulsifiers such as polyethylene glycol (PEG) is preferred option to destabilise emulsions by destroying the hydrophobic of emulsification and allowing coalesce of water droplet (Hamadi, 2010). However, the use of chemical demulsifiers alone can be insufficient for breaking down complex emulsions. It also poses environmental concerns due to high dosage requirements and potential toxicity (Faizullayev et al., 2022). Therefore, combination treatment such as ultrasonication technique is warranted.

Ultrasonication has emerged as a promising alternative or complementary method for demulsification. The application of ultrasonic waves generates intense localised shear forces and cavitation effects (Khajehesamedini et al., 2018). The interfacial film is disrupted to promote more droplet coalescence and enhance separation efficiency. While many authors have explored the use of ultrasonic treatment as a standalone technique (Volkova & Yudina, 2020; Xu et al., 2019), the synergistic effects of combining ultrasonication with PEG demulsifiers for O/W emulsion separation remain underexplored. So, it is hypothesised that the ultrasonic fields will facilitate the homogeneous dispersion of PEG within the emulsion, thereby better demulsification efficiency and smaller oil droplet size will be resulted. Therefore, gear up the present study.

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Crude oil is an unrefined natural resource primarily utilised for producing petrochemicals and fuels such as gasoline, jet fuel, and lubricants through refining processes (Al-Samhan et al., 2022). Among its derivatives, diesel oil is a major refined product widely used as a fuel in transportation, power generation, and industrial applications. In terms of emulsion composition, crude oil emulsions typically consist of crude oil (dispersed phase) and produced water (continuous phase) that stabilised by naturally occurring surface-active agents such as asphaltenes, resins, waxes, and fine solids. Diesel oil is always an option as a model crude oil (Yonguep & Chowdhury, 2021) due to its stable composition and reproducibility for controlled laboratory testing. Chevron (2007) also indicated diesel oil exhibits comparable hydrocarbon composition, physical characteristics, and phase behavior in emulsions to those of crude oil. In the present study, the artificial crude O/W emulsion was prepared using diesel oil (dispersed phase), deionised water (continuous phase), and Tween 60 (non-ionic surfactant) as a stabilising agent. By doing so, this research aims to investigate the synergistic effects of ultrasonic irradiation and PEG demulsifiers on O/W emulsion separation. The separation efficiency and droplet size distribution under varying ultrasonic exposure durations will also be investigated.

2. METHODOLOGY

2.1 Materials

The materials used in this study included diesel oil (PETRONAS) and deionised water. For emulsion preparation, the Tween 60 and polyethylene glycol 1500 (PEG 1500) were used for stabiliser and chemical demulsifier, respectively. All mentioned chemicals were of analytical grade and purchased from Sigma-Aldrich and used without further purification.

2.2 Emulsion preparation

Fig. 1 (left) shows the preparation of an O/W emulsion conducted in a 400 ml glass beaker. The procedure was adapted from Adeyanju & Oyekunle (2018), with modifications to simplify the process by eliminating the need for electrical equipment. O/W ratio of 2:7 was prepared by mixing approximately 60 ml of diesel oil with 30 ml of Tween 60 (non-ionic surfactant) as tabulated in Table 1. The mixture was then manually stirred using a spatula for 5 min. This manual stirring was to minimise foam formation and discard the droplet re-coalescence problem that is synonym with homogeniser usage (Sultan, 2019). Following the initial mixing, 210 ml of deionised water was added followed by an additional 5 min of manual stirring. This step allows homogeneous dispersion and promotes emulsion stability. Fig. 1 (right) shows the final emulsion mixture transferred into a 100 ml beaker and labelled as Sample A.

Table 1. Volume and material for O/W emulsion

Material	**Volume (ml)**
Diesel oil	60
Tween 60	30
Deionised water	210

Source: Author’s own data

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Fig. 1. (left) Preparation of O/W emulsion; (right) O/W emulsified Sample A

Source: Author's own data

2.3 Chemical demulsification treatment

After the transfer process has been completed, the remaining emulsion mixture of 250 ml was added with polyethylene glycol 1500 (PEG 1500) at 59 ppm concentration. This concentration was identified as the optimum for demulsification process based on preliminary investigations. The mixture was then stirred for 5 min using a spatula. Afterward, the homogenised demulsified mixture of 50 ml was dispensed into a 100 ml beaker and labelled Sample B.

2.4 Ultrasonic treatment

A total volume of 200 ml samples was collected after the chemical demulsifying process. Fig. 2 (left) shows the volume was equally distributed into four 100 ml beakers and labelled as Sample C, D, E, and F. Subsequently, an ultrasonic probe (DW SD20, 1200H) operated at a 20 kHz (Gao et al., 2024) was used for the Sample C treatment. During the process, the ultrasonic transducer was positioned 1 cm from the sample interface for 5 s as shown in Fig. 2 (right). This procedure was repeated for Sample D, Sample E, and Sample F with ultrasonic treatment with durations of 10 s, 30 s, and 60 s, respectively. After treatment, both irradiated and non-irradiated samples were subjected to a bottle test and particle size analysis. Table 2 provides details of the samples and their respective treatments.

2.5 Bottle test

An approximately 47.5 ml sample was poured into 50 ml capacity centrifuge tube. Each tube was immediately placed inside a 60 °C mechanical oven to prevent wax crystallisation (Alpandi et al., 2022). Phase separation was recorded based on the volume of oil layer, emulsion layer and water layer as shown in Appendix A. The layers were examined periodically in 0, 5, 15, 30, 60, 120, 240, 2880, 4320, 5760, 7200, 8640, 11520, 12960, and 18720 min. This interval was consistent with Khaw (2015). Thereafter, the oil separation percentage was calculated using Eq. (1) expressed as:

$$\frac{\text{The volume of diesel oil separated (ml)}}{\text{Initial volume of diesel oil in the emulsion(ml)}}\ x\ 100\% \tag{1}$$

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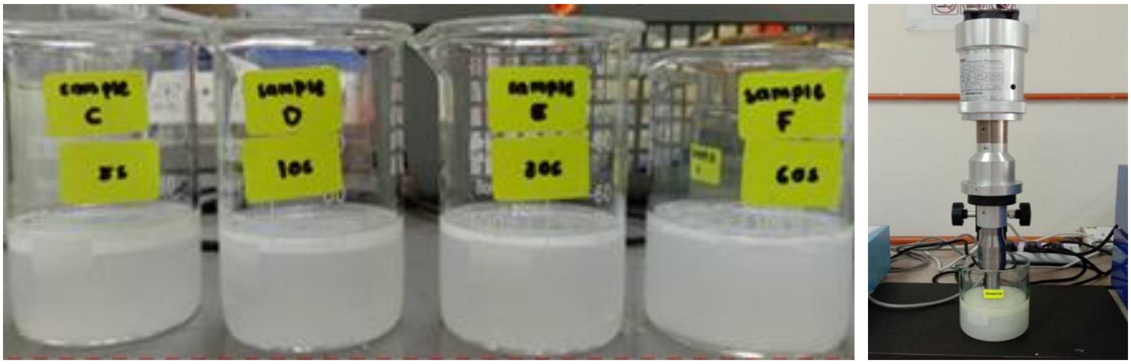

Fig. 2. (left) Preparation of Sample C, D, E and F for ultrasonic treatment; (right) ultrasonic irradiation process

Source: Author's own data

Table 2. Design mix of oil in water emulsion separation

Sample	Treatment
A	Control sample (no treatment)
B	+ PEG 1500
C	+ PEG 1500 + 5 s ultrasonic irradiation
D	+ PEG 1500 + 10 s ultrasonic irradiation
E	+ PEG 1500 + 30 s ultrasonic irradiation
F	+ PEG 1500 + 60 s ultrasonic irradiation

Source: Author's own data

2.6 Particle size analyser

The droplet size distribution was analysed using laser diffraction technique (Atascientific, 2025; Ming et al., 2021) using Malvern Mastersizer 2000. Initially, the instrument and software were activated and keep running until their system was stabilised. A baseline was measured to ensure stable laser intensity and a clean optical path before analysis (Malvern Panalytical, 2023). Subsequently, three to five drops of Sample A were diluted with deionised water in the Mastersizer beaker. The dilution was performed until the obscuring level reached the optimal range of 10% and 20%. This was done to ensure accurate light scattering and reliable droplet size distribution result. After recorded the reading, the cell was rinsed thrice with tap water, followed by triple rinses with deionised water to prevent cross contamination among the samples. The procedure was repeated for Sample B until Sample F.

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3. RESULTS AND DISCUSSION

3.1 Effect of thickness layer in bottle test

The thickness layer is crucial parameter in bottle test as it directly imparts the progression of separation phase within the emulsion. Samples with different treatment influence the coalescence rate and interfacial film rupture thereby affecting the separation thickness (Simonsen et al., 2023). A thinner emulsion layer with thicker distinct oil and water layer indicating more effective demulsification and faster separation phase. Fig. 3 shows the thickness layer result obtained without treatment (Sample A), with PEG 1500 alone (Sample B), and with combination of ultrasonic irradiation and PEG 1500 (Samples C to F).

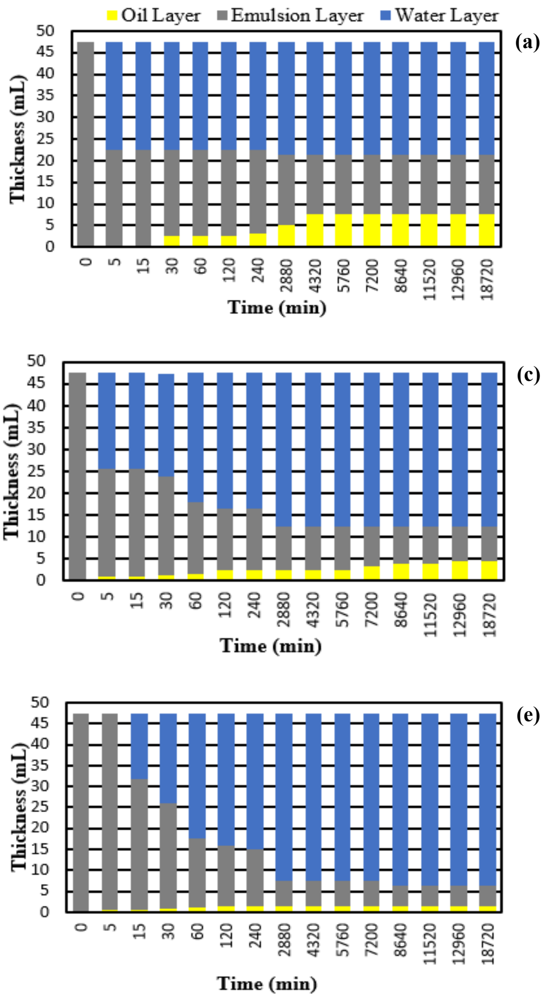

Fig. 3. Layer separation results for (a) Sample A, (b) Sample B, (c) Sample C, (d) Sample D, (e) Sample E, and (f) Sample F

Source: Author’s own data

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The effect of different treatments from 0 min to 18720 min on the separation profile was observed. It appears that all samples accelerated initial coalescence as fast as 5 min except sample F (Fig. 3(f)) and control (Fig. 3(a)) on 15 and 30 min, respectively. The slower separation of control is attributed to the reliance solely on gravitational settling. In other words, the absence of chemical or mechanical assistance interrupted the interfacial stability process (Yonguep et al., 2022; Sjöblom et al., 2021). Meanwhile, the delayed of Sample F treated with the longest irradiation duration of 60 s happened due to intense cavitation and shear forces to broke up the oil droplets (Gao et al., 2024). The smaller droplets sizes exhibited lower buoyancy and higher surface area making them more resistance to coalesce so called over-dispersion and re-emulsification (Pedroti et al., 2018). Therefore, the demulsification should be conducted below this cavitation threshold to discard the drawback.

Fig. 3(b) shows as the duration proceeds, the oil emulsion layer thickness gradually decreases with the increases of oil layer in Sample B. The emulsion layer and oil layer charted 47.5 ml to 14 ml and 0 ml to 7.5 ml after 4320 min respectively. This indicates the chemical demulsifier has supplemented continual effect towards weakening the interfacial film between the oil and water phases. In this study, PEG 1500 served as the chemical demulsifying agent. It has a high hydrophilic–lipophilic balance (HLB) value of above 16. The HLB is an empirical scale used to express the balance between the surface-active molecule hydrophilic and lipophilic group (Raya et al., 2020; Rondón et al., 2006). Emulsifiers with HLB values ranging from 8 to 18 are the most suitable for O/W emulsion (Esfandiarian 2023; Nuraini et al, 2011). However, the oil layer remained thin (5 ml) while the emulsion layer was thick (7.5 ml) after the last duration of 18720 min observation. This indicated the separation was incomplete and mechanical force is warranted.

Fig. 3(c) to Fig. 3(f) shows the emulsion layer of ultrasonic irradiation with demulsifier treated sample was decreased after 18720 min. Samples C, D, E, and F recorded 8.0, 7.5, 5.0, and 4.0 ml, respectively. In contrast, the water layer volume increased over time, reaching 35.0, 37.5, 41.0, and 42.5 ml for Samples C, D, E, and F, respectively. These results indicate that the synergistic of the mechanical and chemical demulsifier is effective in maintaining the emulsion destabilisation. Hence, facilitate the separation of oil and water. Nevertheless, the oil layer volume decreased with increasing ultrasonic irradiation duration. The volumes were 4.5, 2.6, 1.5, and 1.0 ml for Samples C, D, E, and F, respectively. This result indicate prolonged ultrasonic treatment may induce over-dispersion, thereby reducing oil layer volume. Hence, the optimal duration must be selected to balance efficient emulsion destabilisation speed with maximal oil layer. In this case the Sample D with 10 s irradiation period was chosen.

Percentage of oil separation

The percentage oil separation quantifies how effectively the chemical demulsifier and ultrasonic treatment separate oil from the emulsion. Fig. 4 depicts the fraction oil separation results over time. Based on the findings, the control sample exhibited a relatively higher initial percentage of oil separation compared to the other samples. It should be noted that even the percentage was higher the separation remained incomplete. This statement supported the thickest emulsion layer found in Figure 3(a). Meanwhile, Sample B that was treated solely with PEG 1500 demonstrated slightly improved. The oil separation reaches approximately 8% over time but the emulsion is restricted to fully destabilise. This statement was supported by the previous result in Fig.3(b) indicating the emulsion layer still thicker even the last experimental duration.

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However, the combination of demulsifier and various ultrasonic irradiation timing showed varying levels of separation. For instance, moderate separation of oil separation of 7.5% was achieved when the sample was treated with 5 s of ultrasonic exposure. Meanwhile the highest stabilising separation efficiency of 4% was recorded for Sample D. This sample was treated with 10 s ultrasonic treatment. Notably, this optimal duration allowed a great synergistic effect to disrupt the emulsion interfacial tension as well as promoting droplet coalescence and phase separation. However, extended exposure durations over 30 s decline the oil separation percentage. The Samples E (30 s) and F (60 s) recorded resulted the oil separation stabilising at 3% and 1.7%, respectively. This decline can be attributed to excessive ultrasonic energy, which caused droplet fragmentation and re-stabilisation, creating a more stable emulsion that resisted separation.

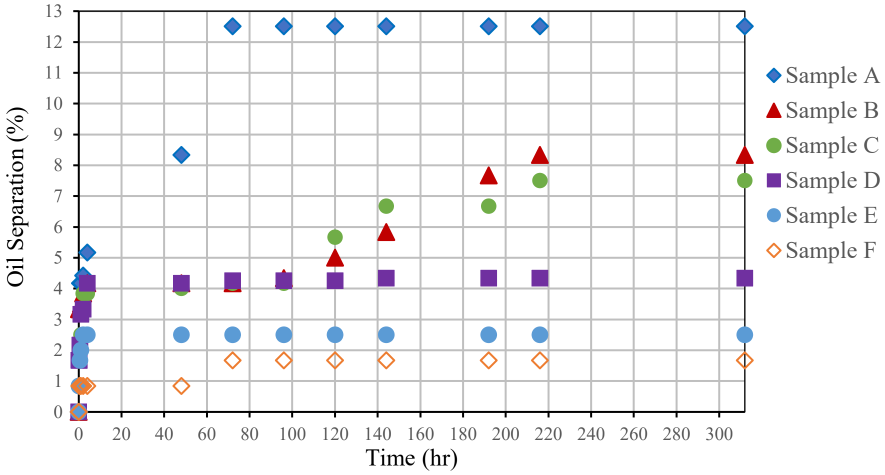

Fig. 4. The oil separation percentage for different samples

Source: Author’s own data

3.2 Effect of particle size distribution (PSD)

The particle size distribution (PSD) is a crucial parameter as it directly impacts the separation behaviour of oil in water emulsion (Upadhyay et al., 2024). From Table 3, it can be asserted that the control sample recorded the biggest particle oil droplet sizes. The size charted 30.939 µm, 63.624 µm,140.176 µm taken at d(0.1), d(0.5) and d(0.9), respectively. This size can be attributed to the fact that poorest droplets breakdown. However, a moderate particle size oil droplet sizes percentage of 6.26%, 24.87% and 44.63% corresponding for d(0.1), d(0.5) and d(0.9) value were observed when the demulsifying treatment alone (Sample B). Moreover, the introduction of ultrasonic irradiation (Samples C until F) facilitated a progressive decrease in particle oil droplet size. Among the samples, sample D recorded the smallest median size of d(0.5) and lower size d(0.9). Apparently, the smallest oil droplet size was observed in Sample F (60s ultrasonic exposure), where d(0.5) and d(0.9) decreased to 41.906 µm and 67.986 µm, respectively. This finding has witnessed the longer ultrasonic exposure produced the smallest size of droplets. The finer size caused to be too stable, leading to re-emulsification instead of separation. Consequently, only a small oil layer is visible.

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Fig. 5 shows the volume-based particle size distribution curves trend for six samples. The untreated (Sample A) exhibited steeper rising curve compared to the treated samples. The sample volume exceeds 130% at 0.9 µm particle size. This result signifies the oil droplets were separated poorly and clumped together (Otumudia et al., 2023). Meanwhile moderate rising curve was found in Sample B. The reduction in volume that peaking below 100% at 0.9 µm resulted. This indicates that demulsifier alone reduces particle size and improves dispersion. But particles remain relatively large. The combination of demulsifier with different durations of ultrasonic irradiation produces much lower slope trend. The volume reduces to 85%, 75%, 72% and 70% for Samples C, D, E and F, respectively. These samples produce relatively small particle sizes. The smallest size and homogeneous particles distribution recorded by Sample F were treated with 60 s irradiation. However, improvements begin to level off after 30 to 60 s of ultrasound treatment, indicating a threshold wherein beyond this duration yields negative benefit for separation. In this case Sample D treated with 10 min irradiation demonstrated the most effective reduction with an optimal balance between irradiation duration and separation efficiency.

Table 3. Particle size distribution

Sample	**Particle size (µm)**		
	d (0.1)	**d (0.5)**	**d (0.9)**
A	30.939	63.624	140.176
B	29.002	47.800	77.609
C	26.684	46.260	77.454
D	22.489	42.002	77.077
E	24.077	41.799	71.220
F	25.207	41.906	67.986

Source: Author's own data

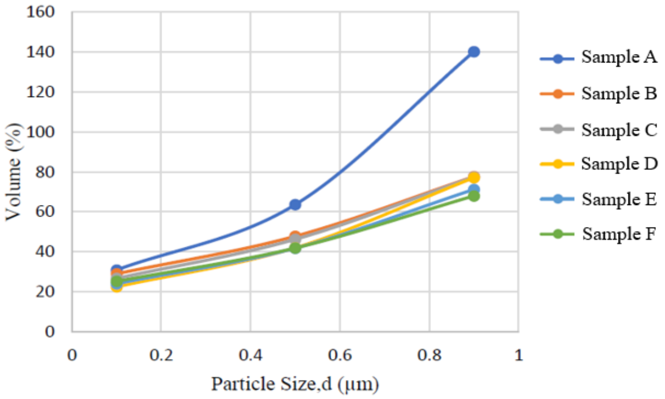

Fig. 5. Volume-based particle size distribution result for various samples

Source: Author's own data

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4. CONCLUSION

The incorporation of polyethylene glycol (PEG 1500) and ultrasonic irradiation provide excellent synergic effect on the separation efficiency of oil-in-water emulsions. Although PEG 1500 alone improvised the separation compared to the untreated (control) but the integration of ultrasonic treatment significantly enhanced demulsification. The optimal separation was achieved with 10 s of ultrasonic exposure resulting in better oil separation and minimal residual emulsion. However, prolonged ultrasonic treatments (30 and 60 s) led to poor efficiency due to droplet fragmentation. Owing to these findings, this study offers valuable insights into optimising the combined chemical with ultrasonic demulsification under controlled laboratory conditions that mimic real crude oil separation processes.

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CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits, commercial or financial conflicts and declare the absence of conflicting interests with the funders.

AUTHORS’ CONTRIBUTIONS

Nur Ainin Sofiya Mat Yajid: Methodology, formal analysis, investigation and writing-original draft; **Muhammad Shafiq Mat Shayuti**: Supervision; **Siti Aisyah Ghazali**: writing-original draft, supervision; **Tengku Amran Tengku Mohd**: Supervision; **‘Azzah Nazihah Che Abdul Rahim**: Supervision; **Suriatie Mat Yusuf**: Supervision.

DATA AVAILABILITY

Data available on request from the authors.

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SUPPLEMENTARY MATERIALS

Appendix A

A. Design mix of oil in water emulsion separation

	Thickness (ml)					
	Sample A			**Sample B**		
Time (min)	**Oil layer**	**Emulsion layer**	**Water layer**	**Oil layer**	**Emulsion layer**	**Water layer**
0	0	47.5	0	0	47.5	0
5	0	22.5	25	2	14	31.5
15	0	22.5	25	2	14	31.5
30	2.5	20	25	2	13.5	32
60	2.5	20	25	2.1	12.9	32.5
120	2.65	19.85	25	2.3	10.2	35
240	3.1	19.4	25	2.5	10	35
2880	5	16.5	26	2.5	10	35
4320	7.5	14	26	2.5	10	35
5760	7.5	14	26	2.6	9.9	35
7200	7.5	14	26	3	9.5	35
8640	7.5	14	26	3.5	9	35
11520	7.5	14	26	4.6	7.9	35
12960	7.5	14	26	5	7.5	35
18720	7.5	14	26	5	7.5	35

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