Selection of Optimum Surfactant Formulations with Ultralow Interfacial Tension for Improving the Oil Washing Efficiency

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ABSTRACT: Ultralow oil–water interfacial tension (IFT) has provided an important basis for screening optimum surfactant formulation for improving the oil washing efficiency. Thus, it is of great significance to further investigate the selection method for surfactant systems with ultralow IFT. In this study, a selection of surfactant systems with ultralow IFT was simplified by a method of comparing the equivalent alkane carbon number (EACN) of crude oil with the minimum alkane carbon number \( n_{\text{min}} \) of surfactant mixtures. The results show that the ultralow IFT can be achieved when the \( n_{\text{min}} \) of optimum surfactant formulation is equal to the EACN of crude oil. Meanwhile, the oil washing efficiency experiments show that the oil washing efficiency increases with the decrease of IFT, and the optimum surfactant formulation with ultralow IFT has the highest oil washing efficiency. This study provides a more efficient way for selecting optimum surfactant formulation systems with ultralow IFT for improving the oil washing efficiency.

1. INTRODUCTION

Chemical flooding could enhance residual oil recovery effectively, which has played a crucial role in enhanced oil recovery (EOR) in the last decades. Specifically, for surfactant–polymer (SP) combination flooding (SP flooding), owing to the synergistic effect of the polymer and surfactant, the efficiency sweep and oil washing efficiency have both improved significantly. SP flooding has obvious advantages over polymer flooding due to its low interfacial tension (IFT) and high oil washing efficiency. Compared with alkaline-SP flooding, SP flooding can avoid some problems caused by alkali, such as scaling and difficulty in oil–water separation because of strong emulsification.

In tertiary oil recovery, the oil washing efficiency refers to the ability of chemical agents to remove oil in the formation. The previous studies have shown that the reduction of oil–water IFT by the surfactant system is a key factor in determining the oil washing efficiency. In view of the importance of surfactants in enhancing oil recovery, a lot of research work has been done on the surfactant types, molecular structure, molecular weight and distribution of surfactants, oil characteristics, salt content, co-surfactants, and so forth. The surfactants currently synthesized for SP flooding are mainly alkylaryl sulfonate with side chains and dual tails, gemini surfactant and amphoteric surfactant aboard and amphoteric betaine surfactants, and gemini surfactant and long-chain alkanolamide nonionic surfactant or two surfactant mixtures in China. At present, it is crucial to select a high-performance surfactant system for SP flooding. The polymeric surfactant was considered to be a potential system for SP flooding with the advantage of avoiding negative interaction between polymer and surfactant components. However, it is unable to reach ultralow IFT, which hinders the use of the polymeric surfactant. The research on palmitoylglycolamide by Pei et al. indicated that surfactants with a small cross-sectional area at the interface give excellent performance for lowering IFT. Li et al. indicated that betaine surfactants could achieve ultralow IFT between water and different crude oils only by changing the molecular structure. The alkali-free petroleum sulfonate surfactant studied by Weng et al. showed that the petroleum sulfonates with high molecular weight are able to reduce the IFT more effectively. Surfactants with highly branched large hydrophobes are required to obtain the ultralow IFT for future applications. In most cases, it is hard for an individual surfactant to achieve ultralow IFT but relatively easy for a surfactant mixture. The study showed that with the addition of \( \alpha \)-olefin sulfonates, the oil–water IFT of double-chain single-head nonionic and zwitterionic surfactant binary mixture such as guerbet alcohol ethoxylates and didodecylmethyldihydroxylpropylsulfobetaine (diC12HSB) can reduce to an ultralow value. The mixture of tyloxapol and
cetyltrimethylammonium bromide can achieve ultralow IFT in the presence of hydrolyzed polyacrylamide according to the research of Zhu et al. 20 Guo et al. reported that nonionic surfactant Triton X-100 (TX-100) has the ability to weaken the disadvantage of the interaction between HAPAM and the anionic gemini surfactant. 21

The selection of the surfactant formulations with ultralow IFT is a challenging and time-consuming task because different reservoirs exhibit different conditions such as the oil composition and brine salinity. Some screening methods for an appropriate surfactant in EOR have been proposed in recent years. 22 Babadagli and Boluk introduced a new technique for surfactant selection based on inorganic and organic property values and organic conception diagrams. 23 Khorsandiet al. used a new EOS model to estimate oil recovery accurately in SP flooding. 24 Torreala and Johns proposed a model that could predict two- and three-phase IFTs and phase behavior for changes in composition and hydrophilic—lipophilic deviation. 25 Kothenczet al. developed a new evaluation method with a more accurate relationship with additional oil yield. 26 The equivalent alkane carbon number (EACN) was introduced to characterize crude oil, which corresponds to carbon atom’s number of linear alkane equivalent to the given oil. 27 The optimum parameters, such as solubilization ratio and salinity, are a function of the EACN for a pure alkane/surfactant system. 28 Wan et al. used extended surfactants to determine the EACN of crude oil for designing surfactant flooding. 29

In the past, the selection of surfactant systems with low IFT was mainly aimed at specific oilfields. According to the salinity of formation water and the characteristics of crude oil, the corresponding surfactants were selected and prepared. Due to the different characteristics of each oilfield, the properties of crude oil produced from different layers of the same oilfield are also different. A surfactant system may be suitable for a certain oilfield but not necessarily suitable for another oilfield. Therefore, it is very important to screen and develop suitable surfactant formulations with low IFT for oils with different characteristics. This study provides a simplified method for screening surfactant systems with low IFT. By determining the minimum alkane carbon number \( n_{\text{min}} \) of the surfactant system and the EACN of crude oil, it is easy to find a series of surfactant systems that produce low IFT with the crude oil. This developed method can significantly reduce the cost and time required for the selection of surfactant formulations with ultralow IFT.

2. EXPERIMENTAL SECTION

2.1. Experimental Materials. The crude oil and the formation water used in this study were obtained from the Zhuangxi (ZX) oil reservoir in the Shengli oilfield in China. The properties of the ZX crude oil are listed in Table 1. The ZX crude oil has a density of 0.92 g/mL and a viscosity of 208 mPa·s at the ZX reservoir temperature of 55 °C. The simulated formation water was prepared with the salinity of 5149 mg/L according to Table 2. There were four kinds of surfactants used in this study. A nonionic surfactant coconut diethanolamide (CDEA, with a purity of 98%) was obtained from Jiangsu Jintan Organic Chemical Co., Ltd. An amphoteric surfactant lauramidopropyl hydroxysultaine (LHSB, with a purity of 35%) and an anionic surfactant sodium dodecyl benzene sulfonate (SDBS, with a purity of 90%) were both obtained from Linyi Green sense Co., Ltd. The cationic surfactant dodecyl trimethyl ammonium bromide (DTAB, with a purity of 98%) was obtained from Sinopharm Chemical Reagent Co., Ltd.

2.2. Measurement of IFT. The IFTs between crude oil and surfactant systems were measured at the ZX reservoir temperature of 55 °C by a Texas-500C spinning drop tensiometer (CNG, USA). Before the determination, the densities of crude oil and chemical solution were measured by a petroleum densimeter, and the refractive index of the chemical solution was measured by a WZS-1 Abbe refractometer. This spinning drop tensiometer equipped with image-acquisition software could automatically capture and measure the dynamic IFT value. The dynamic IFT value changes with time and finally reaches an equilibrium value after tens of minutes to several hours. The IFT discussed in this study is the equilibrium value of the dynamic IFT.

2.3. Determination of the \( n_{\text{min}} \) Value of the Surfactant System. The average molecular weight of the surfactant has a corresponding relationship with the carbon number of \( n \)-alkane, and only one alkane carbon number can reach the lowest IFT. The alkane carbon number that can produce the lowest IFT is defined as the minimum alkane carbon number \( n_{\text{min}} \) of the surfactant.

Under the condition of the \( n_{\text{min}} \) of two single surfactants, the \( n_{\text{min}} \) of two surfactant mixtures can be predicted by a linear average approach according to eq 1.30

\[
(n_{\text{min}})_{m} = x_{A}(n_{\text{min}})_{A} + x_{B}(n_{\text{min}})_{B}
\]  

where \( x_{A} \) and \( x_{B} \) are the mole fractions of two kinds of surfactants, respectively, \((n_{\text{min}})_{A}\) and \((n_{\text{min}})_{B}\) are the alkane carbon numbers of two surfactants, respectively, and \((n_{\text{min}})_{m}\) is the alkane carbon number of the mixed surfactant system.

2.4. Determination of EACN of Crude Oil. The EACN value of the crude oil was determined, respectively, by direct and indirect methods to ensure the reliability. The direct method was to determine the IFTs between 0.3 wt % of four kinds of surfactant solutions (CDEA, LHSB, SDBS, and DTAB) and different \( n \)-alkanes with a known carbon number and ZX crude oil with unknown EACN, and therefore, the EACN of the oil was obtained by comparing the IFTs of the ZX crude oil system with that of \( n \)-alkane systems.

The indirect method was to first determine the IFTs between the 0.3 wt % surfactant whose \( n_{\text{min}} \) value is known and each mixed oil sample which mixed the oil with \( n \)-pentane and \( n \)-dodecane, respectively, at different molar ratios and then to calculate the EACN of the oil by applying eq 2 based on the formulation of the mixed oil sample with minimum IFT. 31

\[
(EACN)_{x_{A}}x_{A} + (EACN)_{x_{B}}x_{B} = (EACN)_{x_{m}}x_{m}
\]

where \( x_{A}, x_{B}, \) and \( x_{m} \) are mole fractions of alkane, crude oil, and mixed oil samples, respectively, \((EACN)_{A}\) and \((EACN)_{B}\) are EACNs of alkane and crude oil, respectively, and \((EACN)_{m}\) is equal to the \( n_{\text{min}} \) value of the surfactant solution used in the method.

2.5. Evaluation of the Oil Washing Efficiency. Before the oil washing experiment, the characteristic wavelength of the ZX crude oil was determined by a Spectrum 722 visible spectrophotometer (China, Spectrum Instruments) and the

Table 1. Basic Properties of the ZX Crude Oil Sample

| oil sample | viscosity at 55 °C (mPa·s) | density at 55 °C (g/cm³) | acid number (mg of KOH/g) | resin (wt %) | normal heptane asphaltene (wt %) |
|-----------|--------------------------|--------------------------|---------------------------|-------------|-------------------------------|
| ZX        | 208                      | 0.92                      | 0.622                     | 18.2        | 0.945                          |
standard curve of absorbance was drawn. The oil washing efficiency experiment was performed as follows: first, the crude oil was mixed with 60–80 mesh washed and dried quartz sand at a mass ratio of 1:4 in a bottle for aging at the reservoir temperature of 55 °C for 1 week after removing air by nitrogen gas and sealing it. Next, the prepared oil sands were mixed with a 0.3 wt % surfactant solution at a mass ratio of 1:10 for aging for 48 h at the reservoir temperature of 55 °C (the bottle was shaken twice a day). Then, 5 g of the upper liquid was taken out from the aged mixed liquid to extract the crude oil with petroleum ether, and the petroleum ether and crude oil solution was analyzed with the colorimetric method at the characteristic wavelength of the crude oil. Finally, the eluted crude oil concentration $c_o$ in the surfactant solution was calculated by using the standard curve, and then, the oil washing efficiency $R_s$ can be calculated according to eq 3.

$$R_s = \frac{c_o}{0.2 \text{ g/mL}} \times 100\%$$  \hspace{1cm} (3)

where 0.2 g/mL is the maximum elution concentration of crude oil in the experiment.

3. RESULTS AND DISCUSSION

3.1. Determination of the $n_{\text{min}}$ Value of Surfactants.

Due to the complexity of crude oil composition, it is very difficult to study according to the actual crude oil system, so the concept of EACN is proposed. The IFTs are measured with the surfactant and a series of $n$-alkanes instead of the crude oil phase. It was found that only one alkane can give the minimum IFT to the surfactant system. If the crude oil and the surfactant test can also give low IFT ($10^{-2}$ mN/m), then the alkane with similar IFT is equivalent to the crude oil, so the equivalent alkane carbon value of the crude oil is equal to the carbon number of the $n$-alkane. Using $n$-alkanes instead of crude oil for experiments can greatly simplify the work of screening low-tension surfactant systems. The equilibrium values of dynamic IFT between 0.3 wt % of four kinds of surfactant solutions (CDEA, LHSB, SDBS, DTAB) and $n$-heptane, $n$-octane, and $n$-decane are shown in Table 3. The results show that CDEA and LHSB have low IFT values, and their $n_{\text{min}}$ values are 4 and 6, respectively. SDBS and DTAB have high IFT values, and their $n_{\text{min}}$ values are 8 and 10, respectively. Therefore, CDEA and LHSB are more suitable for the study of low-tension surfactant systems.
and DTAB) and different n-alkanes with a known carbon number were determined. As shown in Figure 1, the minimum IFT occurs when 0.3 wt % CDEA, SDBS, LHSB, and DTAB are against the alkane carbon numbers of C₆, C₁₀, C₈, and C₆, respectively, which means that the n_{min} values of the four kinds of surfactant solutions are 6, 10, 8, and 6, respectively.

3.2. Determination of the EACN Value of Crude Oil.

3.2.1. Direct Method to Determine EACN. Figure 2 shows the dynamic IFTs between the above four kinds of surfactant solutions and the ZX crude oil. As shown in Figure 2, the equilibrium values of dynamic IFT of 0.3 wt % CDEA, SDBS, LHSB, and DTAB against the crude oil are 0.003, 0.8, 0.85, and 1.2 mN/m, respectively. Table 3 shows the comparison of IFT values of surfactant/crude oil and surfactant/n-heptane, and it can be concluded that the EACN of the crude oil is approximately equal to that of n-heptane against the same surfactant system. Compared to the result in Table 3, it can be shown that the EACN of the crude oil is 7.

Figure 3. IFTs between the 0.3 wt % CDEA solution and n-pentane/crude oil and n-dodecane/crude oil mixtures at different molar ratios.

Figure 4. Dynamic IFTs between the ZX crude oil and 0.3 wt % CDEA/SDBS mixture at different mole fraction ratios.

3.2.2. Using Indirect Method to Determine EACN. The relative molecular mass of the crude oil is approximately 640 g/mol, which was determined by the National Standard of China (GB/T 17282-2012, test method for the estimation of mean relative molecular mass of petroleum oils from viscosity measurements). Based on this, the oil sample was prepared by mixing the crude oil with n-pentane and n-dodecane, respectively, at different molar ratios. Then, the IFT values between 0.3 wt % CDEA and each oil sample were determined. As shown in Figure 3, the minimum IFT is observed when the mole fraction of n-pentane is 50% in the oil sample. Therefore, the EACN of the crude oil can be calculated by applying eq 2

\[
\text{EACN} = \frac{[(\text{EACN})_{\text{mo}} x_{\text{mo}} - (\text{EACN})_{a} x_{a}]/x_{o}}{\text{n-hexane}}
\]

\[
= (6 \times 1 - 5 \times 0.5)/0.5 = 7
\]

The EACN of the crude oil is the same whether determined by the direct method or indirect method, which indicates that
the experimental result is accurate and reliable. Therefore, the EACN of the ZX crude oil is 7.

3.3. Selection of Surfactant Formulations with Ultra-low IFT. For the screening of surfactant formulations with ultralow IFT, nonionic surfactant CDEA was mixed with anionic surfactant SDBS at different molar ratios due to the synergistic effects between a nonionic surfactant and an anionic surfactant.32 The dynamic IFTs between the ZX crude oil and 0.3 wt % CDEA/SDBS mixture at different molar ratios are shown in Figure 4. It can be seen that the equilibrium value of IFT reaches a minimum of $5 \times 10^{-4}$ mN/m, when the mole ratio of the CDEA/SDBS mixture is 3:1.

The $n_{\text{min}}$ value of the 0.3 wt % CDEA/SDBS system can be obtained by applying eq 1

$$n_{\text{min}} = n_{\text{CDEA}(n_{\text{min}})} + n_{\text{SDBS}(n_{\text{min}})} = 6 \times \frac{3}{4} + 10 \times \frac{1}{4} = 7$$

The $n_{\text{min}}$ value of the CDEA/SDBS mixture at a molar ratio of the 3:1 system obtained by calculation is the same as the EACN of the ZX crude oil, indicating that the minimum IFT occurs when an optimum surfactant formulation and the crude oil have the same alkane carbon number.

To further verify the above result, the IFTs between other mixed surfactant systems and the ZX crude oil were also determined. As shown in Figure 5, the IFT has reduced to the lowest when CDEA was mixed with LHSB at 1:1 in the system. Table 4 shows the comparison of the EACN of crude oil with the minimum alkane carbon number ($n_{\text{min}}$) of optimum surfactant formulation. The result shows that the $n_{\text{min}}$ of the CDEA/LHSB system has the same value as the EACN of the crude oil through the calculation. Similarly, the DTAB/LHSB system has reached the ultralow IFT when the mixed ratio value is 1:1. Moreover, the $n_{\text{min}}$ is also the same as the EACN of the crude oil. Figure 5 also shows that the IFT of CDEA/DTAB and LHSB/SDBS systems against the crude oil cannot achieve ultralow values no matter what the mixed ratio is. According to Figure 1, the $n_{\text{min}}$ values of CDEA and DTAB are both 6, while the $n_{\text{min}}$ values of LHSB and SDBS are 8 and 10, respectively, which makes it impossible to be equal to the EACN of the crude oil for the $n_{\text{min}}$ values of CDEA/DTAB and LHSB/SDBS systems by using the calculation method in eq 1. In conclusion, the IFT between a mixed surfactant system and the crude oil can obtain an ultralow value when the $n_{\text{min}}$ of the surfactant mixture is equal to the EACN of the crude oil.

| optimum surfactant formulation | EACN of ZX crude oil | the calculated $n_{\text{min}}$ of optimum surfactant formulation |
|-------------------------------|---------------------|---------------------------------------------------------------|
| CDEA/SDBS = 3:1              | 7                   | $n_{\text{min}} = 6 \times \frac{3}{4} + 10 \times \frac{1}{4} = 7$ |
| CDEA/LHSB = 1:1              | 7                   | $n_{\text{min}} = 6 \times \frac{1}{2} + 8 \times \frac{1}{2} = 7$ |
| DTAB/LHSB = 1:1              | 7                   | $n_{\text{min}} = 6 \times \frac{1}{2} + 8 \times \frac{1}{2} = 7$ |

Figure 5. IFTs between the ZX crude oil and 0.3 wt % surfactant mixture at different molar ratios.

Figure 6. Absorption spectrum of the ZX crude oil.
3.4. Oil Washing Efficiency of Optimum Surfactant Formulations. The absorbance of the mixture of the crude oil and petroleum ether was determined under whole band, as shown in Figure 6. It can be seen that the mixture absorbance is...
zero below 420 nm wavelength, and when the wavelength is up to 420 nm, there is a jump in the absorbance value. Therefore, the characteristic wavelength of the ZK crude oil is 420 nm.

A series of the crude oil/petroleum ether solutions are prepared with different concentrations of crude oil (0.01–0.20 g/mL), as shown in Figure 7. The absorbance of the mixture of the crude oil and petroleum ether was determined at the wavelength of 420 nm, and the result is shown in Figure 8. The relationship curve was obtained by using a linear regression approach, and the curve formula is as provided in eq 4.

\[ A = 7.77699c - 0.01098 \]  

where \( A \) is the absorbance and \( c \) is the mass concentration of the crude oil in the crude oil/petroleum ether solution, g/mL.

Figure 9 shows the oil washing efficiency of the 0.3 wt % CDEA/SDBS binary mixture at different molar ratios. It can be seen in Figure 9 that the mixed ratio 3:1 of the CDEA/SDBS mixed system corresponding to the lowest IFT has significantly the highest oil washing efficiency of 62%. Pictures of oil sand after the oil washing experiment by the 0.3 wt % CDEA/SDBS mixed system at different molar ratios are shown in Figure 10. The higher the oil washing efficiency, the less the crude oil attached to the oil sands after the oil washing experiment. Comparing the IFT experiment, it is found that the IFT is a key factor that determines the oil washing efficiency, and the oil washing efficiency increases with the decrease of IFT. This is consistent with the results of the displacement efficiency in dilute surfactant flooding conducted by Pu et al.,\(^{33}\) in which the displacement efficiency is obtained at the lowest IFT.

4. CONCLUSIONS

(1) The selection method of optimum surfactant formulation with ultralow IFT was simplified by comparing the EACN of crude oil with the minimum alkane carbon number (\( n_{\text{min}} \)) of surfactant mixtures, and the ultralow IFT can be achieved when the \( n_{\text{min}} \) of optimum surfactant formulation is equal to the EACN of crude oil.

(2) The oil washing efficiency experiments show that the oil washing efficiency increases with the decrease of IFT, and the optimum surfactant formulation with ultralow IFT has the highest oil washing efficiency.

(3) This study provides a selection method for screening surfactant systems with low IFT, which can significantly reduce the cost and time required for the selection of surfactant formulations with ultralow IFT for improving the oil washing efficiency.

Figure 10. Pictures of oil sand after the oil washing experiment by the 0.3 wt % CDEA/SDBS mixed system at different molar ratios.

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Notes
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ACKNOWLEDGMENTS

The authors acknowledge the financial support of the National Key Research and Development Program of China (2018YFA0702400), the Shandong Provincial Natural Science Foundation (ZR2019ME085), and the Fundamental Research Funds for the Central Universities (18CX02096A).
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