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Green Surfactant: Synthesis of Sulfonate Surfactants Using Strecker Modification Techniques and Surfactant Formulation for Chemical Enhanced Oil Recovery (CEOR) Applications

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ABSTRACT - Despite the continuing development of sustainable sources of energy, crude oil and natural gas resources are remaining crucial elements of the international economy. With global petroleum and liquid fuel demand continually increasing, improving the efficiency of extraction from existing natural reserves of petroleum is of utmost importance as the world gradually transitions away from fossil fuels toward more sustainable sources. Toward that end, enhanced oil recovery (EOR) techniques are developed and used to minimize the amount of crude oil and petroleum that is being left behind in underground reservoirs from conventional drilling extraction methods. In this study, surfactants are being synthesized using a fatty acid derived from palm oil as a hydrophobic group and sulfonate as a hydrophilic group. The use of vegetable oil as the raw material is likely due to its abundance and environmental friendliness. Sulfonation of anionic surfactant is performed by utilizing the Strecker modification technique in which an alkali metal bisulfite (versus sulfite) is used to sulfonate the epoxide group. The preferred sulfonating reagent is a mixture of sodium bisulfite and sodium sulfite (1:1; 1:2; 1:2.5), as well as various time reactions. The product surfactant is characterized by thin-layer chromatography (TLC) to determine the optimum conditions and reaction conversion. The molecular structure of the surfactant is confirmed by 1H NMR. Nonionic surfactant is then being analyzed by measuring the interfacial tension (IFT) of oil and water and compatibility. The results show that the optimum conditions to obtain the high conversion are achieved by reacting oleyl glycidyl ether and Sodium Sulfite-Bisulfite at an equivalent mole ratio of 1:2 and 21 hours' reaction time. Oleyl Glycidyl Ether Sulfonate surfactant is able to decrease the IFT of oil and water to 10-2 dyne/cm in a brine salinity condition of 18000 ppm and oil 34,39 OAPI. This study also formulating surfactants OGES and OGEP so that the IFT was up to 10-3. The results are then used to design the synthesis of vegetable surfactant oil with various carbon chain lengths and functional groups as an EOR surfactant hydrophobic group.

Keywords: vegetable surfactant, chemical injection, enhanced oil recovery, strechker reaction.

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INTRODUCTION

It is perceived that primary and secondary recovery mechanisms of oil recovery are incapable of fully draining the reservoir on account of domineering capillary forces or the deficient sweep efficiency of the injection fluid. These highlight the need for more effective and advanced approaches to move the remaining oil and increase drainage efficiency. As the most commonly employed secondary method of improved oil recovery (IOR), water flooding maintains the reservoir pressure and mobilizes the trapped oil.

In 1959, Martin examined the influence of injection brine composition, and the results displayed higher oil recovery because of salinity reduction, due to the migration of clay particles (Martin 1959). The topic remained under-researched until 1967, when it was revived by Bernard's study, deploying laboratory investigations on Berea sandstone cores. The results highlighted the influence of salinity reduction on improving oil recovery (Bernard 1967). Consequently, a new line of research on active mechanisms during low salinity water (LSW) has flourished, considering fine migration (Fouladi et al. 2019; Tang & Morrow 1999).

Chemical flooding is one of the enhanced oil recovery (EOR) technologies that employs chemicals, such as surfactants, polymers, and alkaline solutions, to improve the oil recovery. Each chemicals have its own role in the system to create the most efficient way for increasing oil production (Olaire 2014; Fletcher et al. 2015). Surfactant is used for extracting the oil from the porous rock (Fletcher et al. 2015; Bera et al. 2014), whereas polymer is used as mobility control, leading the oil into the production wells (Sheng 2011). On the other hand, alkaline is utilized to minimize the adsorption effect of the reservoir rock (Hirasaki et al. 2011; Nedjhioui et al. 2005). EOR activities involve the use of suitable mixtures containing them, as in polymer-alkaline, surfactant-polymer, and alkaline surfactant-polymer (ASP) flooding mixtures (Battistutta 2015; Marhaendrajana et al. 2025; Al-Fikri et al. 2025). The use of surfactant has been proven to be able to increase the oil production by generating ultralow interfacial tension at 10-3 dyne/cm (Jang 2016). Surfactants that are commonly used for chemical injection are methyl ethylene sulfonates, which showed good dispersion characteristics and good detergency, especially in hard water and in the absence of phosphate (Hidayati et al. 2012; Sugihardjo 2013). Because the surfactants used are petroleum-based, they are difficult to decompose by bacteria in nature, so they become pollutants that damage the environment, starting from the quality of water and soil, which will have an impact on the environment and the health of living things, MES based on vegetable oil is more preferable to develop rather than petroleum-based surfactants.

Vegetable-based surfactants, derived from natural oils like palm oil, castor oil, and coconut oil, are gaining interest due to their biodegradability and low toxicity. They are synthesized through processes such as esterification, sulfonation, or ethoxylation. These surfactants show excellent performance under high salinity and temperature, making them suitable for sustainable and green EOR applications.

Vegetable oil-based surfactants are gaining attention as environmentally friendly alternatives to synthetic types. Derived from renewable resources like palm, soybean, and castor oil, these bio-surfactants are biodegradable, less toxic, and effective under harsh reservoir conditions. They hold promise for sustainable EOR applications, especially in light of global environmental regulations. Surfactants derived from vegetable oils (nabati-based), such as palm oil, soybean oil, and castor oil, are gaining traction due to renewability and low toxicity. These bio-based surfactants can be tailored for EOR purposes, with promising performance in IFT reduction and environmental compatibility (Zhang et al. 2020). Synthesis of surfactants using vegetable oils is recommended because of the availability and environmentally friendly factors.

In Indonesia, surfactants from vegetable oils have been widely studied using palm oil raw materials, despite Mira et al. having synthesized Methyl Ether Sulfonate (MES) Surfactant from olein. The Surfactants are made by modifying the ester group using methanol and adding sulfonate groups to produce good hydrophilicity (Mira et al. 2011). The same experiment was done by Yulianti et all (2017) synthesizing Polyethylene Glycol Oleate Sulfonate. Putra et al. (2020) synthesized surfactants by reacting oleic acid and polyethylene glycol (PEG) 400 using azeotropic techniques, able to decrease the oil and water interfacial tension up to 10^{-3} dyne/cm in brine salinity conditions of 18000 ppm and oil 34,39 °API. In the research conducted by Putra et al. (2020), they synthesized polyethylene glycol (PEG) 400 oleate compounds using the azeotrope technique. The surfactant produced was able to reduce the surface tension up to 10-3 dyne/cm in brine salinity conditions of 18000 ppm and oil 34.39 °API. However, based on the type of surfactant bond and the surfactant functional group, polyethylene glycol (PEG) 400 oleate, which is an ester bond, is susceptible to hydrolysis. It is also possible that this surfactant cannot be applied to high-temperature reservoir conditions. In this study, surfactant synthesis was carried out by designing surfactants from oleic acid derivatives by modifying ester bonds to make ether bonds and adding sulfonate functional groups. Some surfactants with sulfonate groups have good temperature and salinity resistance. One example is the surfactant Alkyl olefin sulfonate (AOS), which can be an excellent candidate as an EOR surfactant that acts at low, medium, and high salinity (600-80,000 ppm) and a wide temperature range (Baviere et al. 1998).

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Alkyl ether sulfonate surfactants are a group of sulfonate surfactants produced from ring-opening oxirane using a mixture of sulfite/bisulfite salts. Glycidyl ethers are sulfonated by a process known as Strechkerization. The known Strechkerization reactions are reactions that use an alkali metal sulfite, which will sulfonate a chlorine group. However, sulfites do not react with epoxide groups. The solution to this problem can be carried out through modified Strechkerization wherein the alkali metal bisulfites (versus sulfites) are used to sulfonate the epoxide groups. The preferred sulfonating reagent is a mixture of sodium sulfite and sodium bisulfite, although other alkali metals may be used, but will alter the properties of the final surfactant due to differences in the cations present. The initial process begins with the preparation of a mixture of sodium sulfite and sodium bisulfite in the form of an aqueous solution of the two reagents or through the reaction of the bisulfite with an amount of sodium hydroxide to produce the desired amount of sodium sulfite. Usually, the reaction is carried out at a solution pH between 8 and 10 to give a good completeness of the reaction (David 1962). This pH range is recommended for the remainder of the process. Aqueous sulfite/bisulfite solutions are reacted with glycidyl ether to produce a sulfonated final product. In this paper, anionic surfactant synthesis is carried out using the strechkerization technique with the alkyl chain used as a derivative of oleic acid, namely oleyl alcohol. Furthermore, this anionic surfactant will be tested for its solubility and IFT in the form of a single surfactant and formulation with nonionic surfactants. The performance testing of the synthesized surfactant was carried out in a reservoir with a salinity of 18,000 ppm, crude oil of 34° API, and a temperature of 60°C.

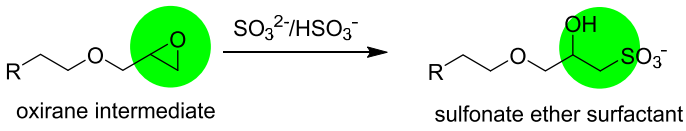


Figure 1. Synthesis pathway that can be used for sulfonation with the Strechkerization reaction (Modified stretcherization for sulfonation using a combination of sodium sulfite and sodium bisulfite)

METHODOLOGY

Technical grade oleyl alcohol from palm oil, Epiclorohydrin (EPH), Tetrabutylamonium Bromide (TBAB), Sodium Sulphite (Na_2SO_4), Natrium Hidrogen Sulphate ($NaHSO_4$), Sodium Hidroxide (NaOH), Tetrabutylamonium Bromide (TBAB) were used as raw material and reagents of the surfactant. Hexane and Aquades were used as solvents to facilitate the reaction. Sulfuric acid, sodium sulfate anhydrate, and cerium sulfate pentahydrate, with analytical grade, were used for the analysis of the product. Crude oil "A" with 34.39 °API was used for IFT measurement.

Optimization of etherification

The first reaction step, etherification of oleyl alcohol to form an epoxide ring using epichlorohydrin (EPH) with a catalyst reaction using 0.2 eq. Sodium hydroxide (NaOH) facilitated the transphase catalyzed by 0.0063 eq—tetrabutylammonium bromide (TBAB). The second stage of the reaction, optimization, was carried out by varying the ratio of the equivalent oleic acid reacting with variations of sodium sulfite and sodium bisulfite, as follows: 1:1; 1:2; and 1:2.5, to obtain the highest yield. Addition of solvent using aquades by heating at 75 ℃, and after 35 minutes for heating sodium sulphite, sodium bisulphite, and OGE to the reaction, the reaction temperature was increased to 100 ℃. The reaction was monitored by the TLC method with hydrated cerium sulfate ($CeSO_4.4H_2O$) and iodine trapping on silica as a staining reagent. The etherification and sulfonation products were then identified with the Spinsolve 43 MHz Proton Magritek.

Compatibility test

Compatibility tests are carried out to evaluate the solubility of surfactants as injection materials against injection water in a field that contains several mineral components, one of which is salinity. The surfactant solution was dissolved in water, which had a salinity of 18000 ppm, and the solubility of the injection material was observed visually at room temperature and reservoir temperature of 70 °C. Changes in surfactant solubility at 0, 1, 7, 14, and 30 days (Sheng 2011).

Interfacial tension (IFT) measurement

The ability of surfactant to decrease the IFT of oil and water, as the main function of surfactant, was evaluated by measuring the IFT using Spinning Drop Tensiometer TX-500 C/D. Surfactant solution with different concentrations at 18000 ppm salinity was prepared and filled into the IFT tube. Two microliters of oil were added and put into the IFT unit. Setting the device at 70 °C and running the measurement at 6000 rpm (Sheng 2011).

RESULT AND DISCUSSION

Optimization of esterification

Synthesis of anionic surfactants from vegetable materials was carried out in two stages: first, the formation of an epoxy ring, followed by a sulfonation process. Formation of an epoxy ring on the carbon chain from oleyl alcohol with epichlorohydrin using a NaOH reaction catalyst and a TBAB phase transfer catalyst.

The first reaction was carried out by reacting oleyl alcohol and epichlorohydrin in a ratio of 1:1.1 equivalents. Based on the experiments conducted, this synthesis strategy succeeded in facilitating the formation of new products as indicated by the results of the TLC test (Figure 1). Chromatogram of TLC in the crude reaction shows a new stain on top of the oleyl alcohol stain. Indicates that the resulting product is more polar than the substrate oleyl alcohol. To determine whether the product resulting from the reaction is oleyl glycidyl ether, the product was confirmed using ^{1}H NMR. NMR measurements of substrates, reagents, and products were carried out by comparing the 1H NMR spectra of the measurements. Based on the measurement results,

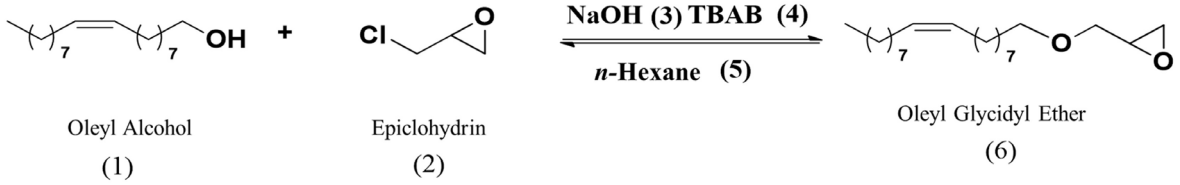


Figure 2. First step reaction to the synthesis of Epoxy Cyclic
(This first step reaction has the function to make the reactivity of the -OH group continue the sulfonation process)

n-Hexane : Ethyl Acetate (8 : 2)

Figure 3. TLC Chromatogram of the Etherification Reaction of Oleyl Alcohol and EPH (Chromatogram TLC from the first step reaction shows Oleyl Alcohol reacted with EPH to form a new product, which is predicted to be Oleyl Glisicdyl Ether. This is proven by the difference in polarity, where OGE is more polar than OG.

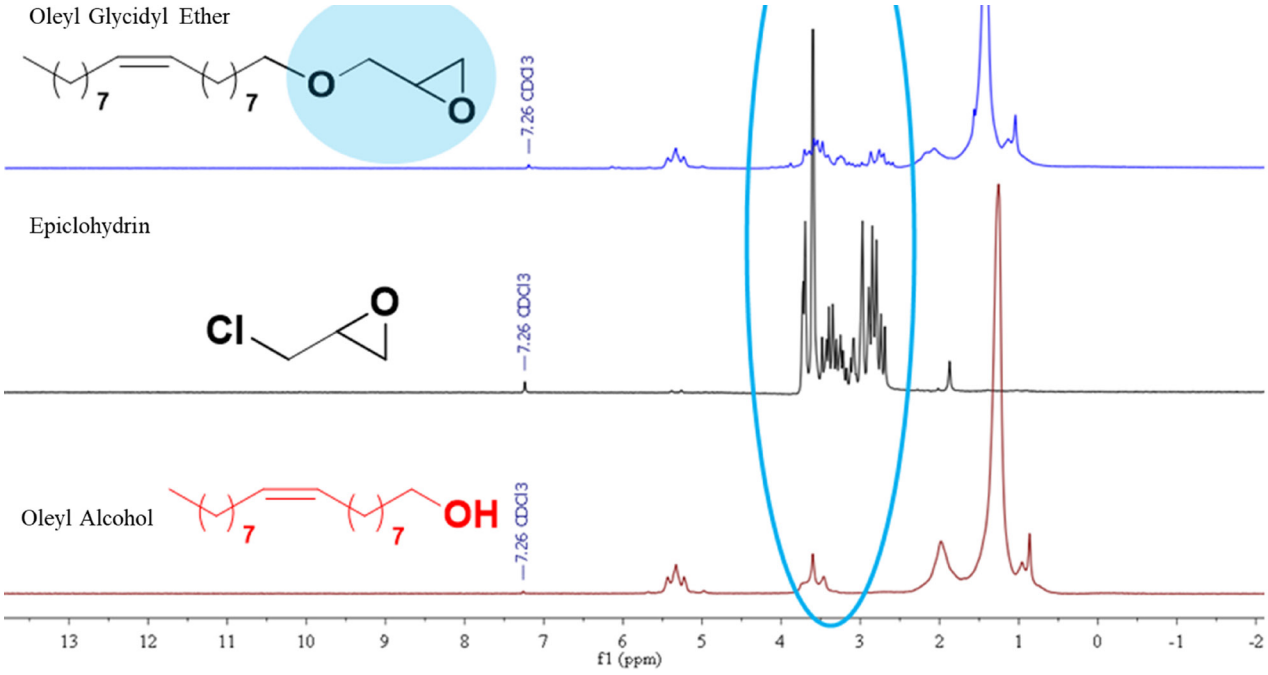


Figure 4. Spectrum of 1H NMR etherification reaction of oleyl Alcohol and epiclorohydrin (EPH).

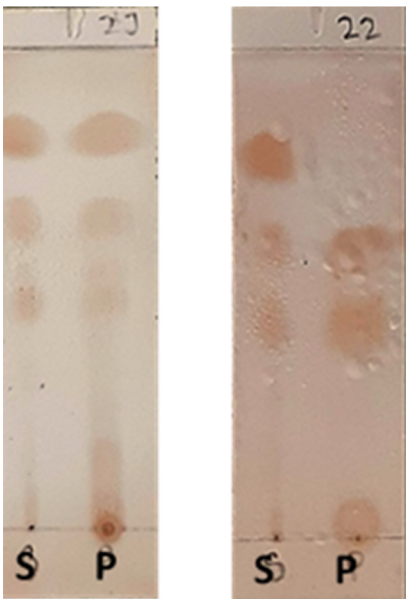
the 1H NMR spectrum of the product shows a spectrum in the chemical shift (δ) = 2.5 ppm – 4 ppm, which indicates the formation of an epoxy ring on the oleyl alcohol chain (Figure 2). The optimization of the reaction was carried out by comparing the equivalent of the epichlorohydrin used to the oleyl alcohol and the reaction catalyst and phase transfer catalyst used. Optimization begins with variations in the concentration of epiclohydrin to oleyl alcohol 1:1.5, 1:2.5, and 1:3.5. For the next step, the first product of Oleyl Glycidyl Ether (OGE) at an equivalent ratio of 1:2.5 was chosen as the optimum condition with few by-products. Furthermore, the OGE product was continued with the Sulfonation reaction using the ratio of sodium bisulfite and sodium sulfite. This reaction is gentler than using oleum or SO3 gas in the sulfonation reaction. So that this method becomes an alternative in the laboratory to carry out sulfonation reactions. The reaction begins with the ratio of OGE at a concentration of 1 M using water as a solvent. The ratios of sodium

bisulfite and sodium sulfite are 1:1, respectively. For the next step, the first product of Oleyl Glycidyl Ether (OGE) at an equivalent ratio of 1:2.5 was chosen as the optimum condition with few by-products. Furthermore, the OGE product was continued with the Sulfonation reaction using the ratio of sodium bisulfite and sodium sulfite. This reaction is gentler than using oleum or SO_3 gas in the sulfonation reaction. So that this method becomes an alternative in the laboratory to carry out sulfonation reactions. The reaction begins with the ratio of OGE at a concentration of 1 M using water as a solvent. The ratios of sodium bisulfite and sodium sulfite are 1:1, respectively.

Observations of product formation using TLC showed the formation of products that had started to form after 2 hours of reaction at Rf=0 (Figure 6). The product formed with Rf = 0 is estimated to be Oleyl Glycidyl Ether Sulfonate (OGES) because it is very polar, so it is not eluted at all using n-Hexane-EA eluent. Then the reaction was continued for up to 22 hours. After 22 hours, the reaction showed the formation of OGES products up to 100% with a marked absence of substrate stains on the crude reaction stain on TLC. The next product is confirmed using ^{1}H NMR at 43 MHz.

The results of the ^{1}H NMR measurement showed a change in the chemical shift (δ) = 2.5 ppm – 3 ppm, and in the substrate spectrum, there was a peak in the chemical shift area. Chemical shift (δ) = 2.5 ppm – 3 ppm is a chemical shift that indicates the presence of an epoxy ring on the OGE substrate (Figure 5). The absence of a peak in this area on the product spectrum indicates an open epoxy ring, and it is possible that the sulfonation process has taken place. This can also be seen from the TLC profile of the product compound, which is below with Rf = 0 using a normal phase plate with n-Hexane-Ethyl Acetate as eluent (9:1).

Furthermore, the optimization of the reaction was carried out by increasing the ratio of sodium sulfite and bisulfite to 1:2 and 1:2.5, Equivalent to OGE. The formation of the products and the rest of the reactants was controlled using the TLC technique. The results of the experiment showed that after 5 hours of reaction, all the rest of the substrate had reacted, both in a 1:2.5 and 1:2 ratio. Thus, the optimum condition of the ratio of Sodium Bisulfite and Sulfite is 1:2.



Reaction after 2 and 22 hours
Eluent n-hexane:ethyl acetate 9:1 with 2x elution
(S: substrate ; P: product)

Figure 6. TLC chromatogram of the sulfonation of OGE
(Plate A: Sulfonation of OGE after 2 hours reaction, Plate B: Sulfonation of OGE after 22 hours reaction, and no substrate stain in the crude reaction indicates that this reaction is completely reacted)

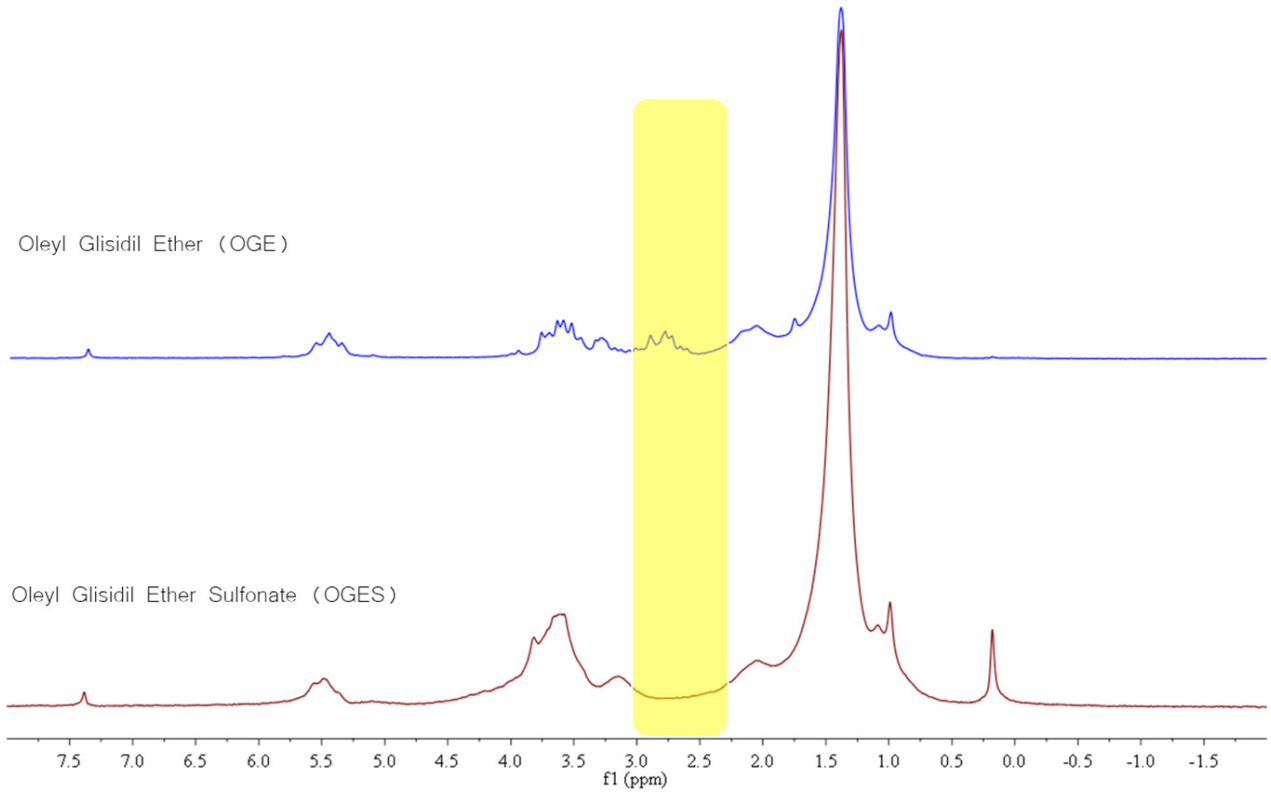


Figure 7. Spectrum of 1H NMR from Sulfonation of OGE
(The 1H NMR-OGE spectrum shows a chemical shift indicating that the cyclic epoxy compound in the OGE compound has been sulfonated to OGES. This is indicated by a chemical shift at 2.25 - 3.00 ppm, confirming that the cyclic epoxy compound was sulfonated.)

Compatibility test

Compatibility test is a test of surfactant solubility against injection water, which will be used in dissolving surfactants for field applications. In laboratory testing, water is prepared by taking into account the hardness and salinity of the field injection water to be studied. In this study, the solubility test was carried out using water with a salinity (NaCl) of 18000 ppm. The results of the experiment are presented in Table 1.

The compatibility test does not have a criterion value that has a threshold value, but insoluble and precipitated surfactants are considered not to meet the requirements in compatibility testing (Sheng 2011). Based on observations, OGES has cloud solubility but no precipitate. The higher the concentration of OGES surfactant, the more turbid the solution will become until it looks like milk. Up to a concentration of 1% W/W, OGES surfactant is still stable in the form of a single phase without forming a precipitate.

Interfacial tension (IFT) measurement

Synthesized surfactants at optimum conditions were tested for IFT values to see their ability to reduce the interfacial tension of water and oil. The interfacial tension is generally defined as the attraction force between the molecules existing at the interface of two fluids. The interfacial tension between hydrocarbons and water molecules causes an increase in the capillary force that plays an essential role in hydrocarbon trapping within porous media. Therefore, surfactant injection is used as an option to reduce the interfacial tension. Studies showed that many types of surfactants can be used at low concentrations (~0.05 0.2%) to achieve low interfacial tension on the order of 10−2 dynes/cm or less (Ge and Wang 2015; Hosseinzade et al. 2016). The test results showed that single surfactant OGES was only able to reduce the surface tension to a value of 10^{-2} dyne/cm (Figure 6).

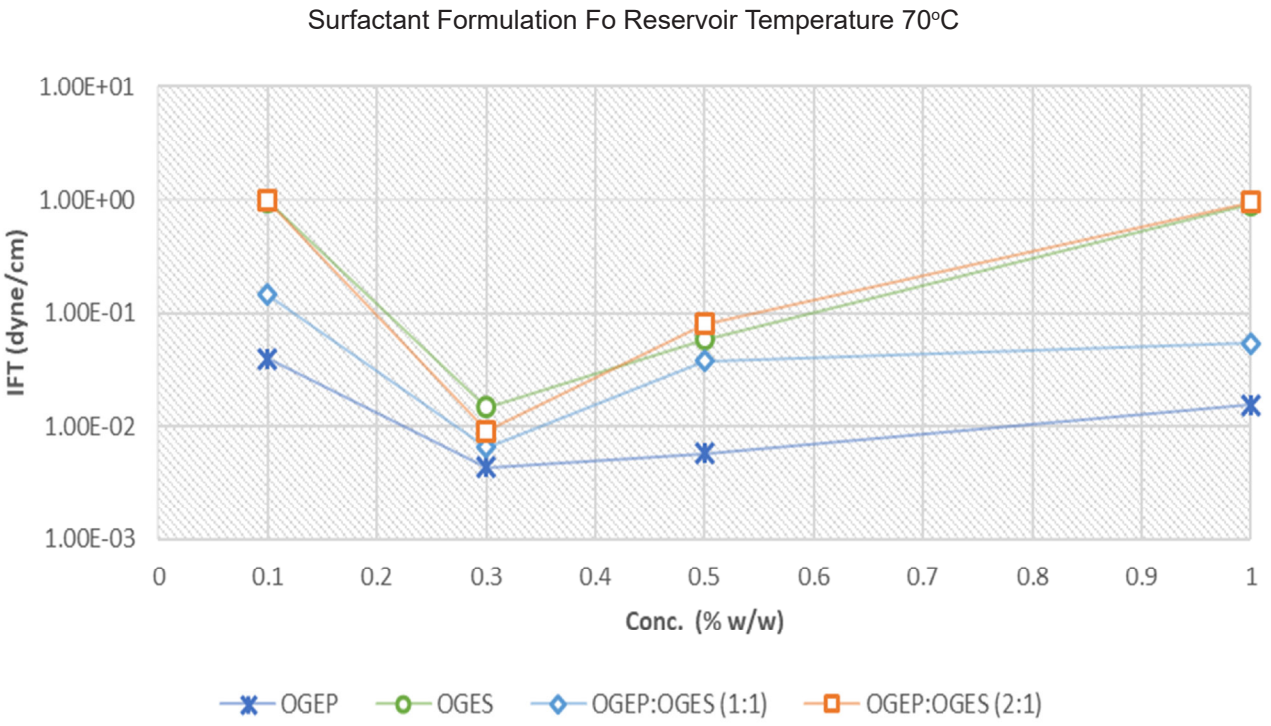


Figure 8. IFT values for CMC surfactants OGEP, OGES, OGEP:OGES = 1:1 (OFTS1), and OGEP:OGES=2:1 (OFTS2). (Surfactants OGEP, OGES, OFTS1, and OFTS2 have an optimum concentration based on IFT measurements of 0.3% W/W to obtain CMC. Surfactants and Surfactant Formulations that can achieve ultra IFT in OGEP, OFTS1, and OFTS2)

Furthermore, the OGES surfactant is a nonionic surfactant that has a polyoxy group with an ether functional group (OGEP). Furthermore, the formulation of surfactants OGEP and OGES with a ratio of 1:1 and 1:2. From the experiments carried out, it was found that the two surfactant formulas were able to reduce the IFT value up to 10-3 dyne/cm at a concentration of 0.3% w/w (Sheng 2011; Mahendrajaya et al. 2025).

CONCLUSION

In general, this research has successfully synthesized sulfonate compounds using the stechkerization technique. The resulting palm oil-based surfactant, with chemical structure modification, demonstrated activity with resistance to salinity and reservoir temperature. This refutes the notion that compounds made from non-petroleum raw materials cannot be used in petroleum applications. Surfactants derived from palm oil as a raw material offer significant advantages due to the availability of a good feedstock and Indonesia's significant palm oil production. While the challenges of converting palm oil directly into fuel or surfactants create different perspectives, both perspectives are expected to continue to be developed. From a surfactant perspective, the use of surfactants as a raw material for surfactant production still offers advantages, as many intermediate raw materials derived from petroleum mixtures remain difficult to produce, both in terms of cost and the synthesis methods for these intermediate compounds.

The synthesis of Oleyl Glycidyl Ether Sulfonate surfactant consists of two reaction steps. The first stage is the formation of an epoxy ring, and the second stage is the sulfonation process using sodium bisulfite and sulfite. The optimum conditions for the first reaction were obtained by varying the equivalent of oleyl alcohol and EPH 1:1.5 equivalents. For the sulfonation reaction of oleyl glycidyl ether with a ratio of sodium bisulfite and sulfite in a ratio of 1:2. The synthesized OGES surfactant, which was tested at 70 °C, 18000 ppm salinity, and oil with 34.39 °API characteristics, had a surface tension drop of up to 10-2 dyne/cm. Furthermore, the surfactant formulations of OGES and OGES resulted in a decrease in surface tension up to 10-3 dyne/cm. The best formula from OGES and OGEP is a ratio of 1:1 with a concentration of 0.3 %w/w. Based on the results of surfactant screening from fluid-to-fluid

analysis, one of the surfactant formulations yielded promising results, indicating that it can be applied from fluid-to-rock analysis. The results of the rock-to-fluid test and the core flooding test will fully demonstrate the performance of the synthesized and formulated surfactant formulation.

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GLOSSARY OF TERMS

Symbol	Definition	Unit
EOR	Enhanced Oil Recovery	
OGE	Oleyl Glycidyl Ether	
EPH	Epiclorohydrin	
IFT	Interfacial Tension	Dyne/cm
IFT	Interfacial Tension	Dyne/cm
CMC	Critical Michelle Concentration	Dyne/cm
RF	Recovery Factor	%
OFTS1	OGEP : OGES = 1:1	
OFTS2	OGEP : OGES = 2:1	

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