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Surfactant Flooding for EOR Using Sodium Lignosulfonate Synthesized from Bagasse

Rini Setiati, Septoratno Siregar, Taufan Marhaendrajana and Deana Wahyuningrum

Abstract

Surfactant injection is one of the types of chemical injections used in enhanced oil recovery (EOR) process. Surfactant can increase the interfacial tension between oil and water in the rock matrix. The surfactant used is an anionic surfactant, which is one of the lignosulfonate surfactants known as sodium lignosulfonate (SLS) surfactant derived from lignin. Bagasse is one of the raw materials having a high content of lignin (24–25%). The synthesized bagasse becomes lignosulfonate used as an isolation of lignin and transformed into sodium lignosulfonate by sulfonation process. Based on the characteristic test, the bagasse's SLS surfactant has some qualified characteristics which are a good aqueous stability, clear solution and not causing turbidity, and capability to form middle-phase microemulsion with light oil. Synthesized SLS has a hydrophilic–lipophilic balance (HLB) value of 11.6 which can be classified as oil in water (O/W) emulsion. Middle-phase emulsion as a characteristic SLS surfactant affects the performance of the SLS surfactant injection. So the use of sodium lignosulfonate surfactant synthesized from bagasse is a challenge to be developed further as a surfactant flooding.

Keywords: adsorption, bagasse, enhanced oil recovery, light oil, middle-phase emulsion, recovery factor, sodium lignosulfonate surfactant

1. Introduction

Enhanced oil recovery is the process of increasing oil recovery by injecting liquid into a reservoir where the fluid can change the physical properties of oil that is still trapped inside it. One method used is chemical injection, using an anionic surfactant. Surfactants have the ability to increase the interfacial tension between oil and water in the rock matrix. The type of surfactant that is used for the EOR is lignosulfonate surfactant known as sodium lignosulfonate surfactant derived from lignin. One of the raw materials having a high content of lignin is bagasse, which contains 24–25% lignin.

The bagasse process becomes lignosulfonate becoming lignosulfonate uses isolation of lignin from bagasse, and the sulfonated lignin becomes sodium lignosulfonate. For sulfonation process sodium bisulfite (NaHSO_3) as reagent can be used. This sulfonation aims to change the hydrophilicity of less polar lignin (insoluble in water) by substituting hydroxyl groups with sulfonate groups so that hydrophilicity increases [1].

In this study, lignin has been successfully isolated from dried bagasse, which was confirmed through the analysis results of Fourier-transform infrared (FTIR). The FTIR showed the spectrum of isolated lignin has the same characteristic within lignin standard, which contains substituted phenolic aromatic groups and aliphatic alkene groups [2]. Sodium lignosulfonate (SLS) surfactant from bagasse has the same characteristics within the sodium lignosulfonate standard, which comprises the absorption peaks corresponding with the stretching vibration of aromatic and aliphatic alkene functional groups, the stretching vibration of sulfonate groups, and the bending vibration of carboxylic groups [3].

Surfactants will spread in water and absorb at the interfacial tension between air and water or at the interfacial between oil and water. The surfactant is an amphiphilic organic compound, which contains a hydrophobic tail and a hydrophilic head. The aggregate shape may depend on the chemical structure of the surfactant, taking into account the balance of hydrophobic tail and hydrophilic head, known as HLB [4]. Hydrophilic and hydrophobic functions are to determine the emulsion of the system. The type of emulsion can be determined using the hydrophilic-lipophilic balance (HLB) test [5]. The value of HLB shows the tendency of surfactants to dissolve in water or oil to form O/W or W/O emulsion types. Low HLB indicates that the surfactant is more soluble in water, named W/O emulsion type. If the formation salinity is low, low HLB surfactant must be chosen, such that surfactants can make intermediate-phase microemulsions with low salinity. When the formation salinity is high, high HLB makes intermediate-phase microemulsions with high salinity. HLB is determined by calculating values for various molecular regions, giving results on a scale ranging from 0 to 20, for nonionic surfactants. The HLB value 0 corresponds to the total hydrophobic molecule, and the value 20 corresponds to a molecule consisting of a hydrophilic component. HLB values can be used to estimate the nature of surfactants. HLB calculations [6] can be determined by the following formula:

$$HLB = 20 * (M_h) / (M_l + M_h) \quad (1)$$

M_h = molecular weight of hydrophilic groups.

M_l = molecular weight of a hydrophobic or lipophilic group.

For surfactant classification as emulsifier in the O/W system, the surfactant should have a range of values between 8 and 18 **Tables 1 and 2** [6].

The definition of emulsion is a two-phase system in which one of the fluids is dispersed in another, in the form of small droplets [7]. There are two types of emulsions based on the type of liquid that function as internal or external phases, namely:

- a. Oil in water (O/W)-type emulsion, which is an emulsion consisting of oil grains scattered into the water
- b. Water in oil (W/O)-type emulsion, which is an emulsion consisting of water droplets which are dispersed into oil

Figure 1 explains about W/O emulsion or O/W emulsion. For W/O emulsion, the water is an internal phase and oil is an external phase. As for the O/W emulsion, the oil is an internal phase and water is an external phase.

Microemulsion can occur due to the equilibrium between hydrophiles (head) and lipophiles (tail) so that surface-active agents (surfactants) are formed and reduce interfacial tension. In systems with the same conditions between the number C belonging to crude oil and the surfactant components, there will be a balance between oil and water so that the hydrophilic component (head) of the surfactant

HLB range	General application
2–6	W/O emulsification
7–9	Wetting and spreading
8–18	O/W emulsification
3–15	Detergency
15–18	Solubilization

Table 1.
HLB value and its application [6].

Application	Range HLB
Defoaming of aqueous systems	1–3
detergency and cleaning	12–15
W/O emulsification	3–6
O/W emulsification	8–28
Solubilization	11–18
Wetting	7–9

Table 2.
Required HLB for a number of applications [4].

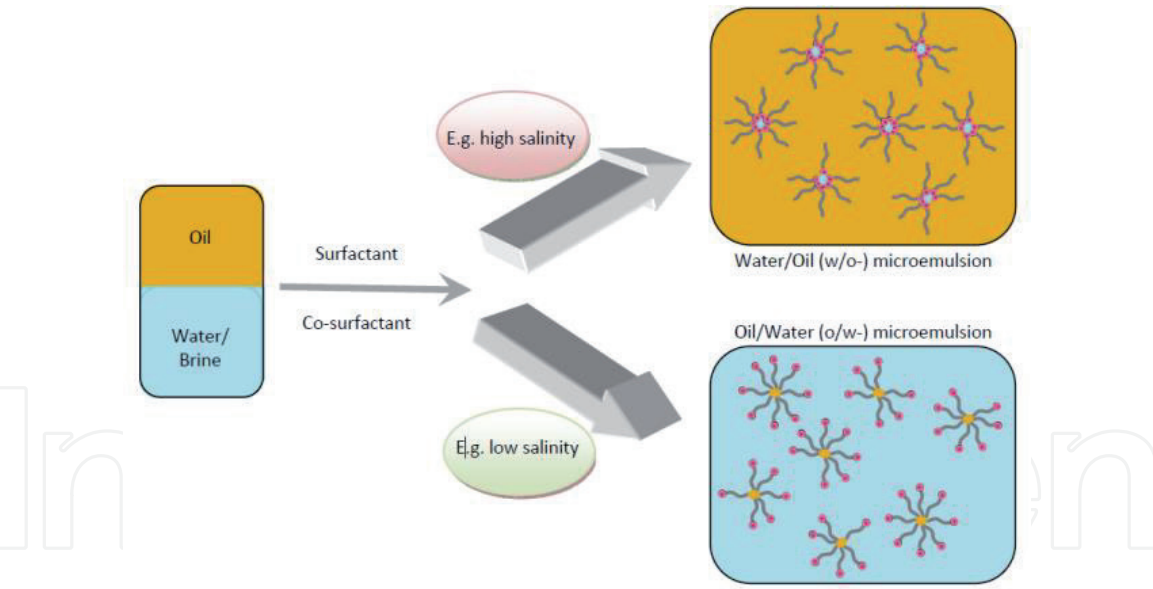


Figure 1.
Oil and water formation forming microemulsion [8].

will go to the surface and form a surface-active agent (surfactant). In a high-salinity oil-soluble surfactant system, there will be ionic strength that is identical to salinity where the hydrophilic (head) component of the surfactant is weak and the lipophile (tail) component is strong. So, in a system with high salinity, the hydrophilic component weakens, and the lipophilic component will be stronger so that a balance occurs in the system and a surface-active agent (surfactant) is formed. At high salinity, the salt component will make a balance between the components C of surfactant and oil, thereby strengthening the interaction between surfactants and oil. Salinity of the formation water affects the reduction of oil–water interfacial tension by surfactant.

The emulsion condition formed can be seen from the results of the phase behavior test, which is a test of mixing fluid reservoir with injection fluid.

Emulsions are made to obtain stable and flat preparations from mixtures which cannot mix together. The behavior of the mixed phase is strongly influenced by solvent water salinity, temperature, type and concentration of surfactant, and oil type. The type of emulsion is also determined by salinity, as shown in **Figure 2**. In the figure, the optimal emulsion is in the middle, where the emulsion occurs in the middle area between water (surfactant) and oil, called the middle-phase emulsion.

Observations of mixed-phase behavior between reservoir fluid and injection fluid can be classified as lower-phase emulsion, microemulsion (middle-phase emulsion), upper-phase emulsion, macroemulsion, and sediment. **Figure 3** can explain about the classification of emulsion. This test is carried out on surfactants dissolved in formation water and then mixed with crude oil. This test aims to get the right conditions for the surfactant by determining the exact salinity and type of emulsions that can mix with crude oil.

The emulsion formed indicates that in the system there is a decrease in interfacial tension between the surfactant and crude oil systems. This phase behavior test is strongly influenced by the salinity of formation water.

Increased salinity will reduce the solubility of anionic surfactants in formation water, because with increasing concentration, the surfactant electrolyte is released from the formation water system [9]. In the middle phase that forms microemulsions, it represents the condition of miscible displacement, whereas in the mixed phase which forms the upper-phase or lower-phase emulsion, it represents an immiscible displacement condition.

The emulsion formed can be an indicator of the IFT value of the system. A high IFT indicates an immiscible fluid, while a low IFT indicates a fluid is miscible.

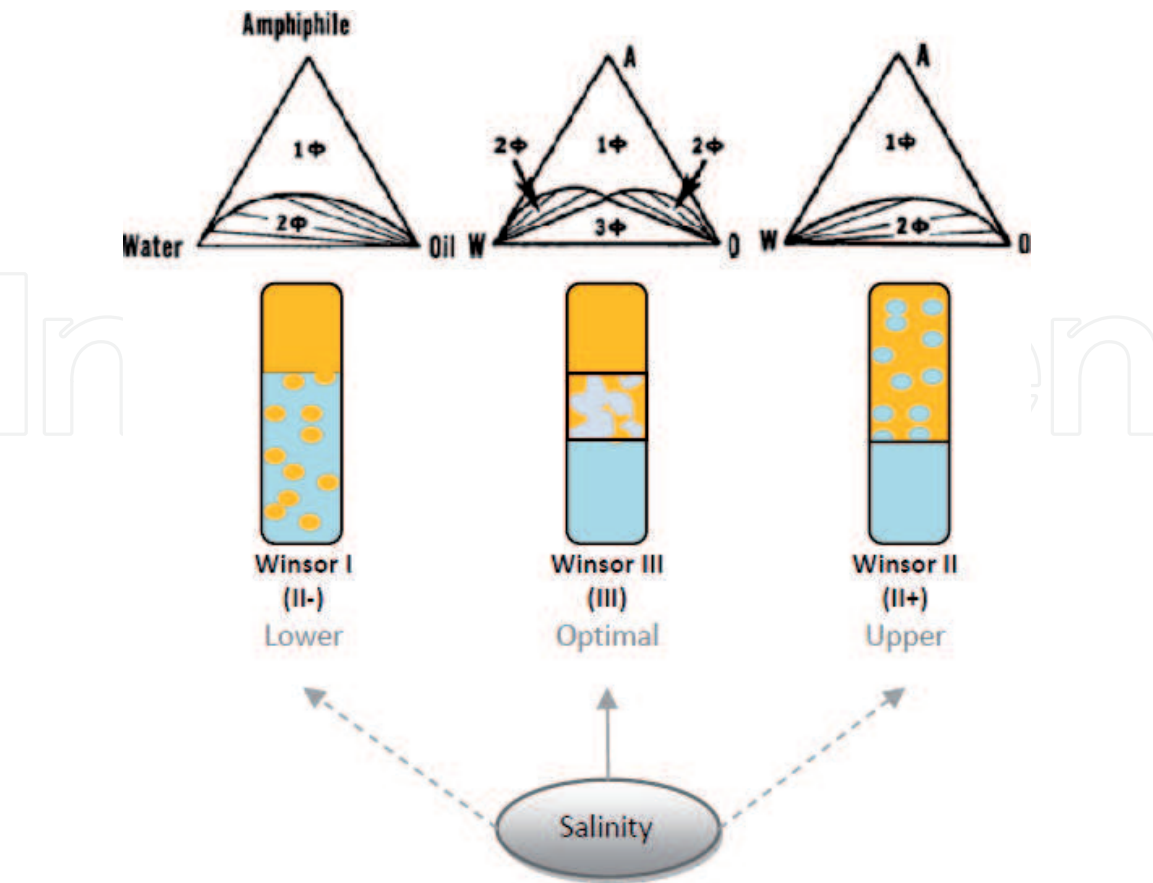


Figure 2.
Emulsion type based on salinity [8].

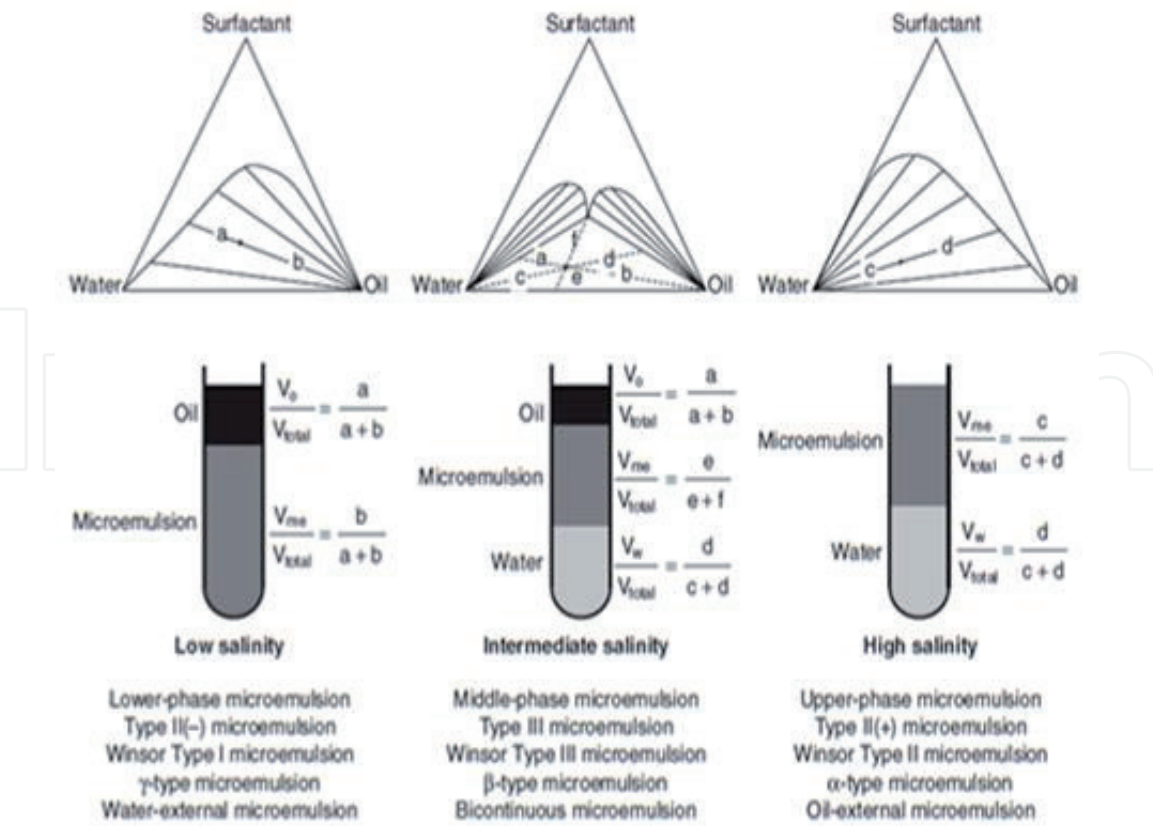


Figure 3.
Three types of microemulsion effect of salinity and phase behavior [5].

IFT at surfactant and oil is a function of formation water salinity, temperature, surfactant concentration, type of surfactant, and oil type, while the phase behavior test is influenced by the type of oil, type of co-surfactant, temperature, structure of surfactant, and pressure [5]. The behavior of the microemulsion phase is very complex and depends on a number of parameters. Surfactants can form microemulsions because of their level of solubility that are both in water and in oil. There is no equation for simple microemulsions. Therefore, the phase behavior for certain microemulsion systems must be measured experimentally. The phase behavior of microemulsions is usually presented using ternary diagrams and empirical correlations. The ternary diagram is a very useful tool in EOR because it can simultaneously represent the composition of phases and the whole and relative numbers. **Figure 4** shows a ternary diagram schematic.

In the ternary diagram, the equilateral triangle represents the components of water, oil, and 100% surfactant from the solution. The concentration can be shown in mole fraction, mass, or volume. The single-phase region is in the zone of high surfactant concentration. The three-phase region is in the middle zone. The two-phase lobes (vertices) are in the upper right and left over the three-phase triangle. There is a third two-phase region which is located at very low surfactant concentrations below the three-phase region. Type III ternary-phase behavior consists of an area close to the axis of salt water/oil which is bounded by a triangle. The composition in this area will produce three phases; the composition of each phase is the same as the composition of the curved triangular peak.

The microemulsion system can also be designed to obtain a very low IFT value, around 0.001 mN/m by making a stable brine solution or with a hydrocarbon phase [10] which will be advantageous for the EOR process. In the EOR process, an important part of the ternary diagram is the three-phase region. The general form of ternary diagram can be classified as type II (-), namely, lower-phase emulsion

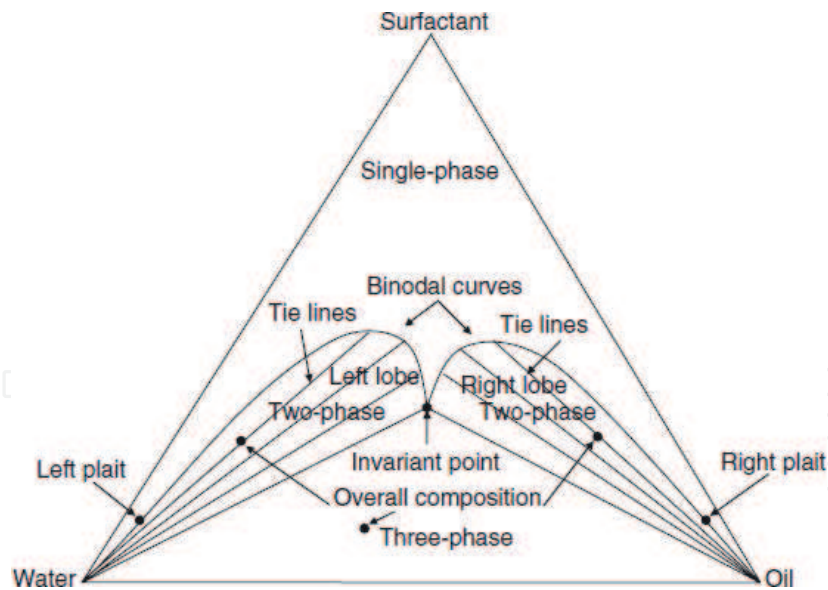


Figure 4.
Ternary diagram schematic [5].

and excess oil phase; type (+), namely, upper-phase emulsion with excess water phase; and type III, middle-phase microemulsion. The lowest interfacial tension occurs because the SLS surfactant can form a microemulsion.

The SLS surfactant from bagasse can function as an oil in water emulsion which means that the SLS surfactant is soluble in water but does not mix. For the certain salinity, NaCl will cause an effective reduction of the oil–water interfacial tension. This condition occurs in the proportion of 20,000 ppm, 4.5%; 40,000 ppm, 1.5%; and 40,000 ppm, 4%, where the increase in salt (NaCl) causes a decrease of interfacial tension and reduces oil recovery. In the proportion of salinity of 20,000 ppm, 4.5%, IFT is lower, but microemulsions are not formed, meaning that in this system the surfactant is not soluble in the oil. This condition causes the reduction of performance of bagasse SLS surfactant and produces less recovery factor of oil [11].

2. Methodology

Bagasse is a by-product of the sugarcane liquid extraction process. Bagasse is one of the sources of biomass, whose utilization is currently mostly only as fuel in steam boilers, as raw materials of paper, or as a source of animal feed. The bagasse used is bagasse which has undergone the fifth milling process from the process of sugar (**Figure 5**) [11].

Bagasse mostly contains lignocellulose. The fiber length is between 1.7 and 2 mm with a diameter of about 20 μm . Bagasse contains 48–52% water, average sugar is 3.3%, and average fiber is 47.7%. Bagasse fiber is insoluble in water and consists mainly of cellulose, pentose, and lignin. Based on chemical analysis, the average bagasse has a chemical composition, namely, ash 3.28%, lignin 22.09%, cellulose 37.65%, juice 1.81%, pentosan 27.97%, and SiO_2 3.01% [6].

The process of bagasse becomes lignosulfonate starting from the separation of lignin from bagasse and then reaction with sodium bisulfite to sodium lignosulfonate. The processing of bagasse to produce lignosulfonate was initially performed by isolation of lignin and the isolated lignin transformed into sodium lignosulfonate by sulfonation process. The process is using reflux equipment with a condenser and bagasse at 80 mesh in size. The lignin isolation process uses reflux for 4 h, with sodium hydroxide reagent. The lignin produced was then processed into lignosulfonate using sodium bisulfite reagent and refluxed for 4 h [11].



Figure 5.
Sugar cane—Bagasse [11].

No.	Surfactant composition
1	5000 ppm—1.5%
2	10,000 ppm—1.5%
3	10,000 ppm—3.0%
4	20,000 ppm—1.5%
5	20,000 ppm—4.5%
6	40,000 ppm—1.5%
7	40,000 ppm—4.0%
8	80,000 ppm—1.5%
9	80,000 ppm—4.5%

Table 3.
Surfactant composition of concentration and salinity [11].

Before use as laboratory-scale injection fluid, the SLS surfactant synthesized from bagasse was tested for its characteristics to match the fluid and reservoir rocks to be injected. A variation of surfactant concentration and variation of formation water salinity were used. The SLS surfactant synthesized from bagasse is injected into a 20% porosity and 100–500 mD Berea cores containing synthetic brine and light crude oil. The injection process with bagasse SLS surfactant has been carried out with variations in various proportions of salinity and surfactant concentration, as stated in **Table 3**.

The core injection process uses a surfactant solution of 1.5, 3.0, 4.0, and 4.5% with salinity of 5000, 10,000, 20,000, 40,000, and 80,000 ppm. The core injection uses core flood apparatus with Berea core synthesis in the core holder, shown in **Figure 6**. The core injection process consists of two stages: water injection and sodium lignosulfonate surfactant injection [11].

Before surfactant injection, cores were saturated with formation water using a vacuum system, until 100% saturated. Formation water is injected into the core with a variation of the flow rate, 0.5, 0.75, 1.0, and 1.5 cc/min, so that the core is filled with formation water. From this stage, effective water permeability (K_w) data will be obtained. In the next stage, crude oil is injected into the core with a variation of flow rate, 0.25, 0.5, 1.0, 1.5, and 2.0 cc/min, so the formation water is pushed out, and the core is filled with crude oil. From this stage, effective oil permeability (K_o) and connate water saturation (S_{wc}) can be measured.



Figure 6.
Core flood apparatus [11].

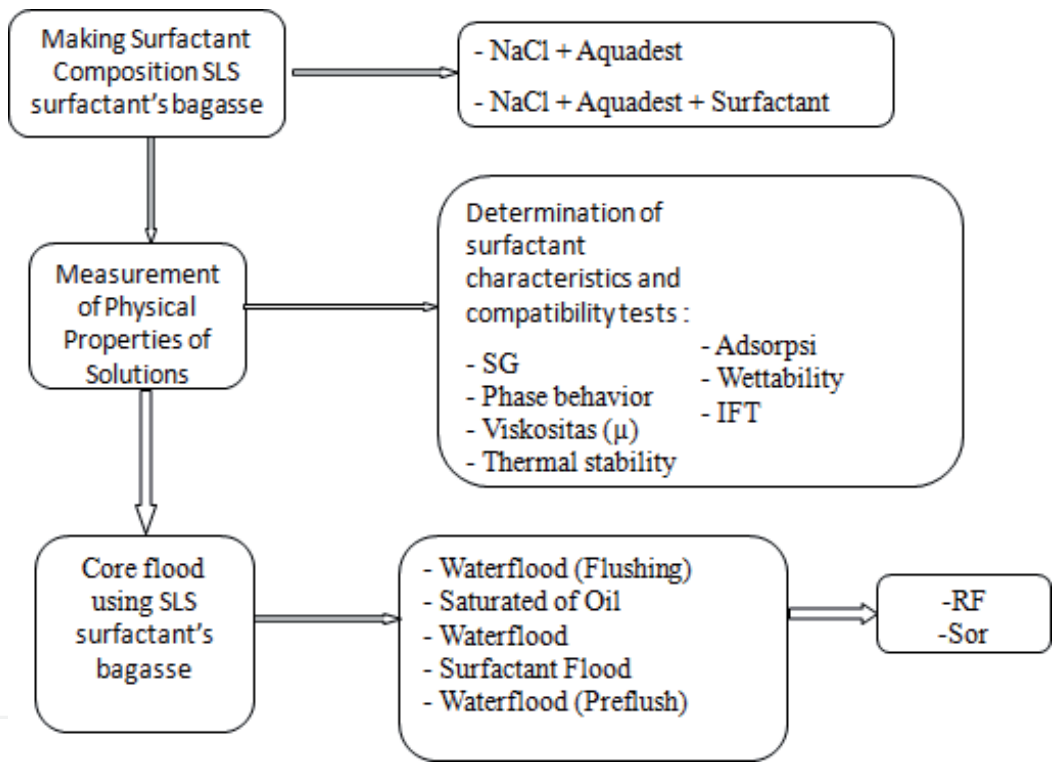


Figure 7.
Scheme of surfactant injection using SLS surfactant synthesized from bagasse [11].

After the core is saturated with crude oil, the first process begins with water injection and then proceeds with injection of surfactant, as shown below (Figure 7).

3. Result and discussion

The analysis of nuclear magnetic resonance (NMR) spectroscopy of SLS surfactant showed that the surfactant consisted of monomer structure having methoxyl and hydroxyl substituted benzene as well as the presence of sulfonate and aliphatic hydroxyl groups. Therefore, according to NMR spectrum analysis, it can be deduced that the monomer of synthesized surfactant has empirical formula of $(C_{11}H_{16}O_8S)_n$ with relative molecular weight of 308.06. The exact molecular weight

No.	Functional groups in structure Lignosulfonate	Wave number (cm ⁻¹)		
		SLS surfactant synthesized from bagasse	Lignosulfonate surfactant (Aldrich)	Lignosulfonate surfactant (Patricia)
1.	Alkene —C=C—	1635.34	1608.34	1630–1680
2.	Sulfonate S=O	1384.64	1365	1350
3.	Carboxylic C=O	1114.64	1187.94	1000–1300
4.	Ester S—OR	462.832	499.831	500–540

Table 4.
Results of FTIR of SLS surfactant synthesized from bagasse [2].

of the synthesized surfactant should be further determined using mass spectrometry measurement. The monomer of the synthesized SLS has a hydrophilic–lipophilic balance value of 11.6 which can be classified as O/W emulsion [12, 13], which means that the SLS surfactant is water soluble. Thus, the SLS surfactant derived from bagasse can be used as an injection fluid and formed middle-phase emulsion that is required in surfactant injection performance.

The results of the Fourier-transform infrared test on bagasse SLS surfactant have shown the existence of components contained in the SLS surfactant which consists of four main components forming lignosulfonic surfactants: alkene stretching groups, sulfonate stretch groups, carboxylic buckling groups, and ester buckling groups with wave numbers from the results of the FTIR [2]. As shown in **Table 4**, it turns out that the wave numbers in the SLS bagasse surfactant component have similarities with the same component wave number for standard lignosulfonic surfactants, namely, standard lignosulfonates from Aldrich and Patricia [14].

Before the characteristics of SLS surfactant synthesized from bagasse was carried out, the physical properties of the SLS surfactant were first measured. The physical properties of the SLS surfactant measured included the viscosity, density, and pH of the SLS surfactant at various proportions of the composition to be used in the injection. The measurement results can be seen in **Table 5**.

From the measurement of physical properties, it turns out SLS surfactant solution in various proportions has physical properties that are almost the same as the viscosity data range 1.5350–1.8375 cP, density 0.9970–1.0657 g/cc, and pH 5.91–6.67. The physical properties of this surfactant are in accordance with the characteristics of the surfactant as described previously.

The characteristic test of the bagasse SLS surfactant consists of:

- a. Aqueous stability test.
- b. Phase behavior test/emulsion test.
- c. Interfacial tension (IFT) test.
- d. Thermal stability test.
- e. Adsorption test.
- f. Wettability test.

From the characteristic test, some of the properties of SLS surfactant from bagasse can be known as seen in **Table 6**.

No.	Surfactant composition	Viscosity (cP)	Density (g/cc)	pH
1	10,000 ppm—1.5%	1.5350	1.0003	6.39
2	10,000 ppm—3.0%	1.5443	1.0113	6.67
3	20,000 ppm—1.5%	1.6877	1.0069	5.91
4	20,000 ppm—4.5%	1.5757	1.0280	6.56
5	40,000 ppm—1.5%	1.6697	1.0189	6.12
6	40,000 ppm—4.0%	1.7724	1.0367	6.46
7	80,000 ppm—1.5%	1.7630	1.0438	5.86
8	80,000 ppm—4.5%	1.8375	1.0657	4.38

Table 5.
The physical properties of the SLS surfactant synthesized from bagasse [11].

No.	Salinity (ppm)	Surfactant concentration (%)	Aqueous stability	Middle emulsion (Stability) %	IFT (mN/m)
1.	10,000	1.5	Clear	10.00	2.73
2.	10,000	3.0	Clear	7.50	1.68
3.	20,000	1.5	Clear	5.00	4.13
4.	20,000	4.5	Clear	0.00	1.27
5.	40,000	1.5	Clear	6.00	4.11
6.	40,000	4.0	Clear	0.00	2.72
7.	80,000	1.5	Clear	1.25	3.61
8.	80,000	4.5	Clear	1.00	1.09

Table 6.
Characteristic test results in various variations of SLS surfactant concentrations [11].

No.	Salinity (ppm)	Concentration surfactant (%)	IFT (mN/m)	Recovery factor from surfactant flooding (%)
1	5000	1.5	6.81	7.00
2	10,000	1.5	2.73	9.25
3	20,000	1.5	4.13	8.55
4	40,000	1.5	4.11	1.80
5	80,000	1.5	3.61	10.71

Table 7.
IFT and contact angle to recovery oil by SLS surfactant injection of bagasse (concentration of surfactant 1.5%).

In this research, the surfactant composition of 4.5% of SLS surfactant in 80,000 ppm NaCl gave the lowest IFT value of 1.091 mN/m. From the characteristic test, SLS surfactant synthesized from bagasse has aqueous stability, is clear, and does not cause turbidity. It has a hydrophilic–lipophilic balance value of 11.6 which is classified as oil in water emulsion and can form microemulsion with light oil. This SLS surfactant has middle-phase emulsion with volume fraction of 1–10% micro-emulsion and IFT of 1.091–6.81 mN/m.

Surfactant injection processes in this research using a variety of concentrations using a variety of concentrations and salinities have shown in **Table 7** and **Figure 8** the contour of relation between salinity and IFT to recovery factor.

From **Table 7**, optimal enhanced oil recovery reaches 10.71% at a concentration of surfactant of 1.5% and 80,000 ppm of surfactant solution. From the oil recovery data, the salinity proportion of 10,000 ppm of 1.5% oil recovery with surfactant injection (RF SF) reached 9.25% with stable middle-phase emulsion up to 10% and IFT value of 2.73 mN/m (**Table 6**). This is consistent with the theory that surfactants serve to lower IFT causing a breakdown of the water-to-water interfacial tension resulting in emulsions being formed up to 10% because they are soluble in oil and water. This microemulsions cause the SLS surfactant to produce the lowest value interfacial tension IFT 2.73 mN/m [15].

From **Figure 8**, in the relation contour between salinity and IFT, there are two areas: high recovery factor (red–orange color) and low recovery factor (purple–blue color). **Figure 8** and **Table 7** explain that at low salinity, 10,000 ppm (1.5% surfactant), the recovery factor is 9.25% and at high salinity, 80,000 ppm (1.5% surfactant), the recovery factor is 10.71%. These are the highest recovery factors at this surfactant injection using SLS surfactant synthesized from bagasse. The lowest recovery factor occurs at 40,000 ppm, only 1.80%. The results of injection of core using SLS surfactant from bagasse at concentration of 1.5% can be seen in **Table 7**.

Furthermore, the surfactant with the optimum composition has been tested toward its static and dynamic adsorptions. At the same surfactant concentration in different salt contents, it showed that the higher the salt content, the lower the adsorption value, which means that the surfactant is less absorbed into the rock when the surfactant was injected to the core. Since the less surfactant was

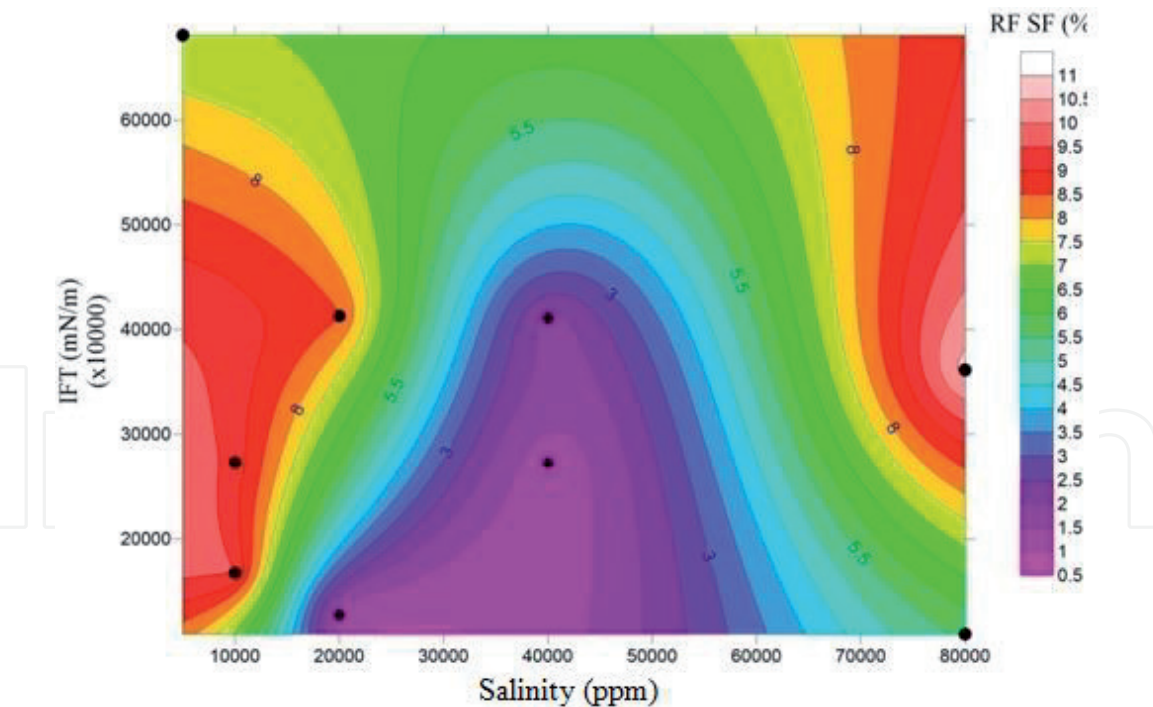


Figure 8.
Relation contour between salinity and IFT to recovery factor [11].

No.	Surfactant composition	Static adsorption (%)	Dynamic adsorption (%)
1.	20,000 ppm—4.5%	20.533	29.16
2.	80,000 ppm—1.5%	8.071	1.37

Table 8.
The result of static and dynamic adsorption test [15].

absorbed into the core, it means that more surfactants were available and capable to decrease the interfacial tension between oil and water; therefore the more oil can be produced by the surfactant injection. Wettability test results also showed that the higher salt content produces a larger contact angle which means the system becomes more water wet. These conditions have made the synthesized SLS surfactant derived from bagasse more dissolved in formation water to release grains of oil attached to the core. From the injection process, the composition of surfactant, with a salinity of 1.5% (80,000 ppm), showed the highest oil recovery value up to 10.71%, compared to the other composition. Therefore, the surfactant concentration and salinity affected many factors related to the performance of SLS surfactant of bagasse displacement on light oil.

At the low area recovery factor, the composition of surfactant, with a salinity of 1.5% (40,000 ppm), showed the lowest oil recovery value of 1.80%. At this condition, the value of IFT is 4.11 (mN/m). This value can explain that for this area, they are insoluble for oil and water, so that oil does not move easily. Besides IFT, there are other factors that affect the mechanism surfactant during the surfactant injection process. The other factor is adsorption. **Table 8** indicates the static and dynamic adsorptions of this SLS surfactant on the core surface at the high and the low salinities.

At this salinity of 20,000 ppm, the static adsorption is 20.533%, and the dynamic adsorption is 29.16%. This adsorption is higher than the adsorption at 80,000 ppm. From **Table 7**, recovery factor of the higher adsorption (salinity 40,000 ppm) is 1.80%, and for the lower adsorption (salinity 80,000 ppm), recovery factor is 10.71%. Due to the large amount of adsorption that occurs on the core, the amount of surfactant decreases, so that the surfactant mechanism also decreases. The lower mechanism of surfactant results in a decrease of recovery factor.

4. Conclusion

The SLS surfactant synthesized from bagasse with its natural characteristic can be used as an injection fluid in surfactant injection to increase oil recovery for sandstone with low to high salinity reservoir. The SLS surfactant synthesized from bagasse has displaced some of the residual oil after waterflood, depending on the IFT reduction, middle-phase emulsion (microemulsion), and adsorption value. The results of this study showed that the synthesized SLS surfactant of bagasse with its natural characteristics can be used as injection fluid in surfactant injection to increase oil recovery, especially for the light oil recovery. So the use of sodium lignosulfonate surfactant synthesized from bagasse is a challenge to be developed further as a surfactant flooding.

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