The Comparison of Surfactant from Palm Oil and Stearic Acid to Improve Thermal Stability

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ABSTRACT

The most efficient enhanced oil recovery (EOR) method is chemical EOR (CEOR) using surfactants. Indonesia has successfully produced biodegradable surfactants from palm oil; however, these surfactants have the disadvantage of being less stable at high temperatures (above 60°C). The aim of this research is to improve the thermal stability of surfactants by selecting raw materials that have higher saturated fatty acid content, thus preventing the surfactants from easily degrading at high temperatures. The synthesized surfactants were characterized using various analytical methods, including FTIR, EDX, IFT, and TGA. The results of FTIR analysis indicated the presence of sulfonate groups in the 1160-1120 cm-¹ range, while EDX analysis confirmed the presence of elemental sulfur, indicating that the sulfonation process was successful. IFT analysis revealed that the palm oil-based surfactant (surfactant 1) had a lower IFT value than the stearic acid-based surfactant (surfactant 2). The IFT values obtained at concentrations of 0.1; 0.5 and 1% were 4.9 × 10^{-2}; 4.3 × 10^{-2} and 2.8 × 10^{-2} dyne/cm for surfactant 1 and for surfactant 2 were 5.8 × 10^{-2}; 5.4 × 10^{-2} and 4.5 × 10^{-2} dyne/cm, respectively. To assess their thermal stability, TGA analysis was performed, which showed that both surfactants have resistance to temperatures above the reservoir temperature. However, the stearic acid-based surfactants showed better thermal stability compared to the palm oil-based surfactants. The specific conditions of the reservoir and the needs of a particular application determine the selection between palm oil-based and stearic acid-based surfactants.

1. Introduction:

Enhanced oil recovery (EOR) is the final stage of crude oil recovery by injecting materials (chemicals or gas and/or heat) into the reservoir. The injected materials will increase the overall efficiency of the oil transfer by modifying the forces on the fluid to mobilize and produce oil [1]. The mechanisms that occur in the EOR process are a decrease in interfacial tension, oil swelling, reduction in oil viscosity, and changes in wettability. The EOR method chosen must be suitable for the nature and conditions of the reservoir and economically feasible [2]. EOR is generally classified into four categories: thermal, chemical, gas, and microbial applications. Of these four EOR methods, chemical-enhanced oil recovery is the most efficient [3].

Chemical enhanced oil recovery (CEOR) is a stage in crude oil extraction from reservoirs using chemical agents, and this process can recover about 40% of the remaining oil. Among the CEOR methods, the use of surfactants is considered the most beneficial and efficient process. However, the use of surfactants also has drawbacks, such as environmental damage. Therefore, many researchers are currently working to synthesize biodegradable and environmentally friendly surfactants derived from plant oils, such as palm oil [4], mahua oil [5], soap nut oil [6], linseed oil [7], coconut oil [8], and jatropha oil [9]. Currently, Indonesia has been able to produce environmentally friendly biodegradable surfactants, known as green surfactants, synthesized from palm oil. However, palm oil-based surfactants still have a drawback, which is their instability at reservoir temperatures of 60°C. Some studies have shown that the presence of saturated and unsaturated fatty acids in the oil can support the thermal stability of the surfactants [4]. A high content of saturated fatty acids can make the surfactant less susceptible to thermal degradation [8]. This study aims to improve the thermal stability of surfactants to prevent degradation at high temperatures by selecting raw material compositions that contain a higher proportion of saturated fatty acids. Surfactants derived from palm oil and stearic acid will be synthesized because they have different compositions of saturated fatty acids. Palm oil contains 49.3%–50.2% saturated fatty acids and 46.3%–51.9% unsaturated fatty acids [10]. On the other hand, stearic acid is a 100% saturated fatty acid. The resulting surfactants will then be characterized to determine their properties, such as FTIR, TGA, EDX, IFT and compatibility analysis.

2. Materials and Experimental Methods

i. Materials

The palm oil used in the synthesis of the surfactant was procured from the local market under the brand name of Bimoli. Stearic acid and potassium hydroxide of analytical grade were obtained from commercial sources, while the methanol of analytical grade was obtained from Anhui Fulltime Specialized Solvents & Reagents Co., Ltd. Sulfuric acid, chlorosulfonic acid, anhydrous sodium carbonate, sodium bicarbonate, and n-butanol were obtained from Merck, while pyridine and petroleum ether were obtained from PT Smart Lab Indonesia.

ii. Methods

The preparation of Fatty Acid Methyl Esters (FAME): Palm oil transesterification and stearic acid esterification reactions, in which FAME is produced as an intermediate product, have been studied. The transesterification or esterification process was carried out with a molar ratio of 1:40 (raw material : alcohol), temperature of 63°C, stirring speed of 350 rpm, catalyst amount of 0.5 wt% and reaction time of 1 h [11]. The palm oil, KOH base catalyst, and methanol were weighed. The KOH base catalyst and methanol were mixed in a round-bottom flask to dissolve and then palm oil was added. The reaction temperature was maintained at 63°C. As the reaction progressed, the mixture changed color to darker, indicating the formation of glycol. Next, the mixture was transferred to a separatory funnel and allowed to stand for 24 h. After this period, two layers can be distinguished: a light-yellow upper layer, which indicates the presence of esters, and a gray lower layer, which indicates the presence of glycerol, residual catalyst, and residual alcohol. The glycerol is then carefully removed from the separatory funnel by allowing it to flow through the bottom hole. After the entire glycerol was removed from the separatory funnel, the crude FAME 1 was purified using centrifugation at 2000 rpm to remove the remaining glycerol. Next, centrifugation was carried out with a series of washes using distilled water at 50°C, which was repeated two to three times until the pH reached neutral. After the washing was completed, the centrifugation was heated at 110°C with the help of a rotary vacuum device to remove the remaining water. In addition, an esterification reaction between stearic acid and methanol, catalyzed by H_2SO_4, is used to obtain FAME 2. The weighed stearic acid was first heated separately at 60-70°C in a round-bottom flask. Then methanol and H_2SO_4 are mixed. During the reaction, the temperature is kept at 63°C to ensure that the process takes place properly. After the reaction is complete, the heating process is carried out at 110°C using a rotary vacuum device. This procedure was implemented to facilitate the removal of water as a by-product.

The Preparation of Surfactant: The FAME obtained was then utilized in the synthesis of the sodium methyl ester sulfonate surfactant, with FAME 1 being employed to synthesize surfactant 1 and FAME 2 being used to synthesize surfactant 2. The sulfonation of surfactants 1 and 2 was achieved through a reaction with chlorosulfonic acid. Chlorosulfonic acid (2.63 g) was meticulously added to pyridine (30 ml) in a 250 ml round-bottom flask under cold conditions (0-3°C). Thereafter, FAME (2.6 g) was added and stirred for 30 min. After stirring, the mixture was heated at 65°C until a clear solution was obtained. The clear solution was subsequently mixed with a mixture of sodium carbonate and sodium bicarbonate to neutralize the highly acidic reaction. The mixture of sodium carbonate and sodium bicarbonate consists of 33 g of sodium bicarbonate in 300 mL of distilled water, mixed in a cold state (0-3°C), and an adequate amount of sodium bicarbonate to ensure that the solution remains saturated. The unreacted components were then dissolved in 40 mL of n-butanol. The n-butanol solvent was subsequently removed using a rotary evaporator. Thereafter, 40 mL of petroleum ether was employed to remove the organic impurities from the sulfonated product. The product was then dried under vacuum at 60°C for 24 h to obtain a white solid surfactant.

3. Analysis

3.1 FTIR

The identification of the functional groups contained in the synthesized FAME and surfactants was analyzed using Fourier Transform Infrared Spectroscopy (FTIR) Agilent Cary 630, Santa Clara, USA. Spectra were obtained using a spectral resolution of 4 cm^{-1} with a frequency range of 4000-700 cm^{-1}.

3.2 EDX

Energy-dispersive X-ray (EDX) was used for the chemical elemental analysis of the synthesized surfactants. The characterization capability is largely due to the basic principle that each element has a unique atomic structure that enables a unique set of peaks on its electromagnetic emission spectrum (which is the main principle of spectroscopy). EDX analysis using the HITACHI FLEXSEM 100.

3.3 IFT

The drop spinning technique was used to determine the IFT between the crude oil and surfactant solution at 27°C in an SVT20 tensiometer (Data Physics, Germany). A drop of crude oil was introduced into a capillary tube containing the surfactant solution and then allowed to spin at 3500 rpm. The experiment measured the IFT by fitting a profile of the oil drop under these conditions. The capillary tube was first rinsed with benzene before taking measures to remove any residual crude oil. Afterward, the capillary tube was cleaned with acetone to remove any residual salts/alkalis/surfactants and dried. The IFT analysis used light crude oil with a density at 25°C of 0.811 g/cm^3 and API Gravity @60°C = 39.88.

3.4 TGA

The synthesized surfactants were analyzed using a Thermo Gravimetric Analyzer (TGA) (Netzch-STA 449 Jupiter) to determine the decomposition temperature of the surfactants.

3.5 Compatibility

Compatibility analysis was conducted by preparing surfactant solutions with concentrations of 0.1%; 0.5%, and 1% dissolved in formation water with a concentration of 18,000 ppm. After the solution was obtained, it was stored for 7 days at room temperature and 60°C. Then, observations were made on the solution.

4. Result And Discussions

i. FTIR analysis of FAME and Surfactant (CEOR)

The synthesis of the surfactants in this study occurred in two primary stages. The initial stage involved the formation of the hydrophobic tail of the surfactant through transesterification or esterification reactions, depending on the type of raw material used. For instance, when palm oil, a triglyceride, was employed as the raw material, a transesterification reaction was necessary. Conversely, if the raw material is stearic acid, which is classified as a carboxylic acid, an esterification reaction is necessary [11]. In both reactions, methanol was used as the alcohol, resulting in the intermediate product fatty acid methyl ester (FAME). Subsequently, the second stage involves the formation of a hydrophilic surfactant head, in which the FAME produced in the initial stage undergoes a sulfonation reaction with chlorosulfonic acid. This process culminates in the production of the surfactant as the ultimate product.

After synthesis, the FAME and surfactant obtained were characterized to evaluate their quality. One of the characterization methods used is FTIR (Fourier Transform Infrared Spectroscopy), which aims to detect the presence of ester functional groups in FAME and sulfonate functional groups in surfactants. The synthesis process is considered successful if both functional groups are detected.

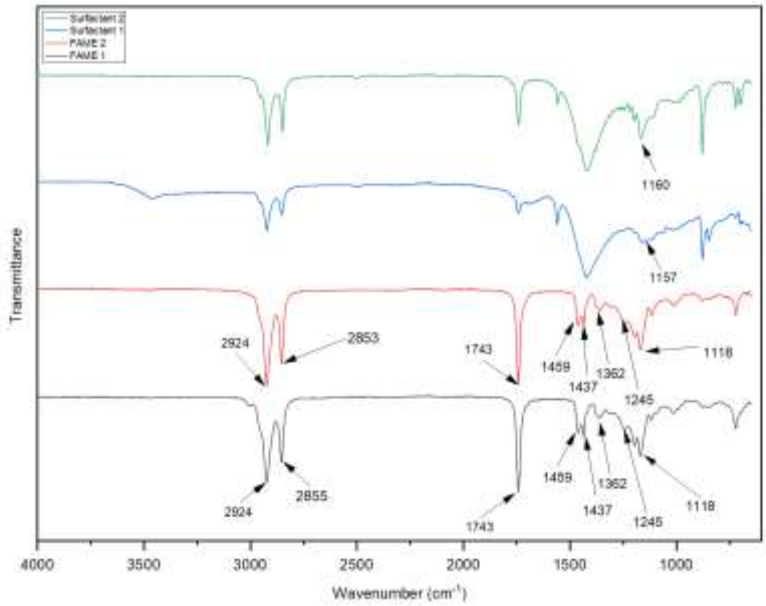

Figure 1. FTIR spectrum of FAME and Surfactant (CEOR)

FAME 1 was synthesized from palm oil and FAME 2 was synthesized from pure stearic acid, showing the same FTIR spectrum as shown in Fig. 1. A peak at 2924 cm^{-1} is present in both FAME, a peak at 2855 cm^{-1} in FAME 1, and a peak at 2853 cm^{-1} in FAME 2, all of which fall within the 3300–2500 cm^{-1} range, representing the O–H stretching of carboxylic acids and the 3000–2850 cm^{-1} range, representing the C–H stretching of alkanes. A peak at 1743 cm^{-1} is observed in FAME within the 1735–1750 cm^{-1} wavelength range, indicating the presence of a C=O bond stretching vibration, which signifies the presence of methyl ester. Additionally, the peaks at 1459 cm^{-1}, 1437 cm^{-1}, and 1362 cm^{-1} in FAME 1 and FAME 2 fall within the 1480–1350 cm^{-1} range, indicating C-H alkyl bending vibrations, which further support the presence of alkyl groups in the FAME structure. The peaks at 1245 cm^{-1}, 1196 cm^{-1}, and 1118 cm^{-1} in both FAMEs fall within the 1320–1000 cm^{-1} range, corresponding to the C–O stretching of the methoxycarbonyl group in the FAME [12]. Fig. 1 also shows the FTIR spectra of surfactant 1 and surfactant 2. The spectra are almost identical to those of the FAME, with the only difference being the peaks at 1157 cm^{-1} for surfactant 1 and 1160 cm^{-1} for surfactant 2, within the 1160–1120 cm^{-1} range, indicating the stretching vibration of the O=S=O bond, which signifies the presence of the sulfonate group in the surfactant molecules [4]. The various peaks obtained in the FTIR spectra are discussed in Table 1.

Table 1. The characteristic adsorption bands correspond to different functional groups in the surfactant (CEOR).

Wavelength (cm^{-1})	Group	Class
3300–2500	O–H Stretching	Carboxylic Acid
3000–2850	C–H Stretching	Alkane
1735–1750	C=O Bending	Methyl Ester
1480–1350	C-H Bending	Alkyl
1320–1000	C–O Stretching	Methoxycarbonyl
1160–1120	O=S=O Stretching	Sulfonate

ii. EDX analysis of the surfactant (CEOR)

In this study, each type of surfactant successfully produced contained sulfonate groups that played an important role as water binders. The sulfonate group is one of the main functional groups in the surfactant structure that contributes to its hydrophilic nature, thus supporting the surfactant's ability to reduce the interfacial tension between two immiscible liquid phases, such as water and oil. The presence of sulfonate groups in the surfactants produced was proven through EDX analysis, where the data obtained showed the presence of elemental sulfur (S) in the chemical composition of each surfactant sample. This sulfur element specifically indicates the presence of sulfonate groups, considering that sulfur is one of the main atoms that make up the sulfonate functional group. This result is an indication of the success of the surfactant synthesis in accordance with the research objectives. Figure 2 shows the EDX results for all the surfactants.

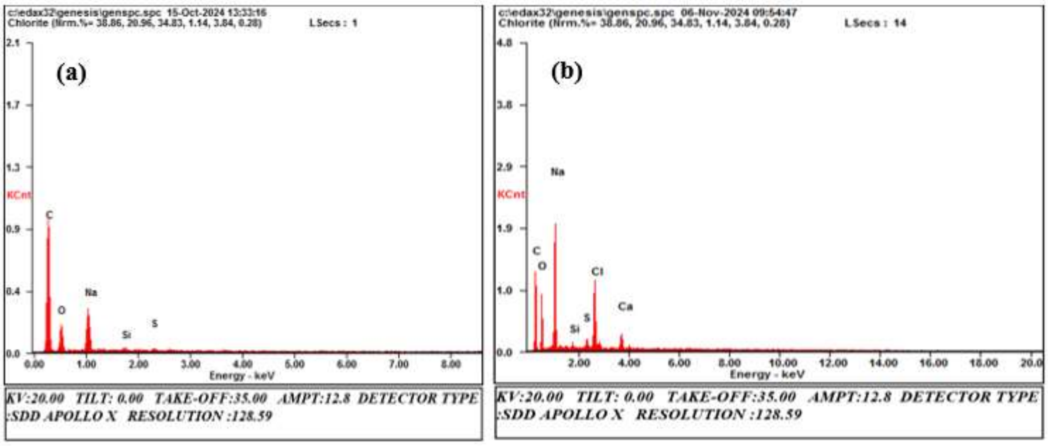

Figure 2. EDX of (a) Surfactant 1 and (b) Surfactant 2 (CEOR)

These results agree with the research conducted by [4], which showed that the surfactants produced contain elemental sulfur (S) as part of the sulfonate group. In that study, sulfonate groups were formed through a sulfonation process using chlorosulfonic acid, which directly added sulfonate functional groups to the surfactant structure. This process results in a surfactant with enhanced hydrophilic properties, supporting its ability to act as an emulsifying or surface tension-lowering agent. These findings confirm the importance of elemental sulfur as an indicator of the success of sulfonation reactions while reinforcing the role of chlorosulfonic acid in the synthesis of effective and fit-for-purpose surfactants.

iii. The IFT analysis of the surfactant (CEOR)

The main parameter of CEOR surfactants is the interfacial tension (IFT) value. The lower the resulting IFT value, the more effective the performance of the surfactant. To improve oil recovery, the IFT should be reduced to the range of 10^{-2} dyne/cm [13]. Fig. 3 shows that the IFT values for surfactant 1 at concentrations of 0.1%, 0.5%, and 1% are 4.9×10^{-2}, 4.3×10^{-2}, and 2.8×10^{-2} dyne/cm, respectively, while for surfactant 2 at the same concentrations, the values are 5.8×10^{-2}, 5.4×10^{-2}, and 4.5×10^{-2} dyne/cm. The IFT value of the synthesized surfactant met the standard, which is in the range of 10^{-2} dyne/cm [14]. Based on previous research that synthesized surfactants from vegetable oils, IFT values of up to 10^{-2} dyne/cm were obtained. Research conducted by (Saxena et al., 2017) who synthesized surfactants from palm oil obtained an IFT value of 1.17×10^{-2} dyne/cm [4], (Saxena, et al., 2019) synthesizing surfactants from mahua oil produced an IFT value of 3×10^{-2} dyne/cm [5], (Saxena et al., 2018) from soap-nut produced an IFT value of 2.123×10^{-2} dyne/cm [15], and surfactant from coconut oil produced an IFT value of 1.15×10^{-1} dyne/cm [8]. It can be observed that the two surfactants produced lower IFT values as the surfactant concentration increased. The decrease in the IFT value occurs due to the increased adsorption of the surfactant at the water-oil interface. The surfactant molecules are adsorbed such that the hydrophobic carbon chains are in the oil phase, while the hydrophilic head groups are in the water phase. The interaction between the carbon chains and the oil component contributes to the reduction of the IFT value. As the concentration of the surfactant in the water phase increases, the diffusion of the surfactant molecules from the solution to the water-oil interface also increases. The minimum IFT was measured at the concentration where the surfactant adsorption rate was equal to the desorption rate. Furthermore, an increase in the surfactant concentration can reduce the interfacial tension between the oil and the formation water, thereby increasing the solubility of the oil phase in the formation water. Strong oil solubility and very low interfacial tension (IFT) are essential for stable microemulsions and effective oil recovery (EOR) [16].

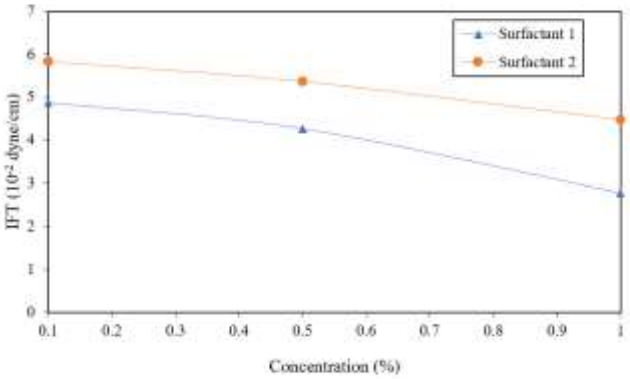

Figure 3. IFT Analysis of Surfactants (CEOR)

As demonstrated in Figure 3, the IFT value of surfactant 1, synthesized from palm oil, is lower than that of surfactant 2, synthesized from stearic acid. This discrepancy can be attributed to the higher proportion of unsaturated fatty acids in surfactant 1 compared with that in surfactant 2. The solubility of surfactants in water and their surface-active properties are known to increase with the degree of unsaturation of their aliphatic chains (Gang et al., 2020). Unsaturated fatty acids are found predominantly in the form of cis bonds, and their molecular arrangement is less compact compared with that of saturated fatty acids (Gang et al., 2020). Research by Gang et al. (2020) has shown that the surface-active properties of pure fatty acids, including palmitic acid, stearic acid, linoleic acid, and ricinoleic acid, as well as their mixtures, are higher in unsaturated fatty acid-based surfactants than in saturated fatty acid-based surfactants [17].

iv. TGA analysis of the surfactant (CEOR)

TGA analysis of the surfactant was conducted to determine the surfactant's resistance to reservoir temperatures. According to data from the Oil and Gas Technology Research and Development Center (LEMIGAS), the typical reservoir temperature in Indonesia is 60°C, so the synthesized surfactant must be able to withstand this temperature. Based on Fig. 4, Surfactant 1 decomposed at 240-300°C with a mass loss of 20% and then experienced further decomposition at 300-500°C with a mass loss of 30%. Surfactant 2 decomposed at 270-320°C with a mass loss of 12% and then experienced further decomposition at 320-500°C with a mass loss of 36%.

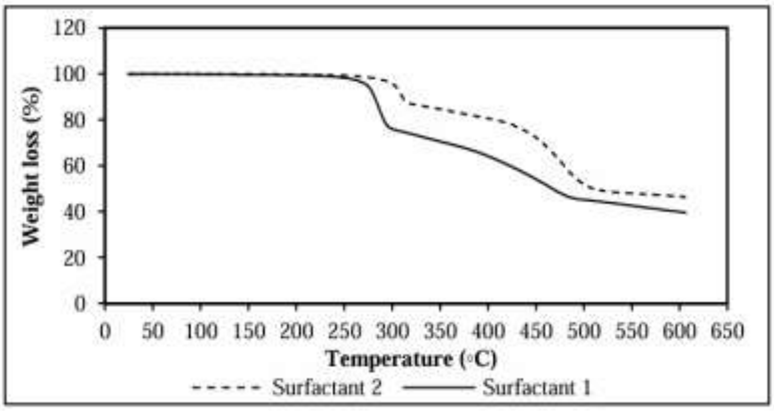

Figure 4. TGA Analysis of Surfactants (CEOR)

The surfactants obtained decomposed above 60°C, indicating that the surfactants could survive at reservoir temperatures. The good thermal stability of the surfactants is due to the presence of fatty acids in the surfactant compounds. Research conducted by (Saxena et al., 2017) stated that the presence of saturated and unsaturated fatty acids in palm oil supports the good thermal stability of α-SEE surfactants [4], because fatty acids require high temperatures for degradation. According to (Goswami, et al., 2019), the content of long-chain fatty acids acts as an antioxidant and prevents oxidation into free radicals at high temperatures [6].

Surfactant 1 starts to degrade at 300°C, while surfactant 2 degrades at 320°C, which indicates that surfactant 2 is more resistant to high temperatures than surfactant 1. This is because surfactant 2 contains higher saturated fatty acids than surfactant 1, which only contains 50% saturated fatty acids. Saturated fatty acids provide thermal stability to the synthesized surfactant [10]. Therefore, fatty acid composition is an important criterion when selecting vegetable oils as raw materials for surfactant synthesis. Based on research conducted by (Pal et al., 2018), surfactant synthesized from palm oil containing 86.32% saturated fatty acids began to degrade at 100 ° C [8], research conducted by (Saxena et al., 2017) from palm oil with a saturated fatty acid content of 50.3% surfactants degraded starting at 96 ° C, research by (Saxena, et al., 2019) which synthesized surfactants from mahua oil with a saturated fatty acid content of 32.54% began to degrade at 80°C [4], and research by (Nafisifar et al., 2021) synthesizing surfactants from linseed oil with a saturated fatty acid content of 11% surfactants degraded starting at 25°C [7]. This research corroborates the basis that the higher saturated fatty acid content of the surfactant makes the surfactant more stable at high temperatures because saturated fatty acids have high packing (due to single carbon-carbon bonds that produce straight hydrocarbon chains), so they are in solid or semi-solid form at room temperature [10].

v. Compatibility of the Surfactant (CEOR)

A compatibility test was conducted to assess the ability of the surfactant to dissolve in the formation water, which is a key factor in ensuring that the surfactant can function effectively in the injection process, as well as identifying potential problems that may arise due to insolubility, such as clogging of the reservoir pores [17]. The formation water used was brine with a concentration of 18,000 ppm [18]. This study tested the compatibility of all surfactants with concentrations of 0.1%; 0.5%, and 1% at 25°C and 60°C. Based on Fig. 5, which shows the results of the compatibility test, only surfactant 2 at a concentration of 0.1% produced a clear solution, while the other surfactants produced a cloudy solution. However, all surfactants did not show signs of precipitation or phase separation, indicating good compatibility. Based on research conducted by Zulkifli et al. (2020), the methyl ester sulfonate surfactant was proven to be insoluble in the formation water [19]. In addition, another study by Eni et al. (2017) showed that the same surfactant produced a cloudy solution and precipitated in seawater. This is due to its sensitivity to the divalent ions present in saltwater [18]. Sulfonate surfactants are very sensitive to the presence of divalent ions such as calcium (Ca^{2+}) derived from NaCl in water. These ions can interact with the sulfonate group, causing the precipitation of the surfactant and reducing its solubility. When divalent ions are present, they can form stronger bonds with sulfonates compared to the interaction of sulfonates with water, resulting in insolubility [20].

Figure 5. Compatibility Surfactant (CEOR)

5. Conclusion

This study compares surfactants derived from palm oil and stearic acid to enhance the thermal stability in Chemical Enhanced Oil Recovery (CEOR). While surfactants play a crucial role in CEOR by reducing interfacial tension (IFT) and improving oil recovery, those based on vegetable oils like palm oil often struggle with thermal stability at high temperatures. This research aims to improve the thermal stability by using raw materials with higher saturated fatty acid content. Surfactants were synthesized through transesterification, esterification, and sulfonation and then analyzed using FTIR, EDX, TGA, and IFT. FTIR confirmed the presence of functional groups, indicating successful surfactant formation. EDX analysis showed the sulphur content, proving the presence of sulfonate groups essential for hydrophilicity. The TGA results indicated that the stearic acid-based surfactants were more thermally stable than the palm oil-based ones, with degradation starting at higher temperatures. IFT measurements revealed that palm oil-based surfactants exhibited lower IFT values than those derived from stearic acid, making them more effective in oil recovery applications. The findings suggest that while palm oil-based surfactants are superior in reducing IFT due to their higher unsaturated fatty acid content, stearic acid-based surfactants offer better thermal stability due to their fully saturated composition. Therefore, the choice between these surfactants depends on the reservoir conditions and the need for either enhanced thermal stability or lower IFT values.

Acknowledgments

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