

Investigating potential application of bio-based polymeric surfactant using methyl ester from palm oil for chemical enhanced oil recovery (CEOR)

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Abstract

Fatty Acid Methyl Ester (FAME) or palm oil methyl ester is one of the palm oil derivatives in which one of the anionic surfactants that can be generated from it is methyl ester sulfonate (MES). This bio-based surfactant can reduce the interfacial tension (IFT) between oil and water. To produce a bio-based polymeric surfactant, sulfonate groups from MES were grafted onto polymer chains. Palm oil methyl ester was reacted with sulfuric acid (H_2SO_4) to synthesize MES. Afterwards, MES was reacted with the Ethyl Acrylate (EA) monomer to synthesize polymeric surfactant. Investigating this route to produce a bio-based polymeric surfactant has become the novelty of this study. This study showed that the best polymerization result was obtained at a mole ratio of MES to EA (1:0.5) with the highest viscosity of 14.47 mm²/s. The critical micelle concentration (CMC) analysis showed 0.5% at a mole ratio of MES to EA (1:0.5) which corresponded to the lowest interfacial tension (IFT) of 1.95 x 10⁻³ mN/m. Meanwhile, the contact angle gradually decreased from 58.44 to 11.79°. The polymeric surfactant, furthermore, was analyzed using FTIR and H-NMR and successfully confirmed the formation of bio-based polymeric surfactant. The core flooding experiment found that approximately 16.57% of oil could be recovered. The results of the study revealed a good potential of the polymeric surfactant to be applied in chemical enhanced oil recovery (CEOR).

Keywords: Polymeric surfactant; ethyl acrylate; chemical enhanced oil recovery; critical micelle concentration; interfacial tension; contact angle

1. Introduction

Enhanced Oil Recovery (EOR) refers to an oil recovery method by injecting gases, chemicals, and/or thermal energy into the reservoir. The best EOR technique must be selected based on the reservoir parameters. Two main goals of EOR method are to increase the reservoir's natural energy output and to interact with the reservoir's rock/oil system to foster the recovery of any remaining oil by reducing the interfacial tension between the displacing fluid and oil as well as increasing the capillary number [1]. In view of the increased desire to preserve finite petrochemical resources and the requirement to protect the environment from existing petrochemical products, the use of renewable and biodegradable raw materials in oil industry has been a focus of research interest [2-5]. Petrochemicals or oleochemicals, such as vegetable oils, can be used to make surfactants [2, 4, 6, and 7]. Oleochemical-derived surfactants are biodegradable, making them environmentally safe and non-toxic [2,8]. Although oleochemical feedstock synthesizes surfactants for years, its potential has not ever been fully maximized for being unable to compete with petrochemical products [9].

Methyl ester from palm oil can be used to synthesize an anionic surfactant such as Methyl Ester Sulfonate (MES). Chemical reagents with sulfate or sulfite groups produced MES through the sulfonation process [10,11]. Several researchers studied the synthesis of MES using chlorosulfuric acid (ClSO₃H), sulfuric acid (H₂SO₄), Oleum (SO₃.H₂SO₄), and SO₃ gas [12]. The linear hydrocarbon moiety in MES offers the essential lipophilic property, whereas the hydrophilic sulfonate group in MES can increase water solubility. Ester sulfonate groups have good wetting, emulsifying, and dispersing characteristics [13,14]. This method is seen effective for synthesizing anionic surfactants. [15]. These substances, derived from coconut oil, perform well compared to C14-C16 olefin sulfonates and C12-C14 fatty alcohol ether sulfates [16,17]. The MES has high detergency and dispersion properties, particularly in hard water [12].

To overcome the issue in traditional ASP (Alkali-Surfactant-Polymer) flooding, the polymeric surfactant can be synthesized from MES without diminishing its effectiveness.

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Due to their distinct properties, the mixtures in ASP occur in two phases in a flow stream. In addition, surfactant loss to the reservoir rock surface is caused by surfactant attraction to the interface of rock water [18]. Because of the discrepancy between the surfactant and the polymer, more concerns such as adsorption, aggregation, and diffusion performance in porous media contribute to the deterioration in polymer behavior [19]. Moreover, despite the fact that ASP injection has been utilized to boost oil recovery in the reservoir, the existence of a strong base reduces the polymer's performance. Furthermore, in many applications, the excess polymer is also deemed necessary to attain the desired viscosity [20]. Sheng, 2014 [21] and Guo et al., 2017 [22] stated that a high alkali concentration may diminish the viscosity of polymers; as a consequence, more polymers are required to achieve the desired viscosity, which raises the cost.

The study by Guo et al., 2017 [22] revealed that scaling and erosion problems were common issues in the ASP flooding field test. Calcium and silica carbonate were the primary scaling sources in the ASP flooding pilot test with strong alkali used at the Daqing oilfield. However, by employing weak alkali, the scalability problem could be mitigated. Several studies mentioned that scaling and corrosion were common issues in ASP applications [23-25]. The use of alkali in ASP flooding has caused scaling and corrosion concerns, leading to damage to the lifting system, shortening the average pumpchecking duration, and increasing the maintenance workload. Another concern was that ASP flooding produced significant liquid treatment problems due to severe emulsification [25].

Sulfonate groups are incorporated into the groups of hydrophobic in the chain of polymer as part of the core notion underpinning polymeric surfactant production. In previous studies, Ye et al., 2004 [26] created an acrylamide-acrylic acid-based polymeric surfactant that served as the foundation for the synthesis of polymeric surfactants via polymerization. It was found out that polymeric surfactants could control viscosity and lower IFT values. As a result, it possessed the combined properties between high polymer viscosity and interfacial properties of surfactant in a single material [27]. In chemical enhanced oil recovery (CEOR), increasing the viscosity of the fluids and the oil to an ultra-low value (10⁻²-10⁻³ mN/m) are crucial [27-29].

Several researchers have studied the synthesis of polymeric surfactants for chemical flooding, as seen from a study to synthesize sodium methyl ester sulfonate (SMES) from castor oil methyl ester [20]. The polymerization procedure was applied using acrylamide monomer and SMES. The IFT value of polymeric surfactants and viscosity measurements then showed that the substance could improve oil recovery and replace the previous ASP flooding technique. Babu et al., 2015 studied the synthesis of polymeric surfactant from castor oil. Based on the investigation, polymeric surfactant had the potential to be used in EOR for being able to lower the IFT to 10⁻³ mN/m. They also reported that the molecular weight of methyl ester sulfonate-based polymeric surfactant was in the range of 2.4x10⁶ to 6.2x10⁶ g/mole [30]. The synthesis of polymeric surfactant from palm oil methyl ester for CEOR was studied in [31]. The polymerization was reacted with monomer of acrylamide for polymeric surfactant production. The study

revealed that, while the IFT value was not extremely low, the product showed a promising future as an alternative surfactant for CEOR applications. In our previous study, Wibowo et al., 2021 [32] synthesized palm oil methyl ester-based polymeric surfactant with H_2SO_4 as a sulfonating agent to produce MES. Polymeric surfactant was obtained by reacting MES with vinyl acetate monomer. The results were excellent, and the product had ultralow IFT (6.7×10^{-3} mN/m) with a low contact angle (19.2207°). Characterization using FTIR and HNMR has proven that polymeric surfactant was formed entirely. Therefore, it has potency for EOR application.

As an EOR system, a block copolymer of acrylic acid, ethyl acrylate and styrene (particularly synthesized using controlled radical polymerizations) has been proposed. In the presence of high salt concentrations, the viscosity of their aqueous solution was preserved [33]. Raffa et al., 2016 [29] reviewed some polymeric surfactants and monomers/polymers for EOR application. Hydrophobic acrylates was proposed as an alternative to vinyl acetate because it was more resistant to hydrolysis. Figure 1 and Figure 2 illustrate the chemical reaction for synthesizing MES and polymeric surfactant respectively. For creating free radicals, the surfactant chain has two potential pathways: first, the -OH bond to disintegrate, and second, the break of the C=C bond. Because the polarity of the -OH bond is greater than that of the C=C bond, it is more likely that free radicals may graft on the -OH bond rather than shattering the C=C bond. In actuality, the hydroxyl substituents (-OH) of MES showed the signs of polymer chain formation instead of the surfactant's unsaturated bond. [34].



Fig. 1. The schematic reaction of MES production



Fig. 2. The schematic reaction of bio-based polymeric surfactants production

Blanksby & Ellison, 2003 [35] stated that all chemical reactions are built in creating and breaking bonds. Bond-cutting requires some energies, and the energies produced during bond creation are crucial for comprehending the chemical reactions. With the increasing bond order, the C-C bond energies increase. Thus, for ethyl acrylate, breaking single bonds (CH₃- CH_2 -) is more accessible rather than double bonds (C=C) because the energy of the single bond is smaller than that of the double bond with 90.1 kcal/mol and 174.1 kcal/mol, respectively. Based upon the former studies, the purpose of this study is to synthesize palm oil methyl ester-based polymeric surfactant using ethyl acrylate monomer. Investigating this route to produce a bio-based polymeric surfactant has become the novelty of this study. MES was polymerized with ethyl acrylate monomer to achieve ultralow IFT and viscosity control. Furthermore, it was envisaged that the previously described

issues with chemical flooding applications would be overcome.

2. Materials and Methods

2.1. Materials

Palm oil methyl ester (POME) was used as the raw material. Meanwhile, Sulfuric Acid (H₂SO₄) 98% Merck p.a, Sodium Hydroxide (NaOH) Merck p.a, Methanol (CH₃OH) 98% p.a, Potassium persulfate (K₂S₂O₈) Merck p.a, Ethyl Acrylate Merck p.a were obtained from a local chemical store.

2.2. Experimental procedure of MES synthesis

MES was synthesized by adding 100 mL (0.35 moles) of POME and 22 mL (0.39 moles) of H_2SO_4 in a glass reactor equipped with a thermometer & reflux. The reaction was performed by stirring & heating on a hotplate stirrer for 1.5 hours at temperature of 65°C. Once the reaction was completed, the purification was carried out using methanol 40% for 60 minutes at temperature of 50°C. After purification, a 30% NaOH was used to neutralize the MES until the pH reached 5-6. To purify the MES product, the final stage was methanol evaporation using a rotary evaporator. Figure 3a illustrates the schematic diagram of MES synthesis procedure.



Fig. 3. Schematic diagram of a) Methyl Ester Sulfonate (MES) synthesis and b) Surfactant polymerization process

2.3. Experimental procedure of polymeric surfactant synthesis

The initiator of $K_2S_2O_8$ 1.23% was made by dissolving the initiator into DI water, and the pH was adjusted to 9-10 using NaOH solution. The polymerization reaction was conducted using a glass reactor installed with a thermometer & reflux. A certain mole ratio of MES (1:0.1; 1:0.5; 1:1; 1:1.5; and 1:2) was added to ethyl acrylate. Afterward, the $K_2S_2O_8$ was added. The polymerization reaction was carried out for 60 minutes using a hotplate stirrer at various temperatures (50-80°C). The schematic diagram of polymerization process of the surfactant is depicted in Figure 3b. Whereas, Figure 4 shows the laboratory equipment used in this study.

2.4. Product analysis

The product analysis of polymeric surfactant was tested for viscosity at the research laboratory, Institut Teknologi

Indonesia, while CMC, IFT and contact angle analysis were determined using Attention Theta Optical Tensiometer at EOR laboratory, Institut Teknologi Bandung (ITB). Subsequently, the optimal mole ratio was tested further to examine the functional groups using FTIR spectrophotometry (Shimadzu), the distinctive resonances using FTIR spectrometer (JEOL) at the Chemical Research Center Laboratory, BRIN. Finally, the performance of a polymeric surfactant for core flooding tests was determined by utilizing sand-pack column apparatus.



Fig. 4. Schematic of laboratory apparatus: a) Polymeric surfactant synthesis, b) Chemical Enhanced Oil Recovery (CEOR) testing equipment

3. Results and Discussion

3.1. The effect of mole ratio and temperature of polymerization reaction on product viscosity

As previously explained, polymeric surfactants has an ability to combine the high viscosity of polymers with surfactant interfacial characteristics in which the water/oil interfacial tension can be reduced while aqueous solution viscosity is increased [27, 29]. Polymeric surfactants have a significant role in ASP flooding by increasing viscosity and thus improving mobility [22]. Figure 5 shows the effect of reactant mole ratio and temperature of polymerization on product viscosity. One fascinating fact as highlighted in Figure 5 is that the higher the polymerization temperature of EA on MES, the lower viscosity of the polymeric surfactant product. It was also similar to a study conducted by Gao, 2013 [36], where polymer viscosity decreased at higher temperatures. At a ratio of MES to EA (1:0.1) from 50 to 80°C, the kinematic viscosity decreased moderately from 14.957 to 12.604 mm²/s respectively.

However, between a ratio of 1:1 and 1:2, increasing the polymerization temperature led to the viscosity of the product being inclined as well. At the 1:1 ratio, the viscosity increased significantly from 5.61 to 17.89 mm²/s. In addition, at a ratio of 1:1.5, the viscosity increased gradually between 5.48 and 10.99 mm²/s. While, at a ratio of 1:2, the rise in viscosity was not so significant. It is noticeable that, with the increase in the EA composition, the viscosity declined. Therefore, it was different from the study conducted by Cao & Li, 2002 [27], Raffa et al., 2016 [29], and Guo et al., 2017 [22]. According to Bajaj et al., 1995 [37], the mechanical stresses would separate loosely cross-linked polymer coils, resulting in a decrease in viscosity. Therefore, as the number of polymer molecules increases, stirring causes the viscosity to decrease. As shown in Figure 5, at a temperature of 60°C from a 1:0.1 to 1:2 ratio, the viscosity

decreased sequentially between 14.57 and 4.8 mm²/s. The typical product of polymeric surfactant can be seen in Figure 6. The similar case also occurred at 70°C in which the value of viscosities was not much different from 60°C. A similar case also occurred with the study conducted by Bajaj et al., 1995 [37] where the increase of EA composition from 28.2 to 38.1% mole and the polymer viscosity reduced from 16000 to 9700 cP.



Fig. 5. The effect of reactant mole ratio and temperature of polymerization on product viscosity



Fig. 6. Product of polymeric surfactant

3.2. Critical micelle concentration (CMC) analysis

Figure 7 depicts the surface tension as a function of synthesized polymeric surfactant concentration with the increasing concentration at 30°C. As seen in the figure, the surface tension of surfactant solutions decreased to a specific concentration of surfactant due to surfactant monomer adsorption onto the air-water interface. This concentration is known as the critical micelle concentration (CMC). Because of surfactant monomer optimization at the air-water interface, there is no further decrease in surface tension after CMC [38]. Surfactant molecules are in the form of monomers below the CMC, and when the concentration of surfactants in the bulk increases, the surface or interfacial tension decreases significantly. On the other hand, above the CMC, the concentrations of monomers are nearly stable [39]. Furthermore, emulsification, solubilization, and dispersion are possible due to the formation of micelles. [40, 41].

As seen in Figure 7, the interfacial tension sharply declined as the concentration of polymeric surfactant increased until 0.5%, at the lowest interfacial tension of 1.95×10^{-3} mN/m. Additionally, the surface tension increased with the increasing concentration of surfactants to 1% at interfacial tension of 0.0028 mN/m. As a result, the CMC of surfactant was determined to be 0.5% due to the lowest interfacial tension value.



Fig. 7. Concentration of polymeric surfactant versus interfacial tension

3.3. The effect of reactant mole ratio versus interfacial tension

According to Babu et al., 2015 [30], the mole ratio of monomer to sulfonate could alter the interfacial tension of the products. The interfacial tension measurement was conducted at a concentration of 0.5% polymeric surfactant in the synthetic formation H_2O . Figure 8 exhibits the effect of reactant mole ratio on interfacial tension of the polymeric surfactant product. As seen in the figure, initially, the interfacial tension decreased slightly from 0.0084 to 0.00195 mN/m at a ratio of 1:0.1 and 1:0.5, respectively. Afterward, the interfacial tension increased to 0.0988 mN/m at a ratio of 1:1.5 and then decreased to 0.0665 mN/m at a ratio of 1:2. The best mole ratio of MES to EA in polymerization was obtained at 1:0.5 due to the lowest IFT, which became an optimum condition for micelle formation. [40].



Fig. 8. The effect of reactant mole ratio on interfacial tension

3.4. FTIR analysis

Based on the results, the optimal condition was attained at a mole ratio of 1:0.5 for having the lowest interfacial tension. In addition, the product was qualitatively analyzed using FTIR spectrophotometry to detect the presence functional groups. Figure 9 depicts the FTIR analysis results of the product. The carboxylic acid and alcohol (-OH) stretching on the hydrogen bond were detected at the wave number of 3420.86 cm⁻¹. The primary band in the infrared (IR) alkane spectra was caused by CH stretching in the wave number between 2922.93 and 2853.34 cm⁻¹. The functional group of the C=O stretching of the ester was detected at 1741.10 cm⁻¹. In addition, the area of the wave number of 1637.46 cm⁻¹ showed the C=C stretching of alkenes, indicating ethyl acrylate. Moreover, the sulfonate group (S=O) was found in the peak from 1464.96 to 1193.00 cm⁻¹. Furthermore, ether was observed with IR absorption characteristics due to C-O stretching at the peak of 1170.72 cm⁻¹. Based upon FTIR analysis, the presence of ester, sulfonate, alkene, and ether in the result indicated that the polymeric surfactant was successfully produced.



Fig. 9. The FTIR spectrum graph for polymeric surfactant at mole ratio of reactant (1:0.5)

3.5. H-Nuclear Magnetic Resonance (H-NMR) analysis

As stated by Bharti & Roy, 2012 [42], the intensity of the signal in the NMR spectrum is directly proportional to the number of nuclei available at a certain resonance, which is the most important fundamental relationship in NMR. The HNMR spectra employed CdCl₃ solvent to validate the polymerization of MES surfactant by ethyl acrylate. The resonance properties of the products were determined using NMR at a mole ratio of 1:0.5. Figure 10 represents the distribution of the spectrums.



Fig. 10. The product's H-NMR spectrum at a mole ratio of 1:0.5

The chemical changes caused by the two protons of an alkene's double bond (R-CH=CH) were identified at = 4.7007 ppm and between δ = 5.7628 and 6.3837 ppm. The distinctive resonances for ester (R-COO-CH₃) attributable to the polymeric surfactant structure were observed from = 4.1590 to 4.2017 ppm. The alcohol functional group (CH-OH) was detected at δ = 3.6257 ppm, which indicated that not all of the OH reacted with the polymer. The sulfonate group (R-SO₃H) was detected at δ = 2.2641 ppm. The chemical shifts resulting

from the polymer chain of alkyl (methine) (R₃-CH) were seen at $\delta = 1.5661$ -1.5953 ppm, thus confirming the polymeric surfactant formation. The polymer chain of EA (RO-CO-CH) was detected at $\delta = 2.2490$ and 2.2792 ppm. The hydrophobic group of polymeric surfactants was detected in the chemical shift of alkyl (methyl) R-CH₃ and alkyl (methylene) R-CH₂-R at $\delta = 0.8293$ -0.8564 ppm and $\delta = 1.2203$ -1.2813 ppm respectively where both indicated the formation of long carbon chains.

3.6. Contact angle analysis

A surfactant is a surface-active material with a nonpolar (hydrophobic or water repelling) tail and a polar (hydrophilic or water-loving) head. It has been demonstrated by researchers that all forms of surfactants have the ability to change the wettability of reservoirs to promote more water-wet conditions and to reduce the IFT between the oil and aqueous phases [43]. The change of wettability can be analyzed by contact angle measurement. The contact angles and interfacial tension parameter help to select a suitable surfactant for chemical-enhanced oil recovery. For rock surface alteration and wettability, the contact angle is crucial, as it represents the degree of wettability during the interaction of solid and liquid interact [44]. Low wettability is indicated by high contact angles ($>90^\circ$), and high one is indicated by modest contact angles ($<90^\circ$) [45].

Attention Theta Optical Tensiometer was used for IFT and contact angle determination for polymeric surfactant solution. By analyzing the contact angles at the interface of oilsurfactant, it was possible to identify how polymeric surfactant affected the wettability alteration mechanism [30]. In this study, polymeric surfactant solution (0.5%wt) was dropped in contact with a thin section of Berea rock. In comparison, synthetic formation water (brine water 10000 ppm) and ndecane as synthetic oil were also dropped onto the same surface. Polymeric surfactants have nonpolar (hydrophobic) tail and a polar (hydrophilic) head, which have a major impact on wettability by significantly lowering the contact angles. The surface of oil-wet changes moderately to a state of water-wet required for CEOR. Figure 11 demonstrates the contact angle characteristics of polymeric surfactants over time.



Fig. 11. The Dynamic two-phases (Solid-liquid) contact angle measurement on polymeric surfactant solution, oil, and brine on the thin surface of Berea rock

The contact angle between the thin surface of Berea rock and polymeric surfactant, crude oil, and brine water has been determined. In the beginning, the contact angle of brine water and polymeric surfactant was almost similar at 58.71 and 58.44°. On the other hand, oil has a reasonably low contact angle of 30.59°. The contact angles of oil and brine water decreased mildly to 16.56 and 44.4°, respectively, after 1 minute. Interestingly, the polymeric surfactant solution decreased rapidly to 11.79° after 1 minute.

According to Babu et al., 2015 [30] the initial polymeric surfactant contact angle is greater due to the sample's viscous solution. Initially, because the polymeric surfactant is viscous by nature, the thin film deposition on the rock surface is stable and affords a greater contact angle. However, studies showed that the oil-wet state of the Berea rock surface has been changed to water wet using a polymeric surfactant solution. Figure 12 depicts a drop of polymeric surfactant solution on the thin surface of Berea rock and clearly, it achieved an excellent result because $<90^{\circ}$ of contact angle indicates strong wettability.



Fig. 12. The contact angle analysis of polymeric surfactant solution (0.5%) falls on the thin surface of Berea rock. a) 0 sec, b)12 sec, c) 24 sec, d) 36-sec e) 48 sec, f) 60 sec

3.7. Core flooding analysis

3.7.1. The effect of product concentration versus oil recovery

The performance of product was conducted using a core flooding test apparatus, as shown in Figure 4b. According to study reported by Buanasari and Pramudono [46], the sandstone to oil ratio in the EOR model was 1:15. First, the polymeric surfactant solution was pumped to 1.83 mL/s through a sand-pack column containing oil. Afterward, the oil recovered by the polymeric surfactant was collected in a bottle for further weighing. Figure 13 shows the effect of concentration of polymeric surfactant on the oil recovery. From this figure, the oil recovery was about 16.2% by 0.1% concentration of polymeric surfactant and then decreased at a concentration of 0.3%. The highest oil recovery was obtained at 16.57% by a concentration of 0.5%. Following this, the oil recovery decreased moderately as the concentration increased.

3.7.2. The effect of polymeric surfactant product in various mole ratios of MES to EA on oil recovery

Figure 14 exhibits the performance of the polymeric surfactant sample on oil recovery. For polymeric surfactant products with a ratio of 1:0.1, the oil recovery was obtained at

12.93% and increased to 16.57% at a 1:0.5 ratio. Afterward, the oil recovery decreased as the number of monomers increased. The low recovery of crude oil by polymeric surfactant was due to the low quality of the polymeric surfactant. It was because MES was not grafted very well onto polymer chains.





Fig. 13. Concentration of polymeric surfactant versus oil recovery

Fig. 14. The effect of mole ratio versus oil recovery

4. Conclusion

Based on the viscosity test, it was found that the higher the polymerization temperature, the lower the polymeric surfactant product viscosity. Additionally, it is noticeable that the viscosity declined with the increase in the ethyl acrylate composition. Based on CMC analysis, it was found that the critical micelle concentration of product was at a concentration of 0.5% due to the lowest IFT value (i.e. 1.95×10^{-3} mN/m). Moreover, it was found that the IFT value fell as the mole ratio methyl ester sulfonate to ethyl acrylate rose. The best mole ratio of methyl ester sulfonate to ethyl acrylate in polymerization reaction was gained at a mole ratio of MES to EA of 1:0.5. It had the lowest interfacial tension (i.e. 1.95x10⁻³ mN/m) and highest viscosity at 14.47 mm²/s. Furthermore, the polymeric surfactant characterization using FTIR and HNMR confirmed that the polymeric surfactant was formed. Based on the wettability alteration analysis with two-phase (solid-liquid) contact angle determination on the product, it demonstrated that the product had an extremely good wettability alteration performance by significantly reducing contact angles from 58.44 to 11.79°. Finally, the product performance was examined by sand-pack column as a CEOR model. Oil recovery decreased moderately as the product concentration

increased, and the number of monomers raised in the polymerization reaction. Overall, according to the study, the products offered ultralow IFT, good wettability alteration, and excellent core flooding test; hence, it could be used as an alternative surfactant for CEOR applications.

Declaration of Competing Interest

The authors confirm that no known competing financial interests or personal relationships could have appeared to influence the work reported in this paper.

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