

Recognition and characterization of active fractions from petroleum sulfonate

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ARTICLE INFO

Keywords:

Interfacial activity
Emulsifying property
Petroleum sulfonate
Recognition
Fraction separation

ABSTRACT

Petroleum sulfonates are widely used in chemical flooding in China. The samples are complicated and not all the fractions are effective. To recognize effective fraction with excellent interfacial activity and emulsifying property, a preparative liquid chromatography technology was adopted to perform the fraction separation for petroleum sulfonate. Structural composition, interfacial activity, emulsifying property and flooding efficiency of obtained fractions were also analyzed and discussed. It was shown that petroleum sulfonate can be effectively separated into different fractions with obvious structural differences, among which one fraction with the most excellent interfacial activity and the emulsifying property was recognized. The average molecular weight and molecular weight distribution range of this active fraction were 455.5 and 428–468, respectively, which are both crucial for activities. Alkylbenzene dicyclohexane sulfonates, alkyl indan sulfonates, and alkyl naphthalene sulfonates are major structure types, and alkyl chain length is mainly distributed between 12–15, 15–17 and 15–17 respectively. Besides, this active fraction is universal, and it can produce ultralow interfacial tension ($\sim 10^{-3}$ mN/m) and good emulsifying property against different crude oils including Xinjiang, Changqing, Liaohe Oilfields, etc. in China. The developed technology herein can be used to track and confirm the characteristics and/or active fraction in petroleum sulfonate accurately.

1. Introduction

Petroleum is one of the most important resources, and meanwhile, it is one of the scarcest resources in China. It is crucial to exploit underground petroleum as much as possible. However still approximate two-thirds of crude oils remain in the reservoir after the primary (pressure flooding) and secondary (water flooding) production (Resen et al., 2005; Wang et al., 2014). As a result, many enhanced oil recovery (EOR) techniques have been developed, especially surfactant flooding (Liu et al., 2007; Khan et al., 2009; Dong et al., 2008; Elraies et al., 2011; Hirasaki et al., 2011; Shahri et al., 2012; Ma et al., 2017; Feng et al., 2018). The key to surfactant flooding is to search suitable surfactants, which can reduce interfacial tension (IFT) to an ultra-low level ($<10^{-2}$ mN/m, even $<10^{-3}$ mN/m), and/or enhance emulsifying property between the crude oil and the surfactant solution (Santanna et al., 2009; Zhang et al., 2010; Wang and Dong, 2010; Nguyen et al., 2012; Xie et al., 2012; Zhou et al., 2013; Jeirani et al., 2014; Shang et al., 2015). Among the surfactant flooding, petroleum sulfonate is most widely used in

China owing to its good interfacial activity, wettability, foam properties, and salinity tolerance, etc. (Sun et al., 2015). Because of differences in feedstock, petroleum sulfonates used in different oilfields in China are not the same, and their flushing efficiency is not the same as well (Wang et al., 2012a,b).

As we know that petroleum sulfonate is a complicated mixed sample, and not all fractions are effective. The identification and separation of active fractions from petroleum sulfonate have not been reported so far. Few reports have described the separation and determination of mono and disulfonates (Zhao et al., 2008; Wang et al., 2011; Duan et al., 2018a,b), however, the mono and disulfonates are indistinct for insight into the performance, and the disulfonates are usually not active in most cases (Wang et al., 2012a,b). Besides, the separation and characterization of low polar sulfonates have been reported (Duan et al., 2018a,b; Liu et al., 2019). But the low polarity sulfonates account for about 70% of the total, and the main contributing fractions are still unknown. The performance of petroleum sulfonate directly affects flushing efficiency and exploitation cost. In practice, we found when the surfactant has a

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moderate hydrophilic-lipophilic ability, the interfacial density of the surfactant is the highest, as a result, low IFT will be obtained. On this occasion, the surfactant possesses a certain length of alkyl chains. Similarly, if the active fractions in petroleum sulfonate can be tracked and recognized accurately (including average molecular weight, molecular weight distribution range, alkyl chain length, etc.), it will offer huge advances in the selection of feedstock and the preparation of products with excellent performance. Besides, it will be also helpful to understand the relationship deeply between the structure and performance of petroleum sulfonate.

Gale and Sandvik (1973) found that varying in molecular weight and aromatic content of feedstock will result in different IFT and oil recovery, etc. The best combination of these properties is achieved when sulfonates have very broad molecular weight distributions. Actually, we have been seeking petroleum sulfonates with broad molecular weight distributions to be suitable for complex reservoir conditions. With the continuous development of chemical flooding in China, it is quite difficult to find a kind of sulfonates with high performance now. We wonder what kind of fraction in complicated petroleum sulfonate samples used in our fields is actually in action. If these fractions do play a key role, we want to obtain them. Sandvik et al. (1977) presented some techniques for the characterization of sulfonate samples, including separation and purification, gravimetric analysis, colorimetric analysis, etc. These methods are helpful under most conditions. With the development of separation materials and chromatographic technology, chromatographic separation can be adopted to analyze complex samples. Compared to Sandvik et al. (1977), the greatest advantages of chromatographic separation are that the sample will be recycled almost in full and the separation efficiency is high. As a result, more fractions can be obtained and used for further investigation.

In this article, to find which fraction plays a key role in flooding efficiency, we adopted the preparative liquid chromatography method to separate petroleum sulfonate. The suitable chromatographic packings have been optimized and chosen, and a dozen of fractions with obvious structural difference has been obtained. Through the evaluation of interfacial activity and emulsifying property, the active fraction was recognized. And then, the structural characterization of this fraction was discussed and the flooding efficiency has been investigated. This work will give great help to the selection of feedstock and the production of petroleum sulfonate. According to the obtained results, we can specify the average molecular weight and molecular weight distribution range of feedstock. The broader molecular weight distribution range or improper average molecular weight will not produce good performance results. Meanwhile, good sulfonate samples will be also suitable for several crude oils rather than one kind of sulfonate sample that is usually suitable for one crude oil. This will greatly enhance the application scope of petroleum sulfonate.

2. Experimental

2.1. Materials

n-hexane, isopropanol, ethanol were of analytical pure grade and were used to purify petroleum sulfonate samples. Methanol was of chromatographic pure grade and was used as the mobile phase in the preparative chromatography. NaCl and CaCl₂ were of analytical pure grade and were used to prepare the saltwater. Petroleum sulfonate, crude oil, and Berea sandstone cores were obtained from Xinjiang Oilfield (China) and used to measure the IFTs, emulsifying property and flooding efficiency.

2.2. Purification of petroleum sulfonate

Petroleum sulfonates are industrial products, and some coexisting components such as unreacted oil, inorganic salts, etc. will affect the separation efficiency. So the coexisting components were removed from

Table 1

Physical parameters of the Berea sandstone cores used in this study.

Core	Size (cm)	Permeability (mDarcy)	Pore volume (cm ³)	Porosity (%)	Saturation with oil (%)
1	Φ2.5 × 10	134.9	7.6	15.5	72.5
2	Φ2.5 × 10	134.2	7.7	15.8	73.2

the sample firstly. The Liquid-Liquid extraction method was adopted to purify petroleum sulfonate (Wang et al., 2011).

2.3. Preparative chromatographic conditions

Agilent 1100 pre-LC was used to separate petroleum sulfonate. The chromatographic column was a C₁₈ column with a diameter of 21 mm and a length of 250 mm and was prepared in our lab. The flow rate was 10 mL/min, the injection load was 10 mL, the detection wavelength was 254 nm and the injected sample concentration of purified petroleum sulfonate was 20 mg/mL. The mobile phases included water (A) and methanol (B), and gradient elution was chosen. The gradient conditions were as follows: 0–20 min, 50% B; 20–30 min, 70% B; 30–40 min, 90% B; 40–52 min, 100% B; 52 min, stop. The collection of fractions began at the fourth min, maintaining 2 min for each fraction.

2.4. Characterization of each fraction

Agilent 1100 LC/MSD was used to characterize the structures of each fraction. Electrospray ionization (ESI) and negative-ion mode were adopted. The nebulizing gas pressure was 5.5×10^4 Pa, the flow rate of the dry gas was 7 L/min, and the temperature was 350 °C. The fraction with the concentration of 0.02 wt% was dissolved in methanol and then injected into the LC/MSD to characterize its structure.

2.5. Measurement of interfacial tension and emulsifying capacity

The sample aqueous solutions were prepared with salt water (0.5 wt% NaCl) and the concentrations were 0.3 wt% normally. The interfacial tension of each fraction against crude oil was measured on a TEXAS500 spinning drop interfacial tension-meter. The rotational speed was 6000 r/min and the experimental temperature was 40 °C, which was consistent with the stratum temperature of Xinjiang Oilfield. The detection time was 120 min, at which the interfacial tension values were stable and unchanged. The emulsifying property was measured in the ruler test tube. 5 mL of crude oil and 5 mL of sample solution were added into in turn. The test tube was shaken thoroughly and stayed about 3 h in the oven of 40 °C for emulsification. According to the emulsification ratio of the oil phase (ERO), the emulsifying property of each fraction can be compared. The ERO is the percentage of emulsified oil volume versus total oil volume.

2.6. Enhanced oil recovery test

Two similar Berea sandstone cores with a length of 10 cm were used for parallel tests to measure enhanced oil recovery (EOR) in-house laboratory investigation. The physical parameters of the Berea sandstone cores were shown in Table 1. Put the Berea sandstone core in the core holder, give a pressure of about 5 Mpa around the core holder, and evacuate for 4 h. Then the core was saturated with 1 wt% brine consisting of 95 wt% NaCl and 5 wt% CaCl₂ dissolved in distilled water. Put the core holder in the calorstat of 40 °C for 48 h, saturate the core with oil, and calculate the saturation. The core holder was put in the calorstat of 40 °C for 24 h to be used for subsequent flooding experiments. The core was flushed with 2.0 pore volume (PV) of water at the flow of 0.1

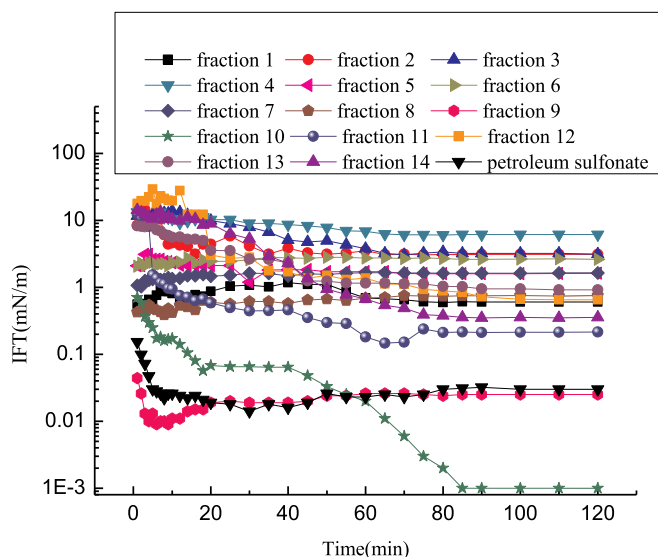


Fig. 1. Interfacial tensions of fourteen fractions and original petroleum sulfonate against crude oil.

mL/min and then flushed with 0.5 PV of surfactant solution with a concentration of 0.3 wt% (containing 0.1 wt% polyacrylamide). The pressure fluctuation range in the whole flooding experiment was 0.15–0.8 Mpa. The EORs of water flooding and surfactant flooding were calculated and compared.

3. Results and discussion

3.1. Separation of petroleum sulfonate

The packing used for preparative chromatography was C_{18} -coated silica. Compared with other kinds of chromatographic packings such as C_8 , C_4 , or CN-coated silica, C_{18} -coated silica exhibited best separation performance for petroleum sulfonate. According to the chromatographic conditions, twenty-four fractions were obtained, among which some fractions are similar. So we merged similar fractions together and obtained fourteen fractions with different structural differences finally. Because the retention time of the above fractions was not the same, the obtained fractions exhibited different polarity. The longer the retention time, the weaker the polarity. So the polarity of the first fraction is maximum and the polarity of the fourteenth fraction is minimum. The fourteen fractions and original petroleum sulfonate samples were used for subsequent analysis.

Duan et al. (2018a,b) have also separated petroleum sulfonate into several different fractions according to polarity. The greatest advantage herein is that the number of obtained fractions can be controlled and adjusted, and the whole mass recovery is high, over 95% in most cases.

3.2. Analysis of interfacial activity

As we know, the hydrophilic-lipophilic ability of surfactants determines its interfacial activity (Wang et al., 2012a,b). When the surfactant has a moderate hydrophilic-lipophilic ability, the interfacial density of the surfactant is the highest, as a result, low IFT will be obtained. Through the separation of petroleum sulfonate, we have obtained a series of fractions with different polarities. Some fractions would be likely to have a moderate hydrophilic-lipophilic ability.

Fig. 1 gives the IFTs of the fourteen fractions and original petroleum sulfonate against crude oil in saltwater. It can be observed that fraction 10 has the lowest IFTs, which is less than 1×10^{-3} mN/m. The other fractions and original petroleum sulfonate cannot achieve ultralow IFTs, which are all over 1×10^{-2} mN/m. To confirm the reliability of

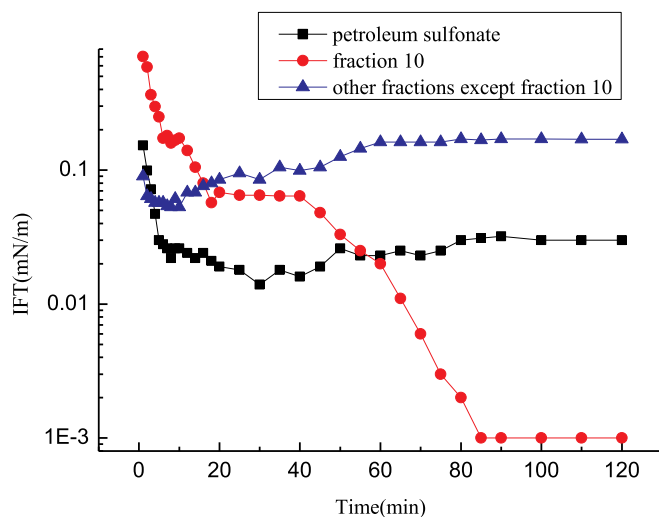


Fig. 2. Interfacial tensions of fraction 10, other fractions and original petroleum sulfonate against crude oil.

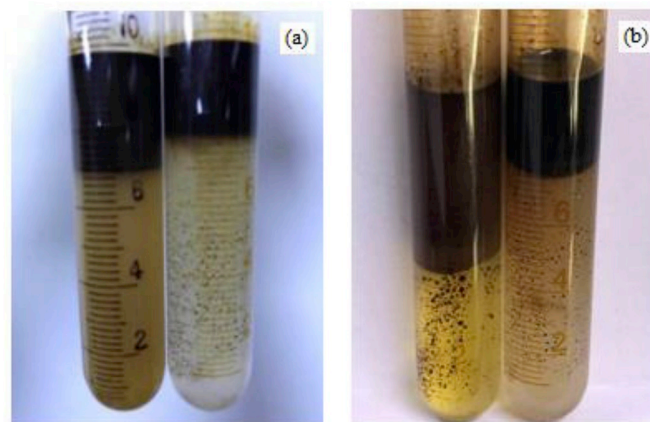


Fig. 3. Emulsifying effects of several representative fractions: a) petroleum sulfonate vs fraction 10; b) other fractions such as 1, 2, etc. vs fraction 10.

experimental results, we have repeated the experiments several times, from the preparation of fractions to the analysis of IFTs, and the results are the same. Besides, we removed the fractions 10 from petroleum sulfonate and found IFT of the remaining fractions increased obviously, higher than that of original petroleum sulfonate. The result was shown in Fig. 2. This confirms further reliability of fraction 10 as an active fraction.

In practical application, it is unachievable to obtain ultralow IFTs ($\sim 10^{-3}$ mN/m) for petroleum sulfonate and single sulfonate compounds. Wang et al. (2012a,b) synthesized a series of standard sulfonate compounds and investigated their interfacial activity. The results showed that the compound with a certain structure will have a better interfacial activity for the specified oil phase. However, the ideal IFT values are usually over 1×10^{-2} mN/m, far away from the value of 1×10^{-3} mN/m. The active fraction separated herein can achieve ultralow IFTs ($< 1 \times 10^{-3}$ mN/m), which is an interesting and encouraging phenomenon.

3.3. Analysis of emulsifying capacity

Research shows that the viscosity reduction of viscous crude oil can promote fluidity and improve the exploitation (Yang et al., 2019). Emulsifying is in favor of viscosity reduction, so the emulsifying

Table 2
EROs results of various fractions.

Sample	ERO (%)
Fraction 1	0
Fraction 2	0
Fraction 3	0
Fraction 4	0
Fraction 5	10
Fraction 6	20
Fraction 7	32
Fraction 8	40
Fraction 9	56
Fraction 10	60
Fraction 11	40
Fraction 12	24
Fraction 13	16
Fraction 14	20
Petroleum sulfonate	44
Other fractions except 10	40

capacity of various fractions has also been investigated. Fig. 3 gives the visual emulsifying capacity of several representative fractions, and individual differences are obvious. To compare the emulsifying capacity of various fractions accurately, EROs have been calculated and shown in Table 2.

From Table 2, it can be observed that fraction 10 possesses the best emulsifying capacity, and ERO is up to 60%, far superior to others, including original petroleum sulfonate. Similarly, if we removed the fraction 10 from petroleum sulfonate, ERO of the remaining fraction would decrease to about 40%, lower than that of original petroleum sulfonate.

Combined with the results of interfacial activity and emulsifying capacity, we can confirm that fraction 10 is the active fraction existing in petroleum sulfonate. Through separation and calculation, the amount of the fraction 10 is about 19.6% of the total sulfonates, which means that most of the petroleum sulfonate samples are void or weak. That is, we have always been using petroleum sulfonate inefficiently in practice. If we can produce petroleum sulfonate with good performance like

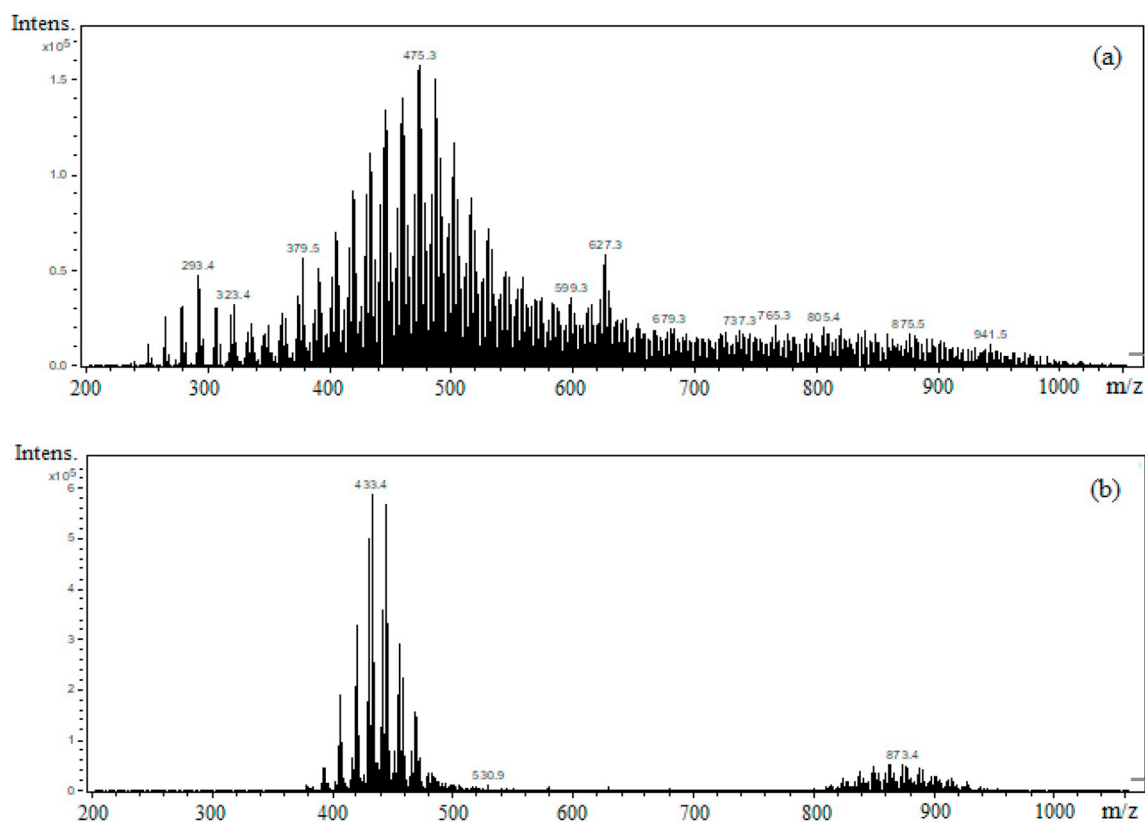


Fig. 4. ESI mass spectra of original petroleum sulfonate and fraction 10: a) petroleum sulfonate; b) fraction 10.

Table 3
Comparative results of original petroleum sulfonate and fraction 10.

Sample	Average molecular weight (not including $-Na^+$)	Distribution range of major molecular weight	Major structures and relative percentage	Major alkyl chain distribution of several structures
Petroleum sulfonate	478.6	260–650	Each structure of sulfonates exists, including dicyclohexane/alkyl indan/alkyl naphthalene/alkyl acenaphthene/phenanthrene/benzene/fluorene sulfonates	Alkyl benzene dicyclohexane sulfonates: 9-20 Alkyl indan sulfonates: 12-22 Alkyl naphthalene sulfonates: 13-23
Fraction 10	432.5	405–445	Alkyl benzene dicyclohexane/alkyl indan/alkyl naphthalene sulfonates: 68.2%	Alkyl benzene dicyclohexane sulfonates: 12-15 Alkyl indan sulfonates: 15-17 Alkyl naphthalene sulfonates: 15-17

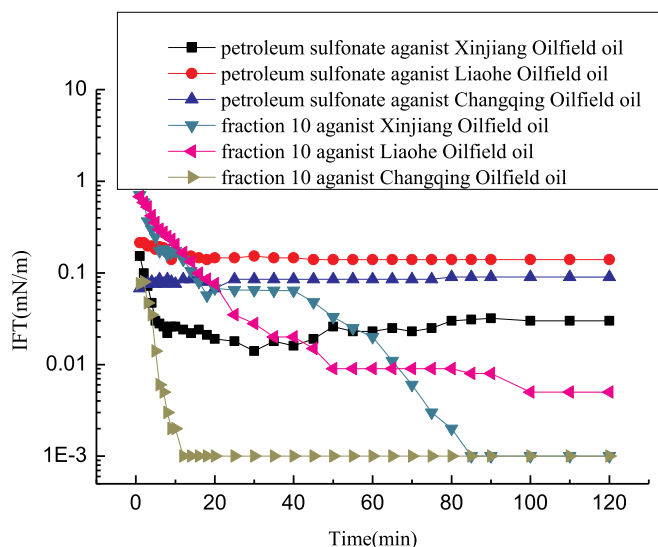


Fig. 5. Interfacial tensions of fraction 10 and original petroleum sulfonate against different crude oils.

fraction 10, it will be great progress in cost and efficiency.

3.4. Analysis of characterization

Fig. 4 gives the mass spectra of original petroleum sulfonate and fraction 10 in the negative-ion mode and the horizontal axis values are the mass to charge (m/z) ratios of $R-SO_3^-$, which is the anionic group of sulfonates. From Fig. 4, it can be observed that the original petroleum sulfonate is much more complicated, and has a broader range of molecular weight. To compare the differences between original petroleum sulfonate and fraction 10, we analyzed mass spectrum data in detail and carried through statistical analysis. Table 3 gives the related analytical results. Because of the complexity of the petroleum sulfonate sample, it is very unlikely to achieve a single compound to identify its structure. To simplify the difficulty of structure analysis, we assume that petroleum sulfonate has seven different types of structures, including alkyl benzene sulfonate, alkyl naphthalene sulfonate, alkyl indan sulfonate, alkyl phenanthrene sulfonate, alkyl fluorene sulfonate, alkyl acenaphthene sulfonate, and alkyl benzo dicyclohexane sulfonate. The different types of sulfonate have definite molecular weight, so we can sort out their corresponding compounds according to molecular weight information in mass spectra, speculate the carbon chain length and compare their relative amounts. The above approach is approximate and not rigorous, but on reflection, it is probably good enough for the use at the present.

From Table 3, it can be observed that both the average molecular weight and molecular weight distribution range are crucial for activities. When the average molecular weight and molecular weight distribution range of fraction 10 are 455.5 and 428–468 (including $-Na^+$), respectively, the performance will be best. Under these circumstances, alkyl benzene dicyclohexane sulfonates, alkyl indan sulfonates, and alkyl naphthalene sulfonates are dominant and their alkyl chain distribution ranges are quite narrow, being at 12–15, 15–17 and 15–17, respectively. Shorter or longer alkyl chains existing in the original petroleum sulfonate are both not in favor of generating good activities.

3.5. Universality analysis of fraction 10

The above experiments of interfacial activity and emulsifying property have been carried out with crude oil of Xinjiang Oilfield as tested oil. Whether the fraction 10 possesses similar activities or not should be further investigated when the tested oils changed. So two other crude oils including Changqing Oilfield and Liaohe Oilfield have been used for

Table 4

EROs results of petroleum sulfonate and fraction 10 against different crude oils.

Sample	Crude oil	REO (%)
Petroleum sulfonate	Xinjiang Oilfield	44
Fraction 10	Xinjiang Oilfield	60
Petroleum sulfonate	Changqing Oilfield	46
Fraction 10	Changqing Oilfield	64
Petroleum sulfonate	Liaohe Oilfield	36
Fraction 10	Liaohe Oilfield	50

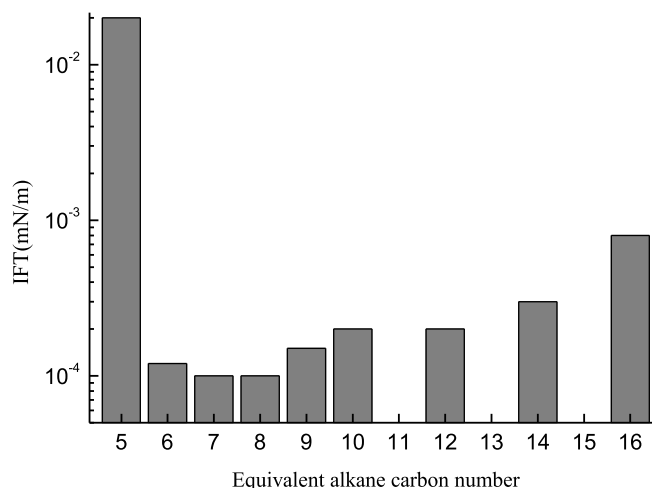


Fig. 6. Scanning curve of equivalent alkane carbon number.

tests herein.

Fig. 5 gives the IFTs of fraction 10 and original petroleum sulfonate against different crude oils. The results show that original petroleum sulfonate itself could not produce ultralow IFT ($\sim 10^{-3}$ mN/m) against different crude oils, especially for Liaohe Oilfield oil, and the IFT was over 1×10^{-1} mN/m. In fact, it is also different to find a suitable surfactant to decrease the IFT of Liaohe Oilfield crude oil to a lower level ($\sim 10^{-2}$ mN/m) in practical applications. However, fraction 10 can produce ultralow IFT ($\sim 10^{-3}$ mN/m). Although it cannot decrease IFT of Liaohe Oilfield crude oil below 1×10^{-3} mN/m, the value is still at the ultralow level, reaching 5×10^{-3} mN/m. Besides, the emulsifying capacity of original petroleum sulfonate and fraction 10 against different crude oils has also been investigated, shown in Table 4. From Table 4, it can be observed that EROs of fraction 10 have all increased in various degrees compared with those of original petroleum sulfonate.

If n-alkanes with various lengths of carbon number are used as oil phase, we can find that fraction 10 can produce ultralow IFT ($< 1 \times 10^{-3}$ mN/m) except n-pentane, as shown in Fig. 6, which further indicates the fraction 10 will produce ultralow IFT ($\sim 10^{-3}$ mN/m) against several oil phase conditions.

Through the above results, it can be sure that the fraction 10 separated from petroleum sulfonate is universal, and it can produce ultralow IFTs ($\sim 10^{-3}$ mN/m) and good emulsifying capacity against several crude oils in China. Whether it is suitable for all or most of the crude oils in China, it is necessary to be further investigated.

3.6. EOR comparison

In the case of water flooding, oil recovery was 35.2%. After surfactant flooding, the cumulative oil recovery values were 64.8% for crude petroleum sulfonate and 83.5% for fraction 10, respectively. EORs were 29.6% and 48.3%, respectively, which means the fraction 10 possesses much better flushing efficiency compared with crude petroleum sulfonate. This result is consistent with the measure results of interfacial

activity and emulsifying capacity. In the chemical flooding mining later period, a little increase in EOR will be popular.

4. Conclusion

Owing to the lack of efficient fraction separation technology for complicated samples, the identification of active fraction from petroleum sulfonate is usually not easy to achieve. To realize the above aim, a preparative liquid chromatography method has been developed and a dozen fractions have been obtained according to polarity. Through analysis and comparison of interfacial activity, emulsifying property, and flooding efficiency, the active fraction can be accurately confirmed and obtained. This fraction has an average molecular weight of 455.5, and narrow molecular weight distribution range of 428–468. This result indicates that the feedstock cannot be selected arbitrarily, and need to match the corresponding molecular weight information of active fraction. The active fraction is universal and can produce ultralow IFTs ($\sim 10^{-3}$ mN/m) and good emulsifying property against several different crude oils, which is beneficial and unusual. This phenomenon indicates good prospects for some form of petroleum sulfonate to be applicable in several reservoir conditions. Surfactants used in chemical flooding are usually a mixture, so the developed method can be referred to track and confirm the characteristic fraction with high activity accurately for other kinds of complex surfactants.

Author contributions section

1. Shuai Wang: Experiment design and completion of the whole study.
2. Huoxin Luan: Enhanced oil recovery test.
3. Xiaojing Liang: Purification of petroleum sulfonate.
4. Licheng Wang: Parts of measurement of interfacial tension and emulsifying capacity.
5. Yong Guo: Direction of MS analysis.

Declaration of competing interest

There are no conflicts to declare.

Acknowledgments

Financial support from the National Science & Technology Major Project of China (2016ZX05010-004) is gratefully acknowledged.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.petrol.2019.106797>.

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