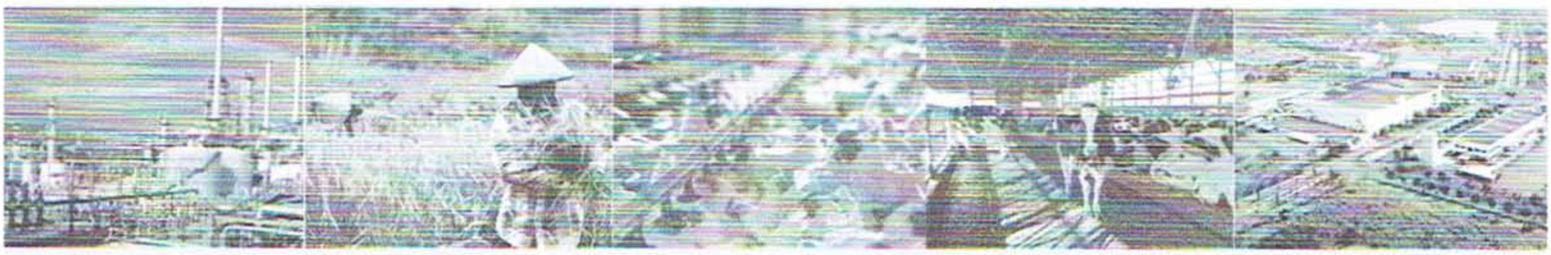
PROCEEDINGS

2015 3rd International Conference on Adaptive and Intelligent Agroindustry (ICAIA)

ICAIA 2015



August 3rd - 4th, 2015
IPB International Convention Center
Bogor, Indonesia

ISBN : 978-1-4673-7405-7
IEEE Catalog Number : CFP15C67-ART

IEEE INDONESIA SECTION

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Frontier Approaches in Process and Bioprocess Engineering

 ISBN : 978-1-4673-7405-7

The Effects of Palm Oil MES Surfactant and Inorganic Salt Concentration on Interfacial Tension Values

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Abstract— **The application of methyl ester sulfonic (MES) surfactant for enhanced oil recovery (EOR) was based on its ability to reduce interfacial tension in crude oil-water system that reached 10^{-3} dyne/cm. Performance of methyl ester sulfonic (MES) surfactant from palm oil can be identified with the spinning drop IFT method. The objectives of this research were to determine MES surfactant performance with the addition of inorganic salt in different conditions. The dispersing media used in this research were demineralized water and formation water. The spinning drop IFT method was conducted using spinning drop tensiometer TX500D tested in 40, 50 and 60°C. Results showed that reduction of IFT values was affected by MES surfactant and inorganic salt concentration in the demineralized water and formation water as dispersion medium. Methyl Ester Sulfonic surfactant could react better at salinity ranges of 5000-25000 ppm.**

I. Introduction

The application of surfactant for enhance oil recovery (EOR) needs particular requirements including ultralow interfacial tension ($\leq 10^{-3}$ dyne/cm), compatibility with formation water and stability in reservoir temperature, pH interval 6-8, having III phase (middle phase) or phase II (-), and oil recovery increment between 15-20% of original oil in place (OOIIP) [1]. A surfactant with ultralow interfacial tension ($<10^{-2}$ dyne/cm) was predicted to be able to increase oil recovery by about 10-20% [2].

A surfactant that needs to be developed for enhanced oil recovery (EOR) requirement is methyl ester sulfonic (MES) surfactant of palm oil. It was known from an analysis that MES had IFT values of 7.7 x 10^{-3} dyne/cm and was stable in reservoir temperature of up to 80°C. This finding approved that MES was suitable for use in sandstone fields in Indonesia and was resistant to reservoir temperature.

Methyl ester sulfonic is an anionic surfactant that developed as petroleum sulfonic substitution. MES has good dispersion characteristic, good detergency, especially in hard water, excellent biodegradability and it does not contain phosphate, ester fat-acid C_{14}, C_{16} and C_{18} gave the best detergention. Compared to petroleum sulfonic, MES surfactant shows some advantages. In lower concentration, MES has equal detergency level, better ability to maintain enzime activities, better tolerance to calcium, and lower salt content [3].

Surfactant injection in a water-oil system makes surfactant dispersed in oil and water. This is then followed by the formation of emulsion in water. As the result of capillary effect and high interfacial tension between oil and water, oil droplets trapped in pore throats cannot be produced by using water injection only. The addition of surfactant is expected to lower the interfacial tension between water and oil allowing a decrease in capillary pressure of oil and rocks. High capillary pressure results in low recovery factor. Low capillary pressure is needed to recover most remaining oil which is trapped after waterflooding. Lowered interfacial tension makes oil concentrated in rock surface [4]. In the end, surfactant binds the oil so that the oil can be produced. The effect of IFT values on oil recovery is modelled by capillary a desaturation curve, where residual oil saturation is correlated with capillary number function. Capillary number (Nc) is

Manuscript received April 27, 2015. Thanks to SBRC LPPM IPB for all facilities, so this research could be done.

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ISBN : 978-1-4673-7405-7

defined as viscosity ratio and capillary force. Generally, capillary number is calculated by using the following equation.

$$Nc = \frac{v\mu}{\sigma \cos\theta}$$

where:
V = effective flow rate (cm/s)
μ = viscosity of pusher solution (cp)
σ = interfacial tension (dyne/cm)
θ = wetting angle

Surfactant and salt concentration in solution is the has great effect on the obtainment of minimum IFT values. IFT values get lower as surfactant concentration increases. IFT reaches its minimum value as the critical surfactant concentration. Higher surfactant concentration than its critical point will lead to increased IFT values [5]. In this study, two types of water with extereme difference in salinity levels (demineralized water and formation water) were used. Formation water was used as electrolyte solution. Electrolytes in surfactant system decrease surfactant-water interaction. Lipophilic groups of ionic surfactant will half-bind or full-bind with electrolytes, so each molecule will bind with the right molecule. Negative charge of active lipophilic group will have a positive interaction with positive charge of salt molecules such as Na^+ molecule in NaCl solution when anionic surfactant is used [6,7]. It is different from demineralized water which has no salt or electrolyte in it, so surfactant-water interaction wil be bigger than surfactant-oil interaction.

II. METHODS

The study was done in two stages. In the first stage, formulation of basic material was done and in the second stage interfacial tensions were tested.

A. Basic Material Formulation

1. Formulation of demineralized water with different surfactant concentration

MES was formulated with demineralized water. Total weight of formulation result was 25 gram for one formulation. Two samples were made for each formulation. Total weight of formulation result for trial procedure was 25 gram. MES surfactant was added in the concentrations of 0.1, 0.3, 0.5, 0.7, 1.0, 1.5, and 2.0%. The example of surfactant weight calculation was shown below:

0.3% x 25 gram = 0.075 gram

Formation water was then added in to the formulation until the mixture weighed 25 gram. The formulation was stirred for an hour by using a magnetic stirrer in room temperature (27 °C). Then, density test and IFT value test were done.

2. Formulation of MES surfactant and demineralized water with different inorganic salt concentration.

In this stage, formulation of demineralized water and 0.3% MES surfactant was done. The value of 0.3% MES surfactant was used based on the optimum surfactant concentration found in the previous study at SBRC. Inorganic salt was added within the concentrations of 0, 1, 3, 4, 5, 6, 7, 8, 9, and 10%.. Total weight of formulation result was 25 gram for one formulation and two samples were made for each salt concentration. Formula 1 was the formulation of demineralized water with inorganic salt of 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, and 10%. The example of salt weight calculation was shown below:

1% x 25 gram = 0.25 gram

Demineralized water was then added in to the formulation until the mixture weighed 25 gram. The formulation was stirred for 15 minutes until it was well blended by using a magnetic stirrer in room temperature (27°C). MES surfactant was weighed (0.075 g) and put in a different tube. Formula 1 was then added into the tube containing MES surfactant until the wieght of the mixture reached 25 grams.

In this study, a case study in an oil field which had salt concentration that fitted with performance of MES surfactant according to IFT test result of demineralized water. Same treatments were done to formation water.

B. Interfacial Tension (IFT) Value Test

Interfacial tension was measured by spinning drop method. The following steps were the procedures of spinning drop interfacial method. The device and the LED button were turned on. The spinning drop device was heated before the temperature was set at 40 °C (fitted to test condition). Once the temperature was stable, surfactant which was prepared in certain concentrations was added into a glass tube. Then, crude oil was added. Air bubble was not allowed in the glass tube. Then, the glass tube was put in the spinning drop device with glass tube surface facing outside. Spinning speed was set to be stable at 6000 rpm. Drop radius was read when the device temperature reached 40°C. The reading was repeated until the value of drop radius was constant. The test was repeated for the temperatures of 50 and 60°C.

When the fluid drop had a cylindrical form, cylinder radius (r), difference of drop density (Δρ) and drop spinning speed (ω) were measured. Spinning Drop

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Tensiometer could measure interfacial tension (IFT) up to 10^{-6} mN/m. Finally, interfacial tension (γ) was calculated by using the following equation [8].

$$\gamma = \frac{1}{4} r^3 \Delta \rho r \omega^2$$

where:

r : radius

γ : interfacial tension

$\Delta\rho$: difference of drop density

ω : spinning speed

III. Results and Discussion

Optimum surfactant concentration for demineralized water formula as dispersion medium was 0.7% of MES surfactant, but it did not meet the ultralow interfacial tension (Figure 1). IFT values for demineralized water as dispersion medium at 40, 50, and 60°C were 6.75×10^0, 1.12×10^1, and 8.31×10^0 dyne/cm, respectively.

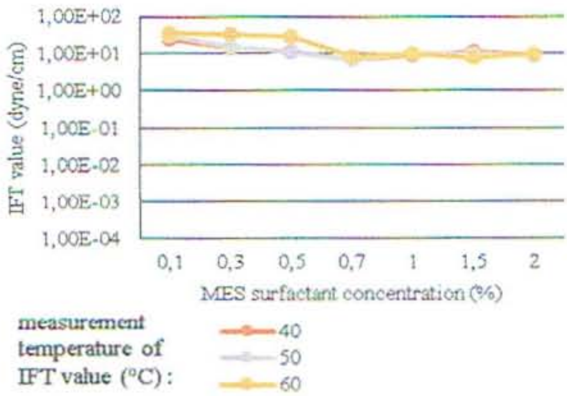

Fig. 1. Effect of interaction of MES surfactant concentration and measurement temperature on IFT values of surfactant solution with demineralized water as dispersion medium.

Next formulation step was optimal salinity by adding NaCl to demineralized water then 0,3% MES surfactant was added. According to previous research by SBRC IPB, surfactant that would be added was 0,3%. Salt addition caused IFT value decreased and then would increased again along with salt concentration increasement at various temperatures, 40°C, 50°C, and 60°C.

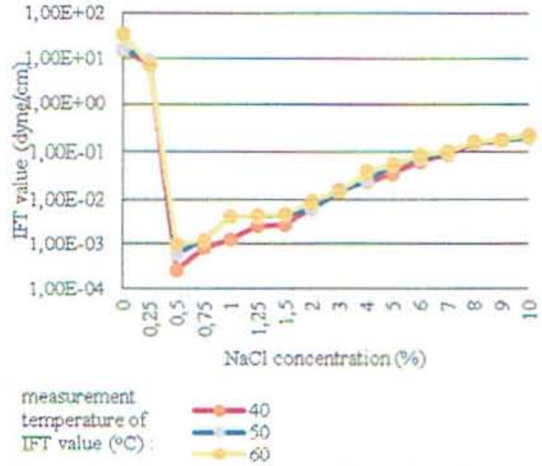

Fig. 2. Effects of Interaction of salt (NaCl) concentration and measurement temperature on IFT value of 0.3% MES surfactant solution with demineralized water as dispersion medium.

It was shown in Figure 2 that demineralized water formula with NaCl and 0,3% SMES addition had minimum IFT value when salt concentration was 0.5% or 5000 ppm. The IFT value required by oil industry was $\leq 10^{-3}$ dyne/cm. Results (Figure 2) showed that optimum salt concentration for MES surfactant was 5000 to 25000 ppm or 0.5 to 2.5%. Therefore, for a field case study, fluid X which had salt concentration of 5795 ppm which was within the optimum range, was used. As for reservoir which has salt concentration higher than 5000-25000 ppm, the surfactant could not be used.

The IFT values for formation water as dispersion medium with 0.3% palm oil surfactant addition were found to be within the ultralow interfacial tension. The IFT values at 40, 50, and 60°C were 4.40×10^{-3}, 1.68×10^{-3}, and 1.10×10^{-3} dyne/cm, respectively. These IFT values were shown in Figure 3.

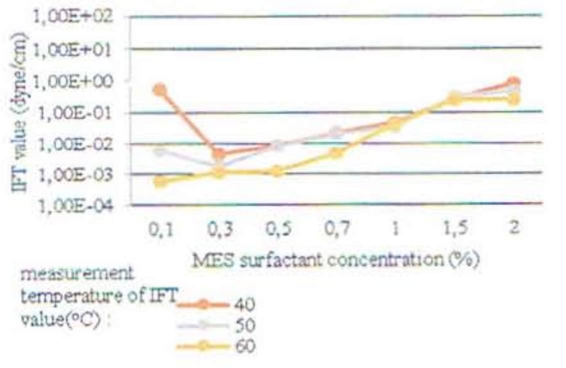

Fig. 3. Effects of interaction of MES surfactant concentration and measurement temperature on IFT values of surfactant solution with formation water as dispersion medium.

In surfactant addition formula, differences in IFT values of surfactant solution dispersed in demineralized

ISBN : 978-1-4673-7405-7

water medium and surfactant solution dispersed in formation water medium were caused by differences in water salinity levels. Formation water salinity reached 5795 ppm (Table 1) while that of demineralized was 0 ppm. When salinity condition was optimum, minimum value of IFT could occur and the emulsion formed could reach phase III. IFT decreased as surfactant concentration increased. However, this IFT value would reach its minimum value at a certain surfactant concentration known as the critical concentration. Increasing surfactant concentration above this critical point would not further lower but rather increase IFT value [5]. This interaction is seen in Figure 1 and Figure 3.

Results of IFT test gave a different description of oil respond in demineralized water and formation water media. When the test was done, in demineralized water medium, the oil used had a round shape. Meanwhile, the oil taken from a sandstone field used in formation water medium was longitudinally twisted indicating that the oil was not well blended although surfactant was added. This might be cause by the fact that there was no salt in it.

Inter-headgroup repulsion occuring in a surfactant molecule is one of the factors most affecting self assembly of surfactant molecules in micelle formation. In anionic surfactant solution, repulsion occurs because surfactant head groups have the same charge. As this repulsion will obstruct surfactant molecules to aggregate, it has to be decreased [9]. Repulsion of surfactant solution occurs because ionic power of the solution is weak. Salt addition, like in the form of formation water used as a dispersion medium, can be done to increase the ionic power of solution. Salt ions give a screen out effect which decreases inter-headgroup repulsion. This leads to a decrease in micelle formation free energy so that surfactant molecules are easier to unite and the micelles formed tend to have bigger size [10].

The inclination of micelle growth is affected by the type and concentration of salt added [11]. As the concentration of salt added gets higher, the screen out effect resulted is also higher leading to an expansive micelle growth forming a flexible cylinder shape (worm like) as seen in the response of oil to formation water dispersion medium [12]. In MES solution, with the existence of NaCl, micelle growth is attributed to the screen out effect resulted from the release of Na^+ ion from NaCl. This ion neutralizes charges of sulfonate groups in the headgroup of MES molecules so that inter-headgroup repulsion caused by similar charges can be minimized. Expanded micelle formation with an increased salt concentration is shown in next formula in Figure 2 and 4.

The mechanism of a decrease in interfacial tension of oil and water is as follows. Organic surfactant has a hydrocarbon basic group (R) which is bonded to SO_3, an inorganic compound (sulfonate group), to form R-SO_3H. in water, this type of surfactant is ionized into SO_3^- and H^+. When an RSO_3^- molecule has a contact with a nonpolar compound (oil), the R-group will form an adhesion force (surfactant-oil) while to surfactant molecule, a cohesion force among RSO_3^- molecules will work. The effect of this adhesion force reduces the resultant value of the cohesion force of the oil which will, in turn, reduce the interfacial tension of oil and water [13]. If IFT could be decreased to 10^{-3} dyne/cm, oil fraction inside rock pores could be better mobilized [14].

In this study, in formation water with 0,3% surfactant and NaCl addition, the IFT values were found to increase with an increase in the concentration of NaCl added.

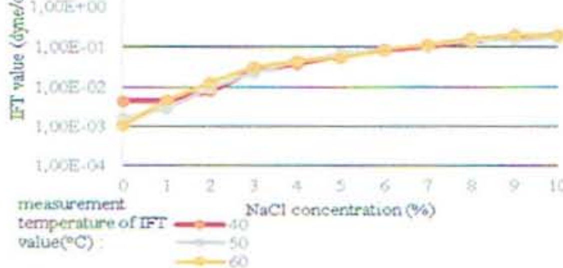

Fig. 4. Effects of NaCl concentration and measurement temperatures on IFT values of 0.3% MES surfactant in formation water dispersion medium.

It was found in this study that the addition of NaCl to demineralized water and formation water resulted in increased IFT values. In water formation dispersion medium samples, the lowest IFT value was found in formulation without NaCl addition. This was supported by the finding that formation water reached its optimum salinity of 5795 ppm with MES surfactant inclusion of 0.3%. Higher concentration of NaCl added resulted in higher IFT values.

Results of this study also showed that salt addition in an optimun optimum concentration decreased IFT values. MES surfactant used in this study had an optimum salt concentration of 5000-25000 ppm. If salt concentration in the solution was above 5000 ppm, IFT values of the solution would start to increase as shown in Figure 2 and 4. This could happen because disodium carboxy sulfonic was formed when salt in the solution was over the optimum salt limit of the surfactant itself. Salt existence in a solution containing

ISBN : 978-1-4673-7405-7

MES would make MES lose its lost its facial active property as MES would get involved in a reaction to form disodium carboxy sulfonate (di-salt). Anionic surfactant (MES) which was originally bonded to a Na molecule would bind to more Na ions from salt (NaCl) so that there would be one molecule containing two Na ions [15,16]. This resulted in a decreased surfactant performance and higher IFT values. The reaction mechanism of disodium carboxy sulfonate formation is depicted in Figure 5..

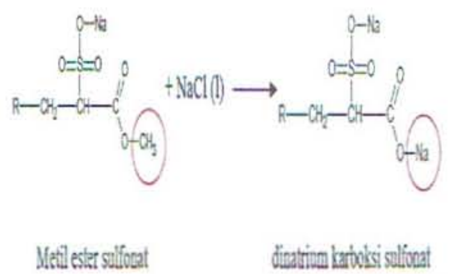

Fig. 5.Reaction mechanism of disodium carboxy sulfonate formation

With similar salt concentration and MES surfactant concentration (0.3%), IFT values of samples of demineralized water dispersion medium were lower than those of formation water. The lowest IFT value was obtained at salt concentration of 0.5% or 5000 ppm which was closed to salt concentration in formation water. Results of the test done at three temperatures with salt addition of 5000-7500 ppm in demineralized water dispersiom medium showed that the IFT values were $2.64 \times 10^{-4} - 1.3 \times 10^{-3}$ dyne/cm. These values were lower than those obtained from formation water ($1.10 \times 10^{-3} - 4.40 \times 10^{-3}$ dyne/cm). This might be caused by less amount of electrolytes, in addition to NaCl, in demineralized water than in formation water. The existence of other electrolytes originating from other molecules was suspected to inhibit the reaction to reduce IFT values by surfactant

Hard water, like formation water used in this study, contains cations including Ca^{2+} (12.2 mg/L) or Mg^{2+} (9.08 mg/L). Physicochemical properties of water are given in Table 1. Cation concentration is water with high hardness level. MES surfactant is an anionic surfactant with active group containing negative charge. When this surfactant meet hard water, the active group would form a bond with Ca^{2+} or Mg^{2+} ions. The bonds between negative ions in surfactant and these cations would decrease performance of MES surfactant at lowering interfacial tension as indicated by high IFT values. The undissolved component formed is $(RCH(SO_3Na)CO_2Ca$ [17]. This undissolved component in surfactant solution decreases surfactant solubility in water making the performance of MES surfactant in lowering interfacial tension reduced.

TABLE 1
PHYSICOCHEMICAL PROPERTIES OF FLUID TAKEN FROM SANDSTONE FIELD WATER USED IN THIS STUDY

Parameter	Unit	Injection Water	Formation water	Methods *) Part Number
Anion				
SO_4^{2-}	mg/L	<1.44	<1.44	4500-SO_4^{2-}E
HCO_3^-	mg/L	2947	1989	2320 B
Cl^-	mg/L	5105	3515	4500-Cl-D
Kation				
Na^+	mg/L	2933	2344	3120 B, 3030 E
K^+	mg/L	24.3	13.9	3121 B, 3030 E
Ca^{2+}	mg/L	25.7	12.2	3122 B, 3030 E
Mg^{2+}	mg/L	12.9	9.08	3123 B, 3030 E
Ba^{2+}	mg/L	0.73	0.14	3124 B, 3030 E
Sr^{2+}	mg/L	3.71	1.87	3125 B, 3030 E
Fe^{3+}	mg/L	0.26	0.1	3126 B, 3030 E
pH	mg/L	8.3	8.5	4500-H^+-B
Salinity as NaCl	mg/L	8417	5795	2520 B
Total Hardness as $CaCO_3$	mg/L	117	67.9	2340 B
Total Suspended Solid	mg/L	32	18	2540 D
Oil & Grace	mg/L	< 2	< 2	5520 B
Dissolved Oxygen	mg/L	5.59	5.74	4500-O-G

TABLE 2
PHYSICOCHEMICAL PROPERTIES OF FLUID TAKEN FROM SANDSTONE FIELD OIL USED IN THIS STUDY

Parameter	Measurement Temperature (°C)					
	40		50		60	
Density (g/cm^3)	0.91483		0.90803		0.90142	
Temperature	40.02		49.99		59.97	
API Density (g/cm^3) (15°C)	0.9314		0.9313		0.9313	
API Gravity (15°C)	20.27		20.3		20.28	
API Specific Grafity (15°C)	0.9323		0.9322		0.932	
Viscosity (cP)	25.60	25.24	16.53	16.45	11.69	11.76
Speed (rpm)	60.00	90.00	60.00	90.00	60.00	90.00
Torque (%)	51.22	75.73	33.09	49.36	23.39	35.28
Shear Stress	20.27	29.98	13.10	19.54	9.26	13.97
Shear Rate (1/s)	79.20	118.80	79.20	118.80	79.20	118.80

IV. CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

Performance of surfactant was affected by the addition of surfactant concentration and inorganic salt. Methyl Ester Sulfonic surfactant reacted well at salinity ranges of 5000–25000 ppm. Minimum IFT values in demineralized water dispersion medium were obtained at salt formula addition of 5000 ppm. The addition of 0.3% surfactant in formation water dispersion medium resulted in IFT values which met oil industry requirements.

ISBN : 978-1-4673-7405-7

B. Recommendations

Further studies on additive inclusion and other tests need to be done in order to make this MES surfactant ready for use in oil industry.

ACKNOWLEDGMENT

Facility supports from SBRC LPPM IPB were acknowledged.

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