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Laboratory Test of Aqueous Stability and Phase Behavior of Sodium Lignosulphonate (SLS) Surfactant With Intermediate Oil at High Salinity

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Abstract

So far, the commonly used surfactant on petroleum industry is petroleum-based surfactant (Petroleum Sulphonate). Although this type of surfactant can perform optimally in reducing Interfacial Tension (IFT), it comes with a high cost, not renewable and not environmentally friendly. That is why plant-based surfactant emerge as alternative on oil recovery effort. One type of alternative plant-based surfactant is Sodium Lignosulphonate (SLS) Surfactant made from lignin. The character of surfactant used on EOR process is important to be identified to make sure it is suitable with the oil condition in reservoir. Surfactant characteristic tests conducted in this laboratory experiment are Aqueous Stability Test and Phase Behavior Test. Based on that, the objective of this research is to identify the characteristic of Bagasse Sodium Lignosulphonate (SLS) Surfactant, by conducting aqueous stability and phase behavior tests, in order to sort the ones that pass the tests to continue to the next stage. There are three variations of brine salinities used in this research namely 90.000 ppm, 100.000 ppm and 100.000 ppm with Bagasse Sodium Lignosulphonate (SLS) Surfactant concentrations of 1%; 1,5%; 2%; 2,5% and 3% with a 1:1 ratio of co-surfactant addition to the SLS surfactant. The results of this research revealed that every surfactant solution involved in Aqueous Stability test has homogenous solubility, which is marked by its transparent color. Based on Phase Behavior Test, surfactant with concentrations of 1,5% and 2,5% with 110.000 ppm salinity were managed to form middle phase with relatively large amount of total emulsion.

Keywords: Aqueous Stability, Bagasse Sodium Lignosulphonate (SLS) Surfactant, High Salinity, Intermediate Oil, Phase Behavior.

Introduction

Surfactant injection is one example of chemical injections used on Enhanced Oil Recovery (EOR) to improve oil production. Surfactant is able to lower water and oil interface tension mechanisms on rock pores and lower capillary force that traps the oil, and finally enables oil to flow out of the rock pores. Surfactant is also able to perform wettability alteration of the rock into water-wet, which cause the rock to be covered by water and release oil droplets from the pores and enable oil production (Rini Setiati, 2017).

Up until now, the commonly used surfactant on petroleum industry is petroleum-based surfactant (Petroleum Sulphonate). Although this type of surfactant can perform optimally in reducing Interfacial Tension (IFT), it comes with a high cost, not renewable and not environmentally friendly. That is why plant-based surfactant emerge as alternative on oil recovery effort (Fitriania, SuryoPurwonoa, & Tawfiequrrahmana, 2017). One type of alternative plant-based surfactant is Sodium Lignosulphonate (SLS) surfactant made from lignin (Setiati, Siregar, Marhaendrajana, & Wahyuningrum, 2018).

The character of surfactant used on EOR process is important to be identified to make sure it is suitable with the oil condition in reservoir. The reason is that surfactant is a highly sensitive chemical compound to temperature, salinity, and other components. Based on that, laboratory test on surfactant characteristic is important and must be conducted before implementing it on a field scale to perform its mechanism. The most common tests of surfactant characteristic on laboratories are Aqueous Stability and Phase Behavior Test.

Aqueous Stability test is conducted to identify the suitability of surfactant and formation water. Test is conducted by mixing surfactant formula with brine, which then observed to witness any changes on the solution and expect it to produce homogenous or clear solution. The reason behind compatibility test is to identify whether a sediment that can plug the rock can be formed (Lemigas, 2008).

Phase Behavior of surfactant/oil/water mixture is the most important factor that the success level of chemical flooding process. The expected ultra-low

IFT on surfactant system is usually measured by testing the microemulsion system phase behavior where area with high solubility is located. Phase behavior is depending on the type and concentration of surfactant, co-surfactant, hydrocarbon and brine (Sandersen, 2012).

Phase behavior is a microemulsion phase nature test formed during oil and water meet with each other. The main objective of this test is to optimize surfactant formulation aimed to be injected on EOR process. Microemulsion can be defined as phase nature that has transparent appearance, thermodynamically stable, a mixture of oil, water/brine, and surfactant (Savero, 2020). Generally the stability formed between hydrocarbon and water is stabilized by surfactant (Chauhan, 2014).

There are three types of microemulsion classification on chemical Enhanced Oil Recovery (cEOR), especially surfactant, namely Winsor Type I (lower phase), Winsor Type II (upper phase) and Winsor Type III (middle phase) (J. Sheng, 2011). As oil-on-water (O/W) microemulsion reach stability with excess oil, it is categorized as Winsor Type I; when water-on-oil (O/W) microemulsion reaches stability with excess water it is categorized as Winsor Type II; if microemulsion reaches oil and water stability it is categorized as Winsor Type III. On optimum salinity condition, stable middle phase microemulsion will be formed and it can lower the remaining oil saturation (Sor). Middle phase microemulsion is also possess ultra-low interfacial tension, wide interface area, thermal stability, and ability to dissolve oil and water (J. J. Sheng, 2015). Generally, Winsor Type III microemulsion is the preferable one on surfactant injection because it can produce the lowest interfacial tension value and improve oil recovery. On the other hand, Winsor Type I and II stabilities are located with either excess of oil or water (J. J. Sheng, 2015).

The conducted laboratory test was formulated based on challenges in research regarding surfactant process flooding by utilizing Sodium Lignosulphonate (SLS) surfactant made from bagasse, that is yet to discover a suitable surfactant characteristic especially on aqueous stability and phase behavior test, with optimum and consistent co-surfactant addition on high salinity intermediate oil. Based on that, the objective of this research is to identify the characteristic of bagasse Sodium Lignosulphonate (SLS) surfactant, by conducting aqueous stability and phase behavior tests, in order to sort the ones that pass the tests to continue to the next stage.

Data and Method

This research consists of a number of testing sequences conducted experimentally by using a mixture of SLS surfactant, brine, and co-surfactant tested on a series of experiments. In this research, similar procedure as mentioned on previous research is conducted to be able to produce a comparison of surfactants with different brine concentration and salinity in order to obtain suitable surfactant solution that would provide valuable insight towards SLS surfactant utilization on EOR, especially on surfactant flooding method.

There are three variations of brine salinity utilized in this research, namely 90.000 ppm, 100.000 ppm and 110.000 ppm Sodium Lignosulphonate (SLS) surfactant made from bagasse with concentrations of 1%; 1,5%; 2%; 2,5%; and 3% which next will be mixed with co-surfactant. The utilized co-surfactant is Ethanol 96% with concentration ratio of 1:1 to SLS surfactant. The utilized oil in this research is intermediate oil obtained from Z field. The temperature used in this test is at 60°C. The tests conducted are initial surfactant screening tests which are Aqueous Stability and Phase Behavior tests. The tools required to conduct this research and to conduct observation are mostly Pipette Tube dan Oven. This research was performed in Universitas Trisakti's Enhanced Oil Recovery (EOR) Laboratory.

Aqueous Stability test is a test to measure compatibility level of surfactant solution and formation water of a reservoir. This test was conducted for 21 days in 60°C heating and ageing inside an oven. Aqueous Stability test was conducted by dissolving surfactants and brines at 90.000 ppm, 100.000 ppm, and 110.000 ppm salinities, each with surfactant and co-surfactant concentrations of 1%; 1,5%; 2%; 2,5%; and 3%. After that, the solutions were inserted inside pipette tubes and observed on the mentioned period of time. The surfactant is categorized as good solubilization surfactant when it is completely dissolved, does not show murkiness, sediments or lumps that would cause clogging during reservoir injection process.

Phase Behavior test is conducted to identify the type of phase and emulsion total volume formed between surfactant solution and oil. Phase behavior test procedures are as follows:

1. Prepare 30 pieces of 5ml Pipette Tube.
2. Prepare SLS surfactant solution previously mixed with brines and co-surfactant, with total samples of 15 with 90.000 ppm, 100.000 ppm, and 110.000 ppm salinities, each of them with surfactant and co-surfactant concentrations of 1%; 1,5%; 2%; 2,5%; and 3%.
3. Prepare ±100 ml of intermediate oil sample (obtained from field Z).
4. Pour the surfactant and oil into the pipette tubes, each of them with a volume of 2 ml.
5. Mix well until they are blended.
6. Set aside and observe the solutions starting from hour 0, up to hour 504 or until both phases reach stable condition and form microemulsion, so that the type of surfactant phase can be determined.

Result and Discussion

All tools used have been calibrated before the research was conducted. Data obtained from Aqueous Stability and Phase Behavior tests, were collected through 504 hours of observation in Universitas Trisakti's Enhanced Oil Recovery (EOR) laboratory.

Aqueous Stability Test (Compatibility Test)

Aqueous Stability test is an experiment conducted visually, where in this research, the clearness of the solution is observed with naked eyes immediately after dissolving surfactant into brine, added with co-surfactant and set aside on a reservoir temperature or 60°C oven.

Surfactant concentration variations utilized in this research are at 1%; 1,5%; 2%; 2,5%; and 3%. The utilized co-surfactant concentration is also at 1%; 1,5%; 2%; 2,5%; and 3% to fulfill 1:1 ratio requirement to surfactant concentration. Brine salinities used in this research are at 90.000 ppm, 100.000 ppm, and 110.000 ppm. Data obtained from Aqueous Stability test can be seen in **Table 1**.

Based on Aqueous Stability test results shown in **Table 1**, we can see that up until 504 hours of observation or 21 days, the ageing surfactant and co-surfactant solutions in 60°C oven, produced clear solution on brines with salinities of 90.000 ppm, 100.000 ppm, and 110.000 ppm, even with increased surfactant concentration from 1% to 3%, the solutions are still transparent. This indicates that the produced surfactant and co-surfactant solutions are compatible with the brine and they can be said as homogenous solutions. If the solution is not dissolved perfectly, it will show a little bit of murkiness or often mentioned as milky solution, because surfactant and brine cannot perfectly dissolved and form a single homogenous solution and create precipitation or sediments on the solution. This finding is supported by a finding mentioned by Eni & Tobing (2013) that the observation conducted on aqueous stability or compatibility tests is to see if there is sediments formed that would possibly cause plugging on rocks.

Phase Behavior Test

Phase Behavior test conducted in this research is aimed to observe emulsion formation, microemulsion volume, and to determine phase type formed between surfactant and co-surfactant with concentrations of 1%; 1,5%; 2%; 2,5%; and 3% (co-surfactant and surfactant SLS ratio is at 1:1), and Intermediate Oil (obtained from Field Z). Brine salinities are at 90.000 ppm, 100.000 ppm, and 110.000 ppm. Tests were conducted for 504 hours or 21 days by observing the formed emulsion and the volume of solution phase formed in Pipette Tubes. The temperature of the oven is at 60°C. Phase behavior test result 90.000 ppm salinity solution can be seen on **Table 2**.

Based on Phase Behavior test result data on 90.000 ppm salinity in **Table 2**, we can see that the middle phase was formed on 2,5% and 3% surfactant and co-surfactant salinities with total emulsion volume respectively at 3,75% and 3,13%. Microemulsion that reaches stability with oil and surfactant is categorized as Winsor Type III. Meanwhile lower phase was identified on surfactant and co-surfactant solutions with concentrations of 1%; 1,5%, and 2% with emulsion total volumes respectively at 2,5%; 2,5%; and 1,25%. In this case, the lower phase or Winsor Type I was formed when surfactant is "dissolved" on oil which increase oil volume (excess oil balance) (J. J. Sheng, 2015). Based on that, surfactant and co-surfactant solutions with concentrations of 1%; 1,5%, and 2% on 90.000 ppm salinities are unsuitable to be used on the next stage of tests because it formed lower phase or Winsor Type I.

Next, similar tests were conducted on 100.000 ppm salinity solution. The surfactant and co-surfactant concentration, oil and solution treatments are similar as the ones performed on 90.000 ppm salinity. Phase behavior test result data on 100.000 ppm salinity can be seen in **Table 3**.

Based on the results of the 100.000 ppm Salinity Research Phase Behavior Test in **Table 3**, it can be seen that there is no Middle Phase but only a Lower Phase in all surfactant + co-surfactant solutions with concentrations of 1%; 1,5%; 2%; 2,5% and 3%. Besides that, the emulsion volumes are relatively low, respectively at 2,50%; 1,25%; 4,38%; 3,75%; and 3,13%. In this case, Lower Phase or Winsor Type I occurred when surfactant "dissolved" on oil and increase oil volume (excess oil balance) (J. J. Sheng, 2015). Based on that, every 100.000 ppm salinity surfactant is unsuitable to be used on the next stage because every one of them formed lower phase or Winsor Type I. This finding shows that surfactant solution is unable to form middle phase because the oil phase is higher than surfactant phase (excess oil balance).

Similar tests were also conducted on 110.000 ppm salinity solutions. The surfactant and co-surfactant concentration, oil and solution treatments are similar as the ones performed on 90.000 and 100.000 ppm salinity, the difference is that in this test, 110.000 ppm salinity solutions were used. Phase behavior test result data on 110.000 ppm salinity can be seen in **Table 4**.

Based on the data from the Phase Behavior Test for Salinity Research of 110.000 ppm in **Table 4**, the type of Middle Phase occurs in a surfactant + co-surfactant solution with a concentration of 1%; 1,5%; and 2,5%, with a total emulsion volume of respectively at 6,25%; 6,88%; and 11,25%.

Microemulsions that reach equilibrium with oil and surfactants are called Winsor Type III. While the Lower Phase type occurs in surfactant and co-surfactant solution with a concentration of 2% and 3% having a total emulsion volume of respectively at 6,25% and 2,50%. In this case, lower phase or Winsor Type I occurred when surfactant “dissolved” on oil and increase oil volume (excess oil balance) (J. J. Sheng, 2015). Therefore, the solution with a concentration of 2% and 3% at a salinity of 110.000 ppm is not feasible to continue in the next test. From a concentration of 1%; 1,5%; and 2,5%, the concentrations used in the next test were only 1,5% and 2,5%. This is because the 1% concentration has the lowest total emulsion volume of the three concentrations. Cost and Time are also act as important consideration to choose the two surfactant and co-surfactant concentrations.

Conclusions

Based on the data obtained from the conducted tests, we can conclude that:

1. The Aqueous Stability test results show that all surfactant + co-surfactant solutions possess transparent solubility up to 504 hours of observation. These solutions have the ability to form homogenous phases.
2. Based on Phase Behavior test, surfactant + co-surfactant solutions with 1,5% and 2,5% concentration at 110.000 ppm salinity are the only solutions that can form middle phase with relatively high total emulsion, respectively at 6.88% and 11,25%.

This is an early-stage of surfactant screening test on the characteristics of SLS surfactants with intermediate oil at high salinity. For the next research, advanced tests such as thermal stability test, surfactant adsorption test, and core flooding test are highly important to obtain more accurate SLS surfactant characteristics.

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Table 1: Aqueous Stability Test Results

Brine Salinity (ppm)	Surfactant + Co-Surfactant Concentration (%)	Observation Result of Aqueous Stability Test (Transparent/Milky/Precipitation), Hour						
		0	**2**	**24**	**48**	**168**	**336**	**504**
90.000	**1**	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	1,5	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	2	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	2,5	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	3	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
100.000	**1**	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	1,5	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	2	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	2,5	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	3	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
110.000	**1**	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	1,5	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	2	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	2,5	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent
	3	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent	Transparent

Table 2: Phase Behavior Test Results at Salinity of 90.000 ppm

Type of Oil	Surfactant Composition	Phase	Volume At Observation Time (Hour)									Total Emulsion (%)	Phase Type
			0	**0,5**	**1**	**2**	**24**	**48**	**168**	**336**	**504**		
"Field Z" Oil	**1% Surfactant + Co-surfactant**	**Oil**	0	2	2	2	2	2	2	2	2	2,50%	Lower Phase
		Emulsion	4	0,2	0,15	0,125	0,1	0,1	0,1	0,1	0,1		
		Surfactant	0	1,8	1,85	1,875	1,9	1,9	1,9	1,9	1,9		
	1,5% Surfactant + Co-surfactant	**Oil**	0	2	2	2	2	2	2	2	2	2,50%	Lower Phase
		Emulsion	4	0,15	0,15	0,125	0,125	0,12	0,12	0,1	0,1		
		Surfactant	0	1,85	1,85	1,875	1,875	1,88	1,88	1,9	1,9		
	2% Surfactant + Co-surfactant	**Oil**	0	2	2	2	2	2	2	2	2	1,25%	Lower Phase
		Emulsion	4	0,1	0,075	0,075	0,05	0,05	0,05	0,05	0,05		
		Surfactant	0	1,9	1,925	1,925	1,95	1,95	1,95	1,95	1,95		
	2,5% Surfactant + Co-surfactant	**Oil**	0	1,8	1,8	1,85	1,85	1,875	1,9	1,95	1,95	3,75%	Middle Phase
		Emulsion	4	0,3	0,3	0,25	0,25	0,225	0,2	0,15	0,15		
		Surfactant	0	1,9	1,9	1,9	1,9	1,9	1,9	1,9	1,9		
	3% Surfactant + Co-surfactant	**Oil**	0	1,7	1,7	1,75	1,8	1,8	1,9	1,975	1,975	3,13%	Middle Phase
		Emulsion	4	0,4	0,4	0,35	0,3	0,3	0,2	0,125	0,125		
		Surfactant	0	1,9	1,9	1,9	1,9	1,9	1,9	1,9	1,9		

Table 3: Phase Behavior Test Results at Salinity of 100.000 ppm

Type of Oil	Surfactant Composition	Phase	Volume At Observation Time (Hour)									Total Emulsion (%)	Phase Type
			0	0,5	1	2	24	48	168	336	504		
"Field Z" Oil	1 % Surfactant + Co-surfactant	Oil	0	2	2	2	2	2	2	2	2	2,50%	Lower Phase
		Emulsion	4	0,3	0,3	0,25	0,25	0,1	0,1	0,1	0,1		
		Surfactant	0	1,7	1,7	1,75	1,75	1,9	1,9	1,9	1,9		
	1,5 % Surfactant + Co-surfactant	Oil	0	2	2	2	2	2	2	2	2	1,25%	Lower Phase
		Emulsion	4	0,2	0,13	0,13	0,13	0,08	0,08	0,05	0,05		
		Surfactant	0	1,9	1,88	1,88	1,88	1,93	1,93	1,95	1,95		
	2 % Surfactant + Co-surfactant	Oil	0	2	2	2	2	2	2	2	2	4,38%	Lower Phase
		Emulsion	4	0,2	0,2	0,2	0,2	0,18	0,18	0,18	0,18		
		Surfactant	0	1,8	1,8	1,8	1,8	1,83	1,83	1,83	1,83		
	2,5 % Surfactant + Co-surfactant	Oil	0	2	2	2	2	2	2	2	2	3,75%	Lower Phase
		Emulsion	4	0,2	0,2	0,2	0,2	0,18	0,18	0,15	0,15		
		Surfactant	0	1,8	1,8	1,8	1,8	1,83	1,83	1,85	1,85		
	3 % Surfactant + Co-surfactant	Oil	0	2	2	2	2	2	2	2	2	3,13%	Lower Phase
		Emulsion	4	0,2	0,15	0,15	0,15	0,15	0,13	0,13	0,13		
		Surfactant	0	1,9	1,85	1,85	1,85	1,85	1,88	1,88	1,88		

Table 4: Phase Behavior Test Results at Salinity of 110.000 ppm

Type of Oil	Surfactant Composition	Phase	Volume At Observation Time (Hour)									Total Emulsion (%)	Phase Type
			0	0,5	1	2	24	48	168	336	504		
"Field Z" Oil	1 % Surfactant + Co-surfactant	Oil	0	1,7	1,7	1,8	1,9	1,88	1,9	1,9	1,9	6,25%	Middle Phase
		Emulsion	4	0,5	0,5	0,4	0,4	0,33	0,3	0,25	0,25		
		Surfactant	0	1,8	1,8	1,8	1,8	1,8	1,8	1,85	1,85		
	1,5 % Surfactant + Co-surfactant	Oil	0	1,8	1,8	1,8	1,8	1,8	1,8	1,8	1,8	6,88%	Middle Phase
		Emulsion	4	0,4	0,3	0,3	0,3	0,3	0,3	0,28	0,28		
		Surfactant	0	1,9	1,9	1,9	1,9	1,9	1,9	1,93	1,93		
	2 % Surfactant + Co-surfactant	Oil	0	2	2	2	2	2	2	2	2	6,25%	Lower Phase
		Emulsion	4	0,3	0,3	0,3	0,3	0,25	0,3	0,25	0,25		
		Surfactant	0	1,8	1,8	1,8	1,8	1,75	1,8	1,75	1,75		
	2,5 % Surfactant + Co-surfactant	Oil	0	1,6	1,6	1,7	1,7	1,8	1,8	1,8	1,8	11,25%	Middle Phase
		Emulsion	4	0,7	0,7	0,6	0,6	0,45	0,5	0,45	0,45		
		Surfactant	0	1,8	1,8	1,8	1,8	1,75	1,8	1,75	1,75		
	3 % Surfactant + Co-surfactant	Oil	0	2	2	2	2	2	2	2	2	2,50%	Lower Phase
		Emulsion	4	0,1	0,1	0,1	0,1	0,1	0,1	0,1	0,1		
		Surfactant	0	1,9	1,9	1,9	1,9	1,9	1,9	1,9	1,9		