Optimising the chemical demulsification of water-in-crude oil emulsion using the Taguchi method

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Abstract. This study examines the effect of important factors such as the temperature, pH, freshwater, concentration, and type of demulsifier on chemical demulsification (CD) using the bottle test method. Three Iraqi heavy crude oils, each with different properties, were tested. Each of these samples contained different concentrations of salt solution, which need to be removed to improve the properties of the crude oil. Five different types of commercial chemical demulsifiers, namely oxyalkylated copolyether (D1), Polyethylene glycol octaacyl ether (D2), polyether butoxy ethanol (D3), Ethylene-propylene copolymer (D4), and Cumene 1,2,4-trimethylbenzen (D5), were tested. The Taguchi method was used to design the experiments to obtain optimum separation. The data analysis was carried out using signal-to-noise (S/N) ratio and analysis of variance (ANOVA). The results of the best combination test show that the maximum removal efficiency of CD for the three samples are 94.2%, 95%, and 92%, respectively. In addition, there was a noticeable improvement in the original value of the API from 23.2 to 28.4, 14 to 20.9, and 4.6 to 24.1, respectively. The work plan and the results of this work could be used to improve crude oil processing in the Iraqi oil industry.

1. Introduction
Reservoir pressure drop is one of the most complicated problems in the oil industry, and it requires the use of artificial lift systems for drifting the crude oil from oil wells [1]. Artificial lift systems use electrical submersible pumping (ESP), steam injection, and chemical flooding [2,3]. The use of these techniques results in two main problems: i) increased water and salt content and ii) increased total cost of production and treatment of the oil barrel, with a cost of $10.57 per barrel in Iraq for 2019 [4][5][6]. Flow rates in the production process and continuous shaking form emulsion [7]. Hence, the presence of natural surface active agents (surfactants), such as asphaltenes, resins, organic acid, salts, and clay, stabilise the water droplets in a continuous oil phase, water-in-oil (W/O) emulsion or stabilise the oil droplets in a continuous water phase, oil-in-water (O/W) emulsion [8]. The emulsion is undesirable in the oil industry for the reasons of corrosion damage, increased viscosity, difficulties in oil transporting, decreased American Petroleum Institute (API), and catalyst poisoning [9,10,11]. Therefore, for the operational and economic goals of increasing pipeline usage and decreasing corrosion problems before the refining process, it is important to completely separate water from the oil [12,13]. The separation of water depends on destroying the interfacial film and draining the water or oil droplets; this process is called demulsification [14,15]. The demulsification process includes many types of methods, such as mechanical, electrical, and chemical [16,17]. The chemical demulsification (CD) method is a widespread method, which is indispensable even in combination with other methods, and relies on the use of chemical demulsifiers [18,19].
Chemical demulsifiers are amphiphilic surfactant polymers with interfacial activity more than the indigenous surfactants (natural surfactants) [20,21]. The amphiphilic characteristic enables them to adsorb at the oil/water or water/oil interface and destabilise emulsified droplets [22,23]. The stabilisation of natural surfactants depends on a reduction of interfacial tension (IFT) and the enhancement of emulsification and dispersion droplets [24]. The mechanism of demulsification depends on the substitution of natural surfactants at the interface and changes the gradients of IFT due to the Marangoni effect to promote the separation of oil and water [21,25,26].

There are many studies that have examined the effect of numerous factors on the demulsification process. Biniaz et al. [27] studied the effect of the temperature, pH, freshwater, and concentration of demulsifiers by using response surface methodology (RSM) that depended on central composite design (CCD) to obtain the optimum factor for different types of demulsifiers. The results show that the optimum value of a demulsifier depends on the type of demulsifier; however, in both types, efficiency declined after the optimum value. Freshwater had either a positive or negative effect depending on the solubility of each demulsifier. The demulsification process is proportional to increasing temperature and its effectiveness at the neutral range of pH. Roshan et al. [28] investigated the effect of temperature, demulsifier concentration, pH, and freshwater ratio on dehydration efficiency by using RSM. The results proved that high temperatures and freshwater contribute to increased demulsification efficiency, while the effects of pH and demulsifier centration are dependent on the type of demulsifier. Roostaie et al. [29] examined the effect of treating temperature, agent concentration and type, freshwater dose, and pH on separation efficiency by applying the central composite design (CCD), which is useful in RSM. The results indicate that separation efficiency is proportional to temperature, agent concentration, and freshwater dose, while the optimum value of pH is at a neutral state.

Based on the literature, it can be concluded that the temperature, pH, freshwater, concentration, and type of demulsifier have a critical effect on CD. However, unlike in previous studies, in this study, the Taguchi method was used to design the runs of experiments and to analyse the data. The Taguchi method is a powerful statistical tool for forming the best combination of the most influential factors with minimum time and trials then guessed the contribution for each factor to reach the optimum case [30]. Taguchi can be applied to signal-to-noise (S/N) ratio to predict the performance variability and to analysis of variance (ANOVA) to examine the contribution of each factor [31]. Five different types of demulsifiers were selected as a factor in the work plan to improve CD efficiency at the lowest possible cost and improve the characteristics of crude oil to increase the selling price. Five chemical demulsifiers, namely oxyalkylated copolyether (D1), Polyethylene glycol octaadacyl ether (D2), polyether butoxy ethanol (D3), Ethylene-propylene copolymer (D4), and Cumene 1,2,4-trimethylbenzen (D5), were chosen to be compared using the bottle test method.

2. Experimental methodology and data analysis

2.1. Experimental methodology

In this study, the plan of the experiments was designed based on the Taguchi method, which can be used to determine the optimal combination of factors to increase demulsification efficiency and to evaluate the effect of each individual factor. The steps of conducting the experimental evaluation were i) choosing the CD factors and their levels, ii) designing the experimental runs by using orthogonal array (OA), iii) testing the runs and recording responses, iv) analysing results by using S/N ratio to obtain the optimum levels for each factor and ANOVA to predict the contribution factors, and v) retesting the optimum case to confirm the results.

2.2. The control factors and factor levels

There are five controllable parameters affecting the efficiency of the CD process. The first parameter is the temperature of the crude oil, which must remain constant during the exam time, thus achieved by using a water bath. The water bath was used to regulate the temperature of the samples (model HH-S2/500W, operation range from room temperature to 99 °C). Second, the pH of the crude oil was controlled using HCl and NaOH depending on pH meter ( Hanna: Model -HI98107, range 0 to 14 pH, 0 to 50 °C) [1–3]. Third, freshwater was used to decrease the concentration of salt content in the crude oil
Pipette HO-1630A (10ml) was used to add the exact amount of water. Fourth, the demulsifier concentration was controlled by a micropipette. The type of micropipette was Biozek mechanical pipette/A3024 (range 5–50 µl). Fifth, five types of commercial demulsifiers with unknown chemical composition were used to study their effects on the process of separating water from crude oil. The types of demulsifiers are referred to by their abbreviations (i.e. D1, D2, D3, D4, and D5). The control factors and their levels are shown in Table 1.

**Table 1.** Control factors and factor levels used in Taguchi experimental design.

| Control Factors               | Code | Levels |
|------------------------------|------|--------|
| Temperature °C               | A    | 30     |
|                              |      | 40     |
|                              |      | 50     |
|                              |      | 60     |
|                              |      | 70     |
| pH value                     | B    | 3      |
|                              |      | 5      |
|                              |      | 7      |
|                              |      | 9      |
|                              |      | 11     |
| Freshwater (%)               | C    | 2      |
|                              |      | 4      |
|                              |      | 6      |
|                              |      | 8      |
|                              |      | 10     |
| Concentration of demulsifier (ppm) | D   | 50     |
|                              |      | 100    |
|                              |      | 150    |
|                              |      | 200    |
|                              |      | 250    |
| Type of demulsifier          | E    | D1     |
|                              |      | D2     |
|                              |      | D3     |
|                              |      | D4     |
|                              |      | D5     |

The temperature of the samples (i.e. 30, 40, 50, 60, and 70 °C) in the water bath were selected to include the widest range for testing and also to ensure that light hydrocarbons of crude oil would not be depleted during the treatment processes at high temperatures. Kokal [9] showed that the temperature of treatment must remain as low as possible, because high temperatures can cause: i) increasing the treatment cost, ii) a drop in American Petroleum Institute (API), or iii) scale deposition and corrosion of equipment and pipes. The range of pH included the transfer from acidity to the base case. The freshwater ratio was dependent on salt content. This ratio range was 2–10% in the crude oil sample [32]. Lastly, the concentration and type of demulsifiers were dependent on trial and error [19].

The standard of Taguchi design OA was L25 for Taguchi experimental plan. In this study, the treatment processes take place on crude oil like operational conditions in real plant, and the time spent in each stage was simulated by the bottle test method. The Taguchi experimental plan is shown in Table 2.

**Table 2.** Taguchi experimental plan.

| Run | A  | B  | C  | D  | E   |
|-----|----|----|----|----|-----|
| 1   | 30 | 3  | 2  | 50 | D1  |
| 2   | 40 | 5  | 4  | 100| D2  |
| 3   | 50 | 7  | 6  | 150| D3  |
| 4   | 60 | 9  | 8  | 200| D4  |
| 5   | 70 | 11 | 10 | 250| D5  |
| 6   | 30 | 5  | 6  | 200| D5  |
| 7   | 40 | 7  | 8  | 250| D1  |
| 8   | 50 | 9  | 10 | 50 | D2  |
| 9   | 60 | 11 | 2  | 100| D3  |
| 10  | 70 | 3  | 4  | 150| D4  |
2.3. Materials

Three Iraqi heavy crude oil samples with different water and salt contents and other physical properties were supplied from the Missan oil field. Table 3 shows the characteristics of these samples. The five types of commercial chemical demulsifiers (oxyalkylated copolyether (D1), Polyethylene glycol octaadacyl ether (D2), polyether butoxy ethanol (D3), Ethylene-propylene copolymer (D4), and Cumene 1,2,4-trimethylbenzen (D5)) that were used in this study were provided by the Missan oil company. Sodium hydroxide (NaOH) and hydrochloric acid (HCl) and the organic solvents such as ethanol, acetone, toluene, and xylene were provided by the Chem-Lab NV company.

Table 3. The characteristics of the Iraqi crude oil samples.

| Property                          | Standard | Sample1 | Sample2 | Sample3 |
|----------------------------------|----------|---------|---------|---------|
| Specific gravity at 15 °C        | IP 235/69| 0.9148  | 0.972   | 1.04    |
| API gravity at 15 °C             | IP 235/69| 23.2    | 14      | 4.6     |
| Kinematic viscosity at 40 °C (cSt)| ASTM D445| 24.51   | 86.25   | 863.01  |
| Asphaltenes (wt%)                | IP 143   | 6.2     | 3.28    | 3.86    |
| Sulphur content (wt%)            | ASTM D4294| 3.278   | 1.918   | 1.342   |
| Salt content (mg/L)              | IP 77    | 9425    | 87620   | 184800  |
| BS&W (V%)                        | ASTM D4007| 2       | 30      | 60      |
| pH                               | PH meter | 5.4     | 5.1     | 5.2     |
2.4. Data analysis method

Water separated during the settling time was recorded after 12 hours. The test tube was a centrifuge tube/T.CO-G. R ADARSH. The total amount of water in the sample of crude oil \( W_w \) was produced from i) the amount of water in the crude oil (water content \( W_c \)) and ii) fresh water \( W_f \) used to wash the crude oil and decrease the concentration of salt in the crude oil as much as possible. In this study, the efficiency of chemical demulsification (i.e. responses) is defined as the amount of water that is separated \( W_w \) compared to the total water \( W_T \) in the sample. The efficiency is calculated by using Equation 1.

\[
\text{Efficiency\%} = \frac{W_w}{W_T} \times 100 = \frac{W_w}{W_c + W_f} \times 100
\]  

The effect of studied factors and their levels on CD efficiency was predicted by using S/N ratio. The-larger-the-better of S/N ratio was applied to increase CD efficiency [31]. SNL is shown in Equation 2.

\[
\text{SNL} = -10 \log \left( \frac{1}{n} \sum_{i=1}^{n} \frac{1}{Y_i^2} \right)
\]

Where \( n \) is the number of runs and \( Y_i \) is the response of each experiment at the \( i^{th} \) run. ANOVA is a statistical tool used to find the significance and contribution of each factor on demulsification efficiency [30].

2.5. Prepare the samples and bottle test method

To save time and reduce effort, we examined every five runs at one time and each sample according to the specific experimental plan. For each run, the amount of crude oil tested was 100ml. Hence, 25 runs had to facilitate 2.5l at least. The total amount of crude oil provided was 5l. The total amount of sample was divided into five parts, each part with a volume of 1l to ensure that there was no shortage during the test period and that there was an additional amount of crude oil to examine the optimum case. As mentioned earlier, HCl and NaOH were used to regulate the pH value of the sample for each one of the five parts (i.e. 3, 5, 7, 9, and 11). According to the work plan, the temperature levels are 30, 40, 50, 60, and 70 °C; therefore, we were able to test all five samples simultaneously as follows: i) preparing the five centrifuge tubes so that they were clean and dry; ii) adding 100ml of sample crude oil for each tube with different pH values; iii) heating the samples for 5 minutes; iv) adding the specific demulsifier dose by using the micropipette; v) shaking the tube to reach homogeneity; vi) adding the specific freshwater by using the pipette; vii) shaking the tube to reach homogeneity again; viii) putting the samples in the water bath; ix) taking out the samples from the water bath after 12 hours and recording the amount of water separated; and x) washing the tubes to use them again.

3. Result and discussion

3.1. Effect of the five operating factors on CD efficiency

Figure 1 shows the change in CD efficiency during the experimental runs at 12 hours. The change in CD efficiency is significant. The highest efficiencies were 87.5% (v/v) in Run 20 of sample 1, 90% (v/v) in Run 19 of sample 2, and 90% (v/v) in Run 11 of sample 3. The lowest efficiencies were 0% (v/v) in Runs 5, 9, 12, 13, 16, 17, and 21 of sample 1, 0% (v/v) in Runs 5, 6, 9, 12, 13, 16, 17, and 21 of sample 2, and 0% (v/v) in Runs 5, 9, 12, 13, 17, and 21 of sample 3.

The aim of this study is to evaluate the effect of each control factor and its contribution to the CD process. Hence, analysis of variance (ANOVA) was used to explain the effect of the operating parameters on CD. The contribution ratio means the amount that the factor influences the CD process within the specified level in Table 2. Figure 2 shows the contribution of the five control factors that were mentioned in Table 2. The first contribution values of temperature were 5% of sample 1, 19% of sample 2, and 14% of sample 3. The second contribution values of pH were 64.96% of sample 1, 36% of sample 2, and 57% of sample 3. The third contribution values of fresh water were 3.46% of sample 1, 5% of sample 2, and 16% of sample 3.
Figure 1. Efficiency of 25 experimental runs: a) sample 1, b) sample 2, and c) sample 3.
Figure 2. Contribution factors on the CD process: a) sample 1, b) sample 2, and c) sample 3.

The fourth contribution values of concentration of demulsifiers were 3.46% of sample 1, 5% of sample 2, and 16% of sample 3. The fifth contribution values of type of demulsifiers were 20.77% of sample 1, 35% of sample 2, and 9% of sample 3.
The results show that pH has the most significant influence on CD, because the stability strength of the emulsion is dependent on surfactant polarity. The weakest impact factor on CD is the concentration of the demulsifier. This leads to the conclusion that the performance of the demulsifier does not depend on the concentration in the levels chosen in Table 2. This opens the way for future research into the effect of demulsifiers in wider levels. The contribution of factors on samples 2 and 3 (i.e. type of demulsifier, temperature, and freshwater) is arranged in descending order. In contrast, the contribution of sample 1 is freshwater, temperature, and type of demulsifier. The difference between the contribution ratios is due to the difference in the sample content of water, asphaltenes, and sulphur, as mentioned in Table 2.

S/N ratio showed the optimum levels in the CD process and whether the factors were directed towards enhancing or discouraging the CD process. Figure 3 illustrates the S/N ratio analysis and shows the optimum case of the factors’ level. First, the optimum case of temperature (A) is 60, 70, and 60 °C for the three samples, respectively. Second, pH (B) is 7 (i.e. neutral) for all samples. Third, freshwater (C) is 10% for all samples. Fourth, concentration of the demulsifier (D) is 200, 50, and 50 ppm, respectively. Fifth, type of demulsifier (E) is D2, D1, and D2, respectively.

Figure 3 also shows that the effect of temperature on CD is reflected in samples 1 and 3; however, in sample 2, the increasing temperature has a positive effect. Although the increasing temperature reduced the viscosity of interfacial film and increased the thermal energy of water droplets, the high temperature inverted the reaction that occurred at the oil–water interface from spontaneous to unspontaneous. This leads to conclusion that the optimum temperature depends on the type of demulsifier.

When pH is neutral, the ionisation of surfactants increases and leads to increased charge density so that the repulsion between molecules is enhanced to produce an increase in the CD process.

However, the efficiency of the CD process is directly proportional to increases in freshwater percent. The increase in CD efficiency is slow, as shown in Figure 3. As illustrated in Figure 2, the freshwater contribution to the CD process is inversely proportional to water content in the crude oil.

The optimum dose for the concentration of the demulsifier is proportional to the surfactants present in the crude oil samples (i.e. asphaltenes and sulphur), as shown in Table 1. Figure 3E shows that the concentration of the demulsifier must be accurate. Therefore, If the quantity is small, then separation efficiency is low; on the other hand, if it exceeds what is required, this results in so-called overdose.

As shown in Figure 3, the best type of demulsifier was dependent on the nature of the demulsifier and indigenous surfactants (i.e. anion, cation, and non-ionic). Consequently, the effectiveness of the demulsifier agent changed with variation in the type of crude oil, and this depends on several factors: i) the nature of the demulsifier and indigenous surfactants (i.e anion, cation, and non-ionic), ii) solubility of demulsifier, and iii) the dose used. Due to the many parameters affecting the efficiency of the demulsifier, it is difficult to determine which of the previously mentioned factors is more effective, as mentioned in the introduction.
Figure 3. Mean of S/N ratio of the CD process: a) sample 1, b) sample 2, and c) sample 3.
3.2. Retesting the optimum levels
The performance of both the Taguchi and analysis methods were confirmed by testing the optimum levels obtained by using S/N ratio, as shown in Figure 3. Figure 4 illustrates the dehydration ratio and demulsification efficiency, which were calculated by using Equation 1. The results from this equation show that the highest CD efficiencies for the three samples were 94.2%, 95%, and 92%, respectively, as illustrated in the comparison in Figure 5.

It is concluded that the amount of contribution of the Taguchi method to reach the optimum situation with minimal effort and the fastest time and evidence of the method’s success so the demulsification efficiency was increased.

**Figure 4.** Optimum level results: a) sample 1, b) sample 2, and c) sample 3.

**Figure 5.** Comparison between efficiency of the three samples with the optimum test.
3.3. Improving the price of crude oil
As mentioned in Table 3, the API values for the three samples were 23.2, 14, and 4.6, respectively. After completing the optimum levels test, a sample was sucked from the three tubes using a pipette, and then the API value was measured depending on an IP 235/69 stander. The results show that the API values for the three samples were then 28.4, 20.9, and 24.1. Therefore, the essential parameters that decrease the value of the API are water and salt content, which lead to a drop crude oil price.

4. Conclusion
In the present paper, the results show that pH was the most influential factor on CD and that the weakest was the concentration of the demulsifier. The contribution ratio of temperature, freshwater, and type of demulsifier depended on the type of the crude oil sample. The plan of the experiments was designed based on the Taguchi method, which can be used to determine the optimal combination of factors to increase demulsification efficiency and to evaluate the effect of each individual factor. The results were analysed using S/N ratio to obtain the optimum levels for each factor and ANOVA to predict the contribution factors. The optimum levels for the three samples were respectively: i) temperature at 60, 70, and 60 °C; ii) pH at 7 for the three samples; iii) freshwater at 10% for the three samples; iv) demulsifier dose at 200, 50, and 50 ppm; and v) type of demulsifier of D2, D1, and D2. The results of the retest at the optimum levels show improved CD efficiencies for the three samples with efficiencies of 94.2%, 95%, and 92%, respectively. In addition, there was a noticeable improvement in the original values of the API of the three Iraqi heavy crude oils tested in this study by reducing their water and salt content. The API increased from 23.2 to 28.4, 14 to 20.9, and 4.6 to 24.1, respectively.

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