SUBSTITUTION OF PETROLEUM BASE WITH MES BASE SURFACTANT FOR EOR: LABORATORY SCREENING

MENGGANTI SURFAKTAN BERBAHAN DASAR PETROLEUM DENGAN MES UNTUK EOR: PENYARINGAN LABORATORIUM

Sugihardjo and **Hestuti Eni**

"LEMIGAS" R & D Centre for Oil and Gas Technology
Jl. Ciledug Raya, Kav. 109, Cipulir, Kebayoran Lama, P.O. Box 1089/JKT, Jakarta Selatan 12230 INDONESIA
Tromol Pos: 6022/KBYB-Jakarta 12120, Telephone: 62-21-7394422, Faxsimile: 62-21-7246150
E-mail: sugihardjo@lemigas.esdm.go.id; E-mail: hestuti@lemigas.esdm.go.id

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ABSTRAK

Sebagian besar lapangan minyak Indonesia telah dikategorikan sebagai lapangan tua dengan produksi yang telah menurun untuk beberapa waktu. Maka teknologi EOR (Enhanced Oil Recovery) merupakan keharusan untuk diimplementasikan pada lapangan lapangan tersebut. Ada beberapa teknologi EOR yang telah sukses dilaksanakan pada skala laboratorium maupun lapangan, termasuk injeksi gas, panas, dan kimia. Sebagian besar lapangan di Indonesia mempunyai lapisan produksi dengan kedalaman dibawah 2200ft yang tidak akan cocock diinjeksikan gas. Injeksi kimia menjadi alternative penting yang dapat diimplementasikan pada variasi kedalaman yang luas. Teknologi ini meliputi injeksi alkali, surfaktan, dan polymer. Tulisan ini akan menunjukkan seleksi dan formulasi surfaktan yang diformulasikan dari MES (Methil Ester Sulfonate) yang diproduksikan dari minyak sawit. Beberapa produk Minyak Sawit tersedia melimpah di Indonesia karena banyak perkebunan sawit di Indonesia. Normalnya surfaktan diformulasikan dari petroleum sulfonate dengan bahan dasar petroleum. Dengan menggunakan surfaktan yang akan diproduksikan dari minyak sawit, dapat diharapkan harga akan lebih murah dibandingkan surfaktan dengan bahan dasar petroleum. Satu seri penelitian telah dikerjakan untuk menyeleksi sumber minyak sawit, memproduksi MES dengan proses sulfonasi, dan akhirnya screening surfaktan untuk EOR. Beberapa tipe MES yang diproduksi dari beberapa minyak sawit yang dibeli di pasaran seperti: minyak curah, beberapa minyak kemasan dengan merk yang berbeda telah dibuat. MES 2 ini kemudian diberi tanda untuk membedakannya yaitu: 1. MES CCO (A), 2. MES ME (B), 3. MES Oleic Acid (C), 4. MES-1 Natrium Bisulfit (D), 5. MES-2 ME+H2SO4 (E), 6. MES-CPO (F). Produksi MES ini kemudian diformulasikan untuk menjadi formula surfaktan dengan menambah bahan kimia dan pelarut. Kemudian dioptimasi dengan penambahan alkali (Na2CO3). Semua Surfaktan-MES telah diuji dengan standar uji EOR laboratorium Lemigas yang meliputi: Uji Kompatibilitas, IFT, Thermal Stability Test, Uji Adsorbsi, Uji Filtrasi, Kelakuan Fasa, Imbibition Test, imbibisi, dan Uji core flooding. Hasil uji laboratorium campuran MES-bahan kimia menunjukkan bahwa campuran MES-CPO (F) dengan bahan kimia dan pelarut dengan kode FChS811 mempunyai unjuk kerja paling bagus. Campuran 1% bahan tersebut mempunyai sifat-sifat terbaik untuk EOR setelah ditambah 0.1% Alkali (Na_2CO_3). Hasil uji laboratorium menunjukkan memenuhi kriteria penyaringan seperti: kompatibel dengan fluida formasi, IFT rendah, stabil pada kondisi panas, adsorbsi rendah, filtrasi rasio rendah, kelakukan fasa menunjukkan Winsor tipe-1, RF tinggi dari hasil uji imbibisi dan coreflooding. Selanjutnya formulasi surfaktan ini mempunyai potensi untuk dilakukan uji coba injeksi surfaktan secara Huff and Puff.

Kata kunci: MES (Methil Ester Sulfonat), formulasi, Peningkatan Perolehan Minyak, Perolehan Minyak

ABSTRACT

Most of Indonesian oil field had been categorized as mature filed in which production had been declined for some time. Therefore EOR (Enhanced Oil Recovery) technology is a must to be implemented to these *kinds of field. There are several EOR technologies had been employed successfully in laboratory and also field scales, including gas, thermal, and chemical injection. Most Indonesian oil fields have productive layers depths below 2200ft that will not suitable for gas injection. So that chemical injections become an important alternative that can be implemented to more wide range of depths. These technologies cover alkaline, surfactant, and polymer injection. This paper will highlight the selection and formulation of surfactant formulated from MES (Methyl Ester Sulfonates) produced from Palm Oils. These palm oils are available very abundant in Indonesia due to plenty farm palm oil in Indonesia. Normally Surfactants are formulated from petroleum sulfonates which are generated from petroleum base. By Using Surfactant that will be manufactured from palm oil, it will be expected that the price will be cheaper compare to the surfactant from petroleum. A series of researches have been done to select the sources of palm oils, producing MES by sulfonation processes, and finally surfactant screening for EOR. Several types of MES produced from varies of palm oil taken from market such as: CPO (crude palm oil), several packed palm oils of different trademarks have been generated. These MES, then, have been given codes to differentiate among these MES such as: 1. CCO-MES (A), 2. CCO ME-MES (B), 3. Oleic Acid- MES (C), 4. Natrium Bisulfit- MES1 (D), 5. ME+H2SO4-MES2 (E), 6. CPO-MES (F). These MES production, then, have been formulated to become surfactant formula by adding some chemicals and solvent. After that alkaline ((Na2CO3) with optimized concentrations were added to generate the best EOR properties. All those Surfactant-MES have been tested using Lemigas standard laboratory EOR screening; those are compatibility tests, IFT measurements, thermal stability, adsorption, filtration, phase behavior, imbibitions and core flooding. The result of the screening of the MES-chemicals mixtures shows that mixture of CPO-MES (F) with chemical and solvent with the mixture composition denoted as FChS811 has the best performance. 1% of this mixture has the best properties for EOR after adding 0.1% of Alkaline (Na2CO3). Laboratory test results indicates that fulfill screening criteria suh as good compatibility and no precipitation, low IFT, thermal stability, low adsorption, low filtration ratio, Winsor type-I phase behavior, high RF on imbibition and core flooding tests. This Surfactant-MES mixture has a potential to be implemented for a field trial with Huff and Puff method.*

Keywords: *MES (Methyl Ester Sulfonates), EOR (Enhanced Oil Recovery), RF (Recovery Factor)*

I. INTRODUCTION

The need of EOR technology is very urgent for Indonesian oil field considering that most of oil fields had been matured for long time, while remaining oil left behind in the reservoirs are still very high. For example PERTAMINA EP produces almost 85% from brown fields at primary stage with more than 5 billion barrels oil remaining reserves (Kurnely et al, 2006). EOR with chemical injections are classified as a proven technology and have been successfully implemented in field scale for example Daqing oil field in China (Zhu et al. 2012). While in Indonesia field trials had been carried out in two fields recently for instance Minas ASP (Alkaline-Surfactant-Polymer) and Kaji SP (Surfactant Polymer) floods. Eventhough, the results of both floods have not been published yet.

Surfactant is very essential in chemical injection which its ability to reduce interfacial tension between injected fluid (water phase) to displaced fluid (oil phase) can create more efficient displacement in microscopic way. To determine the surfactant performance can be used CDC (Capillary desaturation curve) which depicts the relationship between ROS and capillary number (Nc). Nc is the ratio of viscous to capillary force. This dimensionless ratio of Nc has been given many expressions in the literature, where the most applied definitions are:

$$Nc = V\mu/(\sigma \cos \theta)$$

V : apparent velocity (m/s), μ : viscosity of displacing fluid (Pa.s), σ : interfacial tension (mN/m),

θ : wetting angle

The forces responsible for retaining oil in a porous media are a complex function between viscous and capillary forces, and are influences by several parameters such as permeability, pore size distribution, wettability, saturations, fluid distribution and saturation history (Garnes et al. 1990). Figure 1 shows the Nc curve proposed by Sughardjo 2009. This picture clearly explain that Nc number can drastically reduce residual oil at CDC (capillary Desaturation Curve). Beside that lowering IFT also can change the relative permeability curve into more higher value and when IFT is approach zero then the relative permeability approach straight lines.

This paper is proposed to substitute the normal used petroleum based by MES based surfactant, which is the availability very huge in Indonesia due to wide palm oil farm. Oleochemical surfactant derived from palm oil and petrochemical surfactant derived from crude oil, both have surfactant properties and can be used for EOR to reduced the interfacial tension. Oleochemical surfactant have sulfonates group the same as petrochemical surfactant, but the different is that oleochmeical similar to the other organic materials are susceptible to high salinity and high temperature.

To generate MES-Surfactant having chemical and physical properties that can withstand in hard environment such as high pressure, elevated temperature, high salinity and hardness is very challenging. Adding some chemicals to create appropriate formula should be done to adapt MES-Surfactant at the reservoir condition which MES is originally very susceptible with those hard environments. Basically MES solution in water has an interfacial tension (IFT) with oil in the range 10^{-1} to 10^{-2} dyne/cm. To produce surfactant MES with 10^{-3} dyne IFT or lower is necessary adding some additional solvent and chemical. Adding alkaline at low concentration is also necessary which is also could reduce adsorption level, solution phase behavior equilibrium time and also better thermal stability characteristic.

II. METHODOLOGY

Surfactant used in this experiment is nonpetroleum based which is formulated from palm. The methodology for this research consists

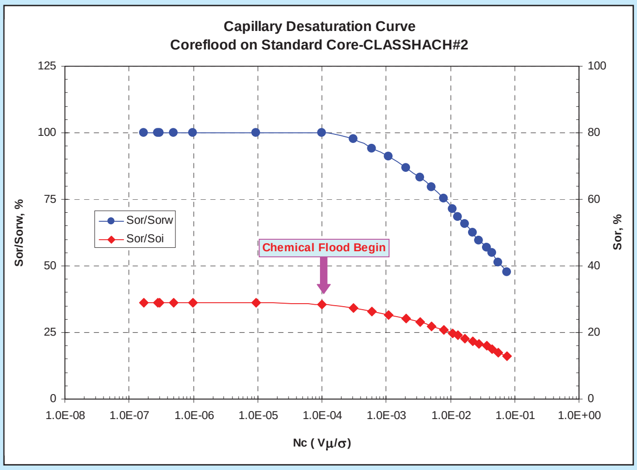

Figure 1
CDC at High Nc (After Sugihardjo 2009)

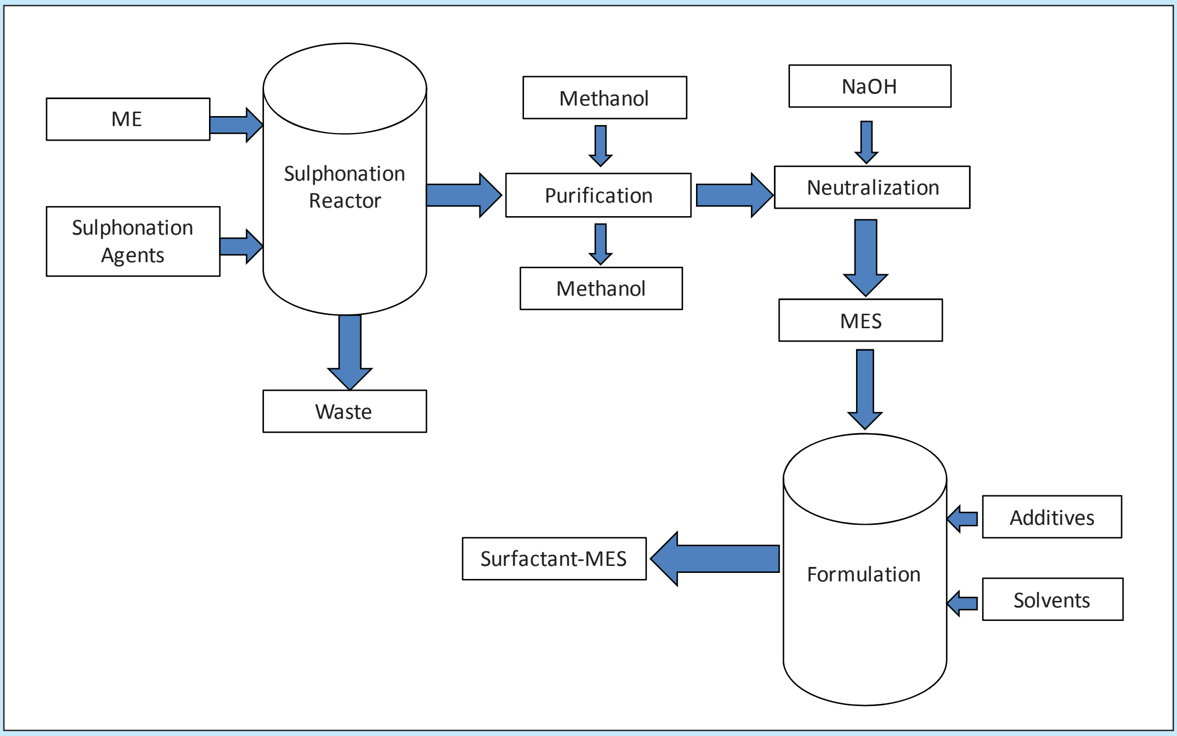

Figure 2
Formulation and Screening Processes of Surfactant-MES

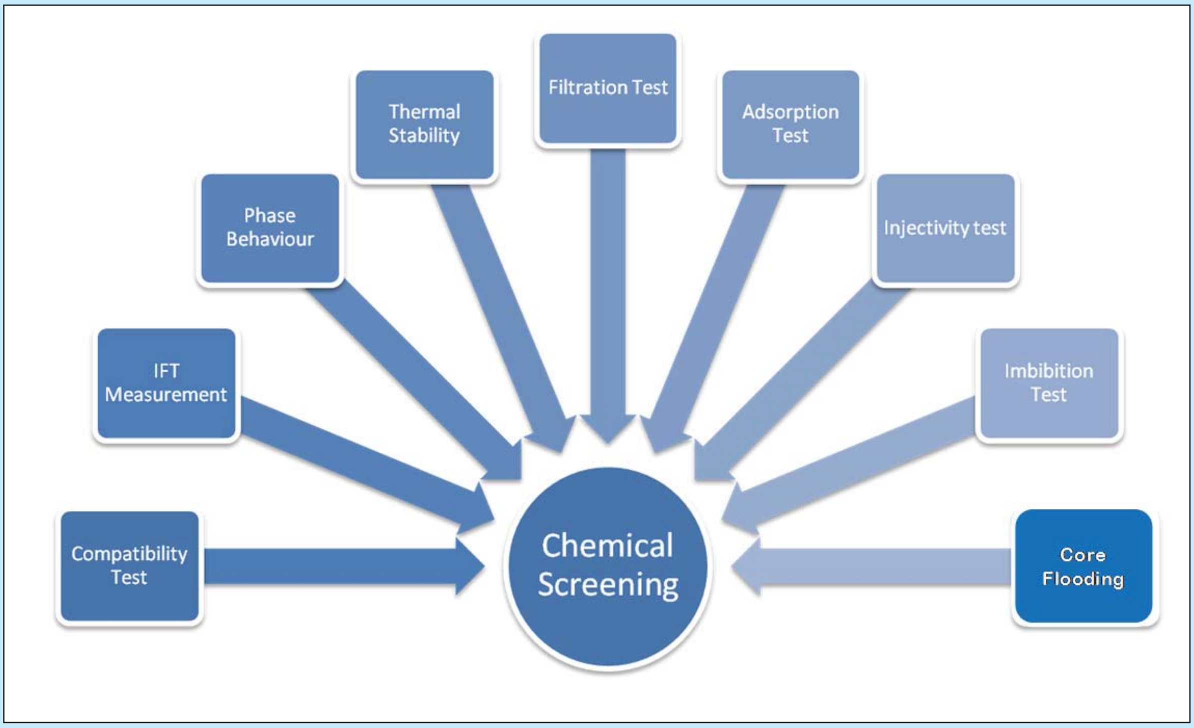

Figure 3
Detail Screening Processes For EOR

of two steps. First step, Palm oil which normally contains *oleic*, *stearin* and other components is put into the estherification processes to produce methyl ester (biodiesel). Subsequently, this ME is sulfonated using sulfonating agents, to promote Methyl Ester Sulphonates, MES. To get MES with expected properties, optimum time span and temperature to be adjusted during sulfonation processes (Astrini et. al., 2007). MES has been categorized as surfactant due to have surfactant properties such as a capability to reduce interfacial tension between water and oil.

The second step, MES from the above experiments, then it is exercised the physical properties by adding some chemicals and solvent. To fulfill EOR screening criteria, mixtures of MES then tested their properties includes compatibility tests, IFT measurements, thermal stability, adsorption, filtration, phase behavior, and core flooding. Figure 2 and 3 depict the formulation and screening processes.

This Surfactant-MES screening should be done to complete the EOR screening criteria successfully and afterward this will be proposed as chemical solution for injection with Huff and Puff method in one of the Java oil field. Therefore sampling of oil and water has been carried out for this project. Besides that, some reservoir data such as reservoir pressure, temperature, porosity, thickness have been also collected. The field has been produced from the layer depth of 670m having reservoir properties of 58^{o}C temperature, 528 psig current pressure, 20% porosity, 70mD average permeability, 40.35^{o}API, and 1.347cp oil viscosity. These data has been used as the parameter test during screening tests.

III. RESULTS AND DISCUSSION

A. Formation Water Analysis

Initial analysis has been done to determine salinity of formation water that will be used to make chemical solutions. Formation water analysis result in the salinity of water is approximately 18,200 ppm with hardness of Ca^{2+} 143.5 mg/l and Mg^{2+} 73 mg/l. On the other hand there was not available native formation rock and standard core therefore used in the core flooding experiment.

B. MES Production

Several type of MES have been from produced sulfonation processes, those are around 6 type of MES with different initial raw material and sulfonation processes (reaction time, temperature, and sulfonating agents). Tabel 1 is the MES types while Figure 4 is the photographs of those MES in bottles. The original MES before added some chemicals were also tested for their interfacial tension to oil to select the most prospective for enhanced oil recovery. Table 2 shows IFT measurement results.

Comparison of IFT numbers among MES, three MES have IFT in the order of 10^{-3} dyne/cm for instance MES-A, MES-E, and MES-F. Those three MES, then, subject to be further evaluated.

C. Compatibility Test

Compatibility is indication that the solution mixtures of chemical such as Surfactant-MES and the formation water do not create precipitation or coagulation, in the contrary is incompatibility. Some surfactant solution may precipitate in a high salinity and hardness formation water. in addition, some surfactants may generate hazy solutions, although a clear solution is more preferable, however just because it is hazy does not mean it is not injectable. The key for deciding injectivity of hazy solution is to make sure that the solution is thermodynamically stable (equilibrated) system which will keep its micellar properties when injected. Figure 5 is the results of the compatibility tests of 1% all Surfactant-MES solution.

The results of the preliminary compatibility tests indicated that all MES produced some coagulations on top liquid surface reveal that they still needed some additive to become better soluble solution.

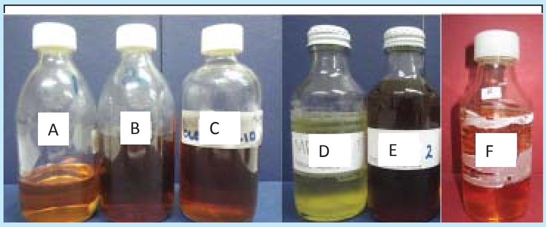

Figure 4
MES solution in Bottles

Table 1
MES Production

No.	Type of MES	Remark
1	MES CCO (A	Crude Coconut Oil
2	MES ME CCO (B)	Methyl Ester
3	MES Oleic Acid (C)	Meth
4	MES-1 Natrium Bisulfit (D)	Sulphonation using NaHSO4
5	MES-2 ME+H2SO4 (E)	Sulphonation of Methyl Ester Using H2SO4
6	MES Curah (F)	Sulphonation of CPO Crude Palm Oil Using H2SO4

Table 2
IFT Measurement Results

Concentration	IFT (Dyne/cm) Surfactan					
%	MES A	MES B	MES C	MES D	MES E	MES F
0.1				4.590E+00	3.530E+00	
0/3	3.800E-03	1.260E+00	2.604E+00	4.400E+00	4.571E+00	8.673E-01
0.5	4.200E-03		1.654E+00	3.731E-03	7.080E-03	1.455E-01
0.8				4.720E+00	5.408E-03	6.760E-03
1.0	3.000E-03	1.326E+00	2.931E-01	4.658E+00	6.100E-03	7.740E-03
1.5	8.850E-02		4.700E-03			2.770E-03
2.0		5.320E-02	8.730E-02	5.253E+00	1.514E-02	
2.5	4.962E-01		1.136E-01			
3.0			1.687E-01			

D. Formulation of Surfactant-MES and IFT Measurement

Three MES of the six MES such as MES-A, E and F have been chosen for the next formulation step to become Surfactant-MES by adding some chemical (Ch)and solvent (S). The formulations have been coded as MChSxyz, X is the name of MES (A, or E, or F), Ch: Chemical, S: Solvent, with solution ratio x:y:z. Then, All solution have been added with alkaline (Na_2CO_3) around 0.1% to reduce loss of surfactant concentration due to adsorption and to faster equilibrium solution. Normally the added Alkaline should be as low as possible to anticipate scaling tendency.

The three variety of solution ratios of each MES have been formulated and investigated their compatibility. Figure 6 exhibits the compatibility of the surfactant-MES formulation. The solutions are very clear without any coagulation and precipitation solids.

After that, All the solutions were measured the order of IFT with the oil. Table 3 is the result of IFT measurement after formulation with MES concentration from 0.3% up to 2.0% and variation of ratio concentration of chemical and solvent in the solution. In general, solution ratios of 811 produce lower IFT compare to the other two ratios (841, and 181). Therefore this 811 ratio of the thee MES have been further intensively investigated their properties for EOR.

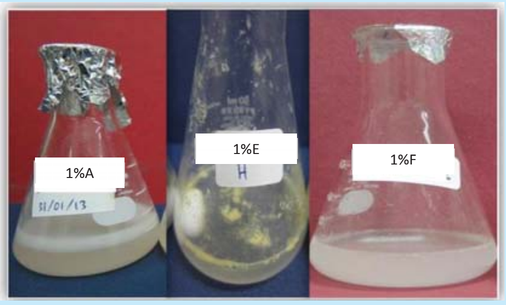

Figure 5
The Result of Compatibility Tests

Figure 6
The Result of Compatibility Tests
After Formulation

Table 3
IFT Measurement After Formulation

Surfactant-MES Formulation		IFT (Dyne/cm) 0.3%	0.5%	1.0%	1.5%	2.0%
AChS	811	2.097E-03	9.361E-03	8.640E-03	2.429E-02	4.585E-03
	841	6.859E-03	2.136E-02	4.199E-02	9.106E-02	9.698E-02
	181	6.206E-02	7.832E-02	1.200E-01	2.018E-01	1.443E-01
EChS	811	3.460E-03	5.670E-03	1.008E-02	4.133E-02	5.398E-02
	841	3.447E-03	1.705E-03	4.782E-02	1.153E-01	5.454E-02
	181	3.823E-02	6.507E-02	7.284E-02	7.888E-02	6.966E-02
FChS	811	5.542E-03	1.147E-02	9.008E-03	2.672E-02	1.029E-01
	841	7.223E-03	1.268E-02	5.469E-02	1.023E-02	7.325E-03
	181	8.994E-02	1.627E-01	1.023E-01	1.095E-01	9.021E-02

Based on above IFT value and considering the availability of raw material of MES production and the price, as a result it was concluded that MES-F has been selected for more focusing on the next steps of screening.

E. Phase Behavior

Phase behavior is to see surfactant preference to be soluble in water, oil phase, or in between. If surfactant prefer more water than oil is categorize as water phase or Winsor type-1, on the contrary prefer more oil is called oil phase or Winsor type-2. If surfactant makes middle solution is classified as middle phase or Winsor type-3 (Sugihardjo, 2008). FChS811 has been tested for phase behavior with concentration of MES-F from 0.3% up to 1.0%. The results indicate that all solutions forming water phase solution or Winsor type-1.

F. Thermal Stability

The direct technique to determine the thermal stability is by measuring the interfacial tension versus time. This, presumably, would measure directly the loss of the property of importance in oil recovery. Figure 7 exhibits the measurement of IFT during thermal stability test for only 30days. Because of the time span and temperature any surfactant may experience degradation, broken chemical bond (thermal decomposition), and change of their properties. Therefore this results can only be proposed for Huff and Puff injection plan.

F. Filtration Test

The work is very simple just measure volume versus time when fluid flows through a filter paper. Surfactant normally is screened using 0.22 micron filter paper. A filtration paper which has a certain pore size will help discern whether a surfactant solution has a single phase fluid or a dispersion of one phase in another. Filtration ratio (FR) of 1.2 normally is still tolerable. Below is the FR of the tests:

G. Adsorption Test

In this experiment the static method has been proposed due to easier and more practical. Static adsorption tests can provide a preliminary screening of surfactants. The tests are fairly simple and inexpensive compared to procedures involving flow in cores. Crush the core and contacted with the surfactant solution for several days and then calculate the concentration reduction and divided by weight of crushed core. The result of the static adsorption tests is presented Table 4. Adsorption surfactant on to rock are about 407 and 459 mgr/gr rock.

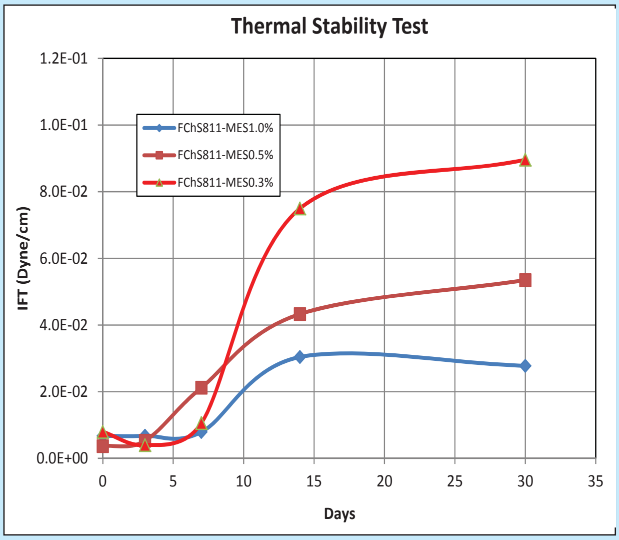

Figure 7
Thermal Stability Test

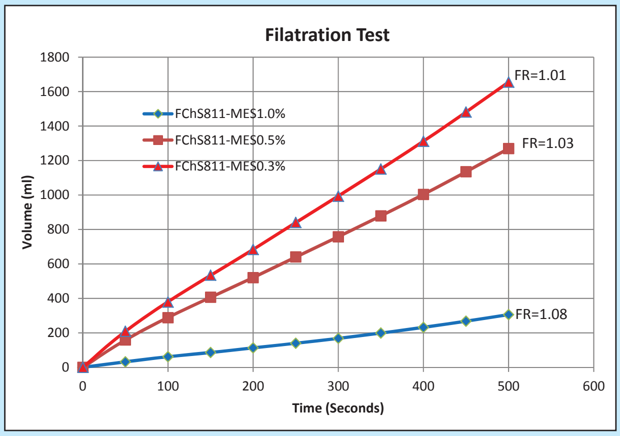

Figure 8
Filtration Test Results

Table 4
Adsorption Results

Formula	MES-Concentration	Adsorption (μg/g)
FChS811	0.3%	407
FChS811	0.5%	423
FChS811	1.0%	459

H. Imbibitions Test

Due to unavailable a native core, so a standard core "Bentheimer" has been used in this experiment to investigate imbibitions ability of this surfactant-MES displacing oil from the cores. 1% of surfactant-MES concentration of FChS811 has been considered to be applied in this experiment to take account some surfactant will be loss by rock adsorption. The result of the experiment is presented in Table 5, which RF of the imbibitions is approximately 92.25%

I. Coreflooding and Injectivity

Same as imbibitions a standard core also used in this experiment. The core flood has been designed to use 0.5 PV injection of 1% Surfactant-MES of FChS811 after water flood terminated. The recovery

Table 5
Imbibitions Results

No Sample	Fluid	PV. (cc)	Por. (%)	K (mD)	Swc.(%)	Soi.(%)	RF.(%)
1	Water	18.480	22.447	2620.0	20.18	79.82	41.69
2	1% Surfactant-MES	18.880	23.098	2664.0	28.23	71.77	92.25

Table 6
Core flood Results

Core Plug No.	Porosity (%)	Permeability to water (mD)	PV (cc)	Soi (%)	Swc (%)	RF Water Flood (%)	RF Surf Flood (%)
1	27.57	515.785	72.81	80.28	19.72	53.64	16.51

Table 7
Comparison Between LEMIGAS' Standard and Test Results

No.	Test Parameters	LEMIGAS' Standard	Formula-MES
1	Compatibility	clear solution no precipitation	clear solution no precipitation
2	IFT	<10-3 Dyne/cm	9.008E-03 Dyne/cm
3	Thermal Stability	- 3 months for pattern flood	
		- 1 month for Huff nad Puff	- 1 month for Huff nad Puff
4	Adsorption	< 400 mg/g	459 mg/g
5	Filatration	FR<1.2	1.08
6	Phase Behavior	Winsor Type-I or III	Winsor Type-I
7	Imbibition	Bigger than RF water	RF-WF: 41.69% RF-SF: 92.25%
8	Injectivity	20% PRF	0% PRF
9	Coreflood	12% above water flood	RF-SF: 16.51%

factor of surfactant flood over water flood is around 16.51%. Table-6 is the data of core flooding.

Injectivity t of surfactant flood normally has no problem and during flooding there was no permeability damage indication and no plugging observed. The permeability to water was 515mD and no pressure fluctuation during flooding indicated that there was very good injectivity.

Originally six of MES produced from different feedstocks have been preliminary tested of their IFT with the reservoir oil. The best three have selected having the IFT value in the order 10^{-3} dyne/cm i.e.: A-MES, E-MES, F-MES. Those three surfactants then have been optimized by adding some chemicals and solvents and the results reveal that those three surfactants basically have almost similar IFT performance. Those three MES have better IFT Values may be resulted of optimized process conditions such as appropriate volume of sulfonating agents, temperature, and reaction time.

Considering the raw material availability and price, therefore F-MES with 811 compositions has been selected for further screening for EOR. Basically F-MES has been produced from CPO which is large quantity available in Indonesia. Furthermore, 811 is optimized composition with lower chemical and solvent concentrations.

F-MES surfactant produced from palm oil will be very potential for substituting surfactant-crude oil based normally used in oil industries. This preliminary research may have some new findings that can bring into more prospective material for EOR. Therefore, the results of all screening tests should be compared to the LEMIGAS' Screening EOR guidance. Table 8 is the comparison between Surfactant-MES formula and the screening guidance.

In comparison between the two group parameters, most of screening parameters of Surfactant-MES of FChS811 are fulfilled the screening guidance and only two of them have different values such as: thermal stability, and adsorption. Thermal stability is only tested and stable for approximately 1 month, furthermore the adsorption values are a little higher than the guidance. All those parameters have been approached the standard criteria after several trials of adding different kinds and concentrations of chemicals and solvents.

IV. CONCLUSION

Oleochemical surfactant derived from palm oil and petrochemical surfactant derived from crude oil, both have surfactant properties and can be used for EOR to reduced the interfacial tension. Therefore, surfactant-MES has surfactant properties which can be used to substitute petroleum base surfactant for EOR.

To formulate and to generate surfactant-MES to substitute the normal used petroleum based is very challenging by adding some chemicals and solvent in order to fulfill EOR screening parameters.

A formulated surfactant-MES of FChS811 has surfactant properties that fulfill the EOR screening criteria for Huff and Puff injection.

To use this kind of surfactant for pattern injection, improvement should be carried out with respect to thermal stability and adsorption.

Native cores are necessary to conduct core flood and imbibitions tests to get the proper recovery factor number as the basis of simulation calibration works.

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