

A STUDY OF SPONTANEOUS IMBIBITION RECOVERY MECHANISM OF SURFACTANT FORMULATED FROM METHYL ESTER SULFONATES

STUDI MEKANISME PEROLEHAN IMBIBISI SPONTAN DARI SURFAKTAN DIFORMULASIKAN DARI MESTIL ESTER SULFONAT

Sugihardjo

“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, Faksimile: 62-21-7246150
E-mail: sugihardjo@lemigas.esdm.go.id

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ABSTRAK

MES (*Methyl Ester Sulfonate*) dapat diformulasikan menjadi Surfaktan-MES yang mempunyai sifat sifat potensi untuk EOR (*enhanced oil recovery*). Formula surfaktan-MES semacam ini ditambah pelarut dan bahan kimia untuk memperbaiki sifat sifatnya agar dapat mempunyai kompatibilitas terhadap fluida dan batuan reservoir telah dikembangkan. Formula surfaktan-MES ini, kemudian, telah diuji sifat sifatnya untuk dapat digunakan dalam *enhanced oil recovery* pada lapangan minyak terpilih. Lapangan minyak ini memproduksi minyak dengan °API sekitar 39.35, dan air formasi mengandung garam 18,900 mg/L padanan konsentrasi NaCl. Sementara batuan reservoir tidak tersedia dan diganti dengan batu inti *standard Bentheimer*. Pada dasarnya larutan surfaktan untuk meningkatkan produksi pada proses EOR dikarenakan perubahan sifat kebasahan batuan and penurunan tegangan antarmuka. Pada percobaan ini, evaluasi telah dikerjakan untuk mengamati kemampuan surfaktan MES yg diformulasikan pada peningkatan produksi minyak termasuk mengubah sifat kebasahan batuan and kemampuan imbibisi. Surfaktan MES dengan tegangan antar muka rendah yang diformulasikan yang akan dievaluasi kemampuan imbibisi spontan dilarutkan ke dalam air formasi dengan konsentrasi 0,5 dan 1,0%. Uji perubahan sifat kebasahan batuan menunjukkan bahwa larutan surfaktan tidak dapat mengubah sifat kebasahan batuan. Pada awalnya sifat kebasahan batuan adalah basah campuran dan masih basah campuran setelah direndam dalam larutan surfaktan. Sebaliknya uji imbibisi spontan menunjukkan hasil peningkatan produksi minyak yang tinggi keluar dari batu inti, yaitu 67,07% peningkatan perolehan minyak dengan surfaktan 0,5% dan 92,25% dengan 1,0%. Tambahan bahwa peningkatan perolehan minyak optimum dengan imbibisi air formasi hanya 41,69%

Kata kunci: Metil ester sulfonat, perubahan sifat kebasahan, penurunan tegangan antar muka, imbibisi spontan, peningkatan perolehan.

ABSTRACT

MES (*Methyl Ester Suffocates*) can be formulated to become surfactant-MES that has surfactant potential properties for EOR (*enhanced oil recovery*). A such formulated surfactant MES by adding some solvents and chemicals to adjust its properties in order to generate a compatibility with reservoir fluid and rock has been developed. This formulated surfactant MES, therefore, has been tested its properties for enhanced oil recovery in a selected oil field. The oil field produces an oil withc39.45°API, and brine content of about 18,900 mg/L equivalent NaCl concentration. While reservoir rock is not available and substituted by Bentheimer standard core. Basically the production enhancements of surfactant solution for EOR processes are wettability alteration and interfacial tension reduction. In these experiments, some evaluations have been done to observe the capability the formulated surfactant MES for oil production enhancements including

wettability alteration and imbibition capability. Formulated low interfacial tension surfactant-MES that will be evaluated for spontaneous imbibition ability have been diluted in the brine of 0.5 and 1.0% concentrations. Tests on wettability alteration indicated that the surfactant solutions could not change the wettability of the rocks. The initial wettability is mixed wet and still mixed wet after ageing in the surfactant solution. On other hand the spontaneous imbibition tests resulted in significant oil production coming out from the cores, i.e. 67.07% oil recovery with 0.5% surfactant and 92.25% for 1.0% surfactant solutions. But optimum oil recovery factor by brine imbibition is only 41.69%

Keywords: Methyl ester sulfonates, wettability alteration, interfacial tension reduction, spontaneous imbibition, recovery factor.

I. INTRODUCTION

Rock and fluid interactions in reservoirs have been recognized as the important factor for determining the recovery mechanism of oil during the life of reservoirs. One of the important category of rock and fluid interactions is characterized as wettability of rock. This rock property is absolutely affected by fluids, water and oil composition, and rock minerals present in the pores. Hence, Wettability is the important rock property which can determine fluid flow characteristics in two phases reservoirs and consequently controlling the magnitude of the optimum oil recovery.

Wettability means that rocks relatively prefer to be wetted by a certain phase compare to another. Rocks can be oil wet, water wet, and intermediate wets. Rock is defined to be water-wet if the rock has more affinity for water than for oil, the major part of the rock surface in the pores will be covered with a water layer. On the other hand, oil wet rock is in which the rock has more affinity for oil and a oil layer will cover surrounding the rock surface. The intermediate state between water-wet and oil-wet can be caused by a mixed-wet system, in which some surfaces or grains are water-wet and others are oil-wet, or a neutral-wet system. Neutral systems indicate that the rock surfaces are not strongly wet by either water or oil.

Clean sandstone or quartz is normally water-wet, but sandstone reservoir rock is usually found to be intermediate wet. On the other hand carbonate rocks are believed to be more oil-wet than clastics. Generally, polar compounds or asphaltene content in the crude oil can be deposited in mineral surfaces of rocks and cause the rocks tend to be oil-wet. Similarly, compounds in oil-base mud also can cause a previously water-wet rock to become partially or totally oil-wet (<http://www.glossary.oilfield.slb.com>). In the laboratory, wettability is characterized usually by the so called Amott and USBM indices

(Widarsono, 2010), while direct contact angle measurement may be also available using pendant drop analysis equipment.

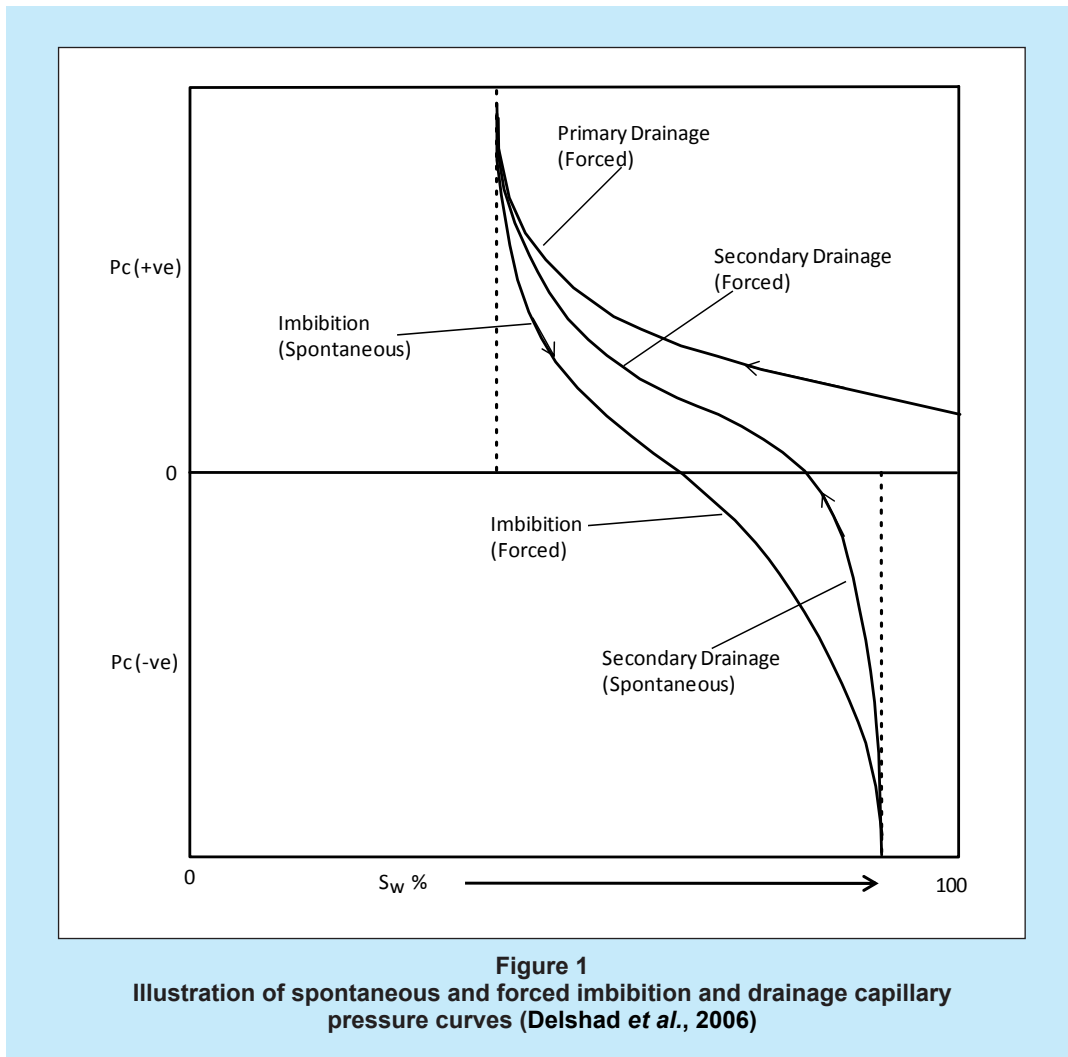
Wettability characteristic of rocks plays an important factor of imbibition of fluid into rocks. Oil-wet rocks preferentially imbibe oil, while in water-wet conditions will imbibe water phase. So, spontaneous imbibition is defined as an immiscible displacement process, whereby a non-wetting fluid within a porous medium is spontaneously expelled by wetting fluid that surrounds the medium and is drawn into the medium by capillary suction. In addition, the process of forcing a nonwetting phase into a porous rock is called forced imbibition. Another process is called drainage for example oil migrates into most reservoirs as the non-wetting phase.

In water-wet conditions, a thin film of water coats the surface of the formation matrix, a condition that is desirable for efficient oil transport. Imbibition has long been recognized as a key parameter in recovering oil water wet reservoirs. Therefore, water imbibition is fundamental to both water flooding and chemical injections at tertiary stage. Figure 1 shows the drainage-imbibition process during coreflooding.

A semi-empirical scaling group to correlate spontaneous imbibition behavior of VSWW (very strongly water wet) media over a wide range of condition has been used by Ma et al (Ma *et al.*, 1977). It has been summarized as follow:

$$t_D = t \sqrt{\frac{k}{\phi}} \frac{\sigma}{\sqrt{\mu_o \mu_w}} \frac{1}{L_c^2}$$

where t_D is dimensionless time, t is time, k is permeability, ϕ is porosity, σ is the interfacial tension, and μ_o and μ_w are the oil and brine viscosities. L_c is a characteristic length that compensates for sample size, shape and boundary conditions.



It is clear that the optimum oil recovery will be also dictated by wettability of reservoir rock. In addition in natural fractured reservoirs the only mechanism could support the production of oil in the matrix will be controlled by the magnitude of spontaneous imbibition of water from the fractures to the matrix and through the fractures to the production well. The recovery can be improved in this case by using chemicals which can stimulate decreasing IFT, changing matrix wettability, and increasing viscosity. Moreover those chemicals can develop transport mechanisms between fracture and matrix by capillary imbibition, buoyancy, viscous forces, diffusion, marangoni effect/spontaneous emulsification.

Spontaneous Imbibition rate of wetting phase can be influenced basically by interfacial tension, viscosity of oil and water phases, core geometry,

the exterior core surface boundary conditions, the detailed core structure, and microscopic boundary condition that determining wettability. An average microscopic pore radius can be defined which is proportional to the square root of permeability over porosity (Ma *et al.*, 1999).

Some parameter will influence spontaneous imbibition of wetting phase into porous medium including interfacial tension, wetting and non wetting phase viscosity, sample size, shape and boundary conditions, and initial wetting phase saturation for a variety strongly water wet rocks that consequently controlling the rate of oil recovery.

Initially research on spontaneous imbibition was focused on fractured reservoirs. Many fractured reservoirs exhibit prolific initial oil production from the fractures, but this can be short-lived. Under water

injection or aquifer drive, the subsequent recovery of oil from the rock matrix, if any, is mainly dependent on the spontaneous imbibition of water, a relatively low process. The recovery depends on crude oil/brine/rock (COBR) interactions.

Countercurrent imbibition, in which water and oil flow through the same face in opposite direction. Oil saturated cores are immersed in water, or sealed such that water in-flow and oil out-flow occur through the same face. Countercurrent imbibition had a slower recovery than the concurrent imbibition. If water inflow and oil outflow through the different faces is called cocurrent imbibition. Oil are mostly produced by cocurrent imbibition rather than counter current (Morrow *et al.*, 2001).

This paper will present a highlight of researches on surfactant solutions of non petroleum based (surfactant-MES) used as an agent for imbibition processes for improving oil recovery in laboratory scale. In addition to that wettability alterations will be also evaluated to support imbibition phenomena.

Basically all surfactants used in EOR projects are formulated from petroleum sulfonates. Therefore, the price of surfactant dependants on oil prices. A country likes Indonesia, where production of palm oil very huge, surfactants with palm oil based will be much cheaper for EOR project. But researches and efforts on the formulation must be achieved continuously in order to fulfill the screening criteria for surfactant flooding.

II. REVIEW OF PAST RESULT ON SPONTANEOUS IMBIBITION RESEARCHES

Carbonate rocks are very common to have double porosity and permeability which permeability could range from 7 to 4000 md, and the wettability tend more towards oil wetness than sandstones. The recovery of oil is much fewer compare to sandstone. Experiment have been done to investigate the effect of spontaneous imbibition in limestone rocks after wettability alteration. The result of spontaneous imbibition indicated that VSWW (very strong water wet) rock had the highest oil recovery compare to the MXW (mixed wet) rocks. The present of Initial water saturation and types of grain stone also have significant effect on oil recovery of MXW (Tie *et al.*, 2005).

Research on fractured carbonate reservoirs had been done by Zhang at all using new chemical as natural imbibition enhancers (Zhang *et al.*, 2008). For improving the oil recovery in mixed and oil wet fractured carbonate reservoirs, a novel experimental method has also been developed to quantify the significant of capillary and emulsification driven imbibition. Surfactant and alkali chemicals were used to demonstrate capillary and emulsification driven natural imbibition mechanism. Co-current and counter current imbibition could individually be explained by these mechanisms. Surfactant were shown to enhance natural imbibition mainly by emulsification and solubilization, while alkali were shown to enhance natural imbibition primarily by mechanism of wettability alteration.

Experiment on sort and long cores of fractured carbonate reservoirs have been done to demonstrated the ability of surfactant for enhancing oil recovery. The experiment were designed to decipher contributions from rock matrix in presence and absence of fractures, and for imbibition and gravity drainage oil recovery with and without surfactant. The results indicate that atypical unfractured long core had a spontaneous imbibition oil recovery of zero, but produced 2% oil in a 5000 ppm anionic surfactant solution. The long fractured cores had a spontaneous imbibition oil recovery of 21% and a surfactant oil recovery of 38%. The oil recovery from gravity drainage of surfactant solution was substantial which indicates that surfactant can invade matrix block by gravity drainage. Gravity drainage plays a major role in recovering oil from fractured carbonates provided there is sufficient gravity head to replace oil (Alamdari *et al.*, 2011).

Sodiumcarbonate and anionic surfactant solutions were evaluated for enhancing oil recovery by spontaneous imbibition from oil-wet carbonate rocks originally and alteration to be more water wet. The degree of wettability alteration with alkaline surfactant systems observed here ranged from preferentially water-wet to intermediate-wet and was a function of the prior aging temperature in crude oil. Oil displacement can occur by buoyancy if an alkaline surfactant solution reduces IFT and/or alters wettability to more water-wet conditions. Oil recovery from oil-wet dolomite cores has been demonstrated by spontaneous imbibition with an alkaline anionic surfactant solution (Hirasaki *et al.*,

2003). Anionic surfactant have been identified to change calcite surfaces to intermediate/water-wet condition and better than cationic surfactant and also can develop low interfacial tension (Seethepalli *et al.*, 2004). Many experiments on NFR (Naturally Fractured Reservoir) carbonate rocks have been performance by several researcher to evaluate the recovery mechanism of oil from the matrix such as wettability alteration and gravity drainage.

Imbibition rate is proportional to imbibition capillary pressure, P_c , which is dependent on saturation. If a cylindrical core plug is immerse in wetting phase, as a common practice, the imbibition process will be far from linier. Water will invade the core by spontaneous imbibition from all directions. The manner in which the water saturation increases may range from frontal to global. Once an imbibition capillary pressure will then decrease globally as the wetting phase saturation increases (Ma *et al.*, 1999).

It is argued that capillary, wettability and interfacial tension retained high remaining oil in fractured reservoir during water flooding. This had been observed using calcite plate experiment that brine could not create spontaneous imbibition but when replaced by alkaline surfactant solution results in mobilization of oil by buoyancy or gravity drainage (Hirasaki *et al.*, 2004).

Modeling of wettability alteration using surfactant have been performed and also to take into account reducing interfacial tension, and mobility ratio to predict imbibition rate into matrix and the effect on oil recovery (Delshad *et al.*, 2006). Other chemical transport in NFR, and also enhanced oil recovery from fractured carbonate core have been evaluate by several reseacher (Abbasi-Ast *et al.*, 2010 and

Alamdari *et al.*, 2011).

III. METHODOLOGY

These experiments were proposed to used a methodology commonly used in the laboratory scale reserches which includes Surfactant-MES formulation, wettability of rock measurement after aging with the surfactant, and Spontaneous Imbibition Experiments. Furthermore the imbibition tests consist of Core Preparation, Imbibition Equipment, and Imbibition Processes. The details will be disussed in the following chapters.

A. Surfactant formulation

Surfactant used in this experiment is nonpetroleum based which is formulated from palm oil. Palm oil which normally contents *oleic*, *stearin* and other components is put into the estherification processes to produce methyl ester (biodiesel). subsequently, this ME is sulfonated using Natrium Bisulfit (NaHSO_3) to promote Methyl Ester Sulfonates, MES (Helianty *et al.*, 2011). This material has been categorized as surfactant due to have surfactant properties such as a capability to reduce interfacial tension between water and oil.

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 such as alkaline at low concentration which is also could reduce adsorption level, solution phase behavior and thermal stability characteristic.

B. Spontaneous Imbibition Experiments

These experiments were done to investigate the capability of the Surfactant-MES solutions to imbibe surfactant solutions into saturated oil cores and displacing the oil to coming out from the cores.

Table 1
Core plugs basic data

No Sample	L (cm)	D (cm)	Weight (gr)	B V (cc)	G V (cc)	PV. (cc)	G D (gr/cc)	Por. (%)	K (mD)
Core-1	7.488	3.764	169.597	82.327	63.847	18.480	2.656	22.447	2620.0
Core-2	7.515	3.763	168.817	81.186	63.526	17.660	2.657	21.753	2523.0
Core-3	7.483	3.763	166.881	81.739	62.859	18.880	2.655	23.098	2664.0

Volume of oil coming out from the cores could determine the effectiveness of surfactant solution to produce oil. Oil production then will be calculated as RF (recovery factor).

Spontaneous imbibition experiments consist of several steps for example: core preparation and basic core data measurement, fluid preparation (brine and surfactant solutions), and imbibition equipment.

- Core Preparation

Core preparation is basically standard procedures which Bentheimer cores were cut to become plugs with around 3.76cm diameter and 7.5cm long . Basic core data is presented in Table 1.

Then they saturated with brine and subsequently displaced by oil to set up Soi and Swc as the initial condition in the cores using core flooding rig. After that they are ready for imbibition tests. Table 2 is the saturated core data and proposed fluid for imbibition tests such as brine (control test) and 0.5% and 1.0% surfactant solutions.

- Imbibition Equipment

Imbibition equipment consist of a combination of two types of glasses i.e. a big glass in the bottom for immersing the cores and a long scaled pipette glass in the top to collect and measure the oil accumulation coming out from the core as a result of imbibition of water phase into the cores. Figure 2 shows the design of the equipment.

- Imbibition Processes

Firstly, the saturated cores at Soi and Swc condition were immersed in the imbibition equipment which initially content fully with fluid such as brine and surfactants up to the top of pipette. Then, observe the oil that coming out from the cores and write down the accumulation of oil in the top of pipette versus times. Normally imbibition processes is very quick for the first time step and followed by gradually slow production until the end of experiment.

C. Wettability Measurements

Sand stone rock and brine have been prepared for this experiment. Bentheimer standard core which consist of almost 100% quartz have been cut into several small pieces of 3cm thickness, see four cuts sand stone rock at Figure 3. Then, oil with 39.45°API and brine content of about 18,900 mg/L equivalent

NaCl concentration from oilfield are also used in these experiments. Table 3 shows the properties of rock and fluid.

Wettability of rocks were measured using DSA PD-E700 new generation pendant drop. This equipment basically measures contact angel of drop of liquid on surface of a pieces of rock. Figure 4

Table 2

No Sample	Fluid	Swc.(%)	Soi.(%)
Core-1	Water	20.18	79.82
Core-2	0.5% Surfactant	29.50	70.50
Core-3	1% Surfactant	28.23	71.77



Figure 2
Imbibition Processes and the Equipment

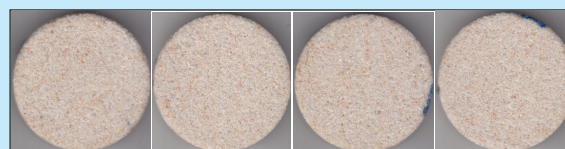


Figure 3
Four Pieces Bentheimer Cores with the same size

Table 3
Rock and Fluid Properties Used Wettability Alteration Experiments

Fluid	Core	Density gr/cc	Viscosity cp	IFT dyne/cm	Phase Behavior
Brine	Bentheimer	0.99562	0.76		
Oil	Bentheimer	0.82775	2.31		
Surfactant MES-0.5%	Bentheimer	1.00718	0.59	2.85E-03	Winsor type-1
Surfactant MES-1.0%	Bentheimer	1.00949	0.71	4.86E-03	Winsor type-1

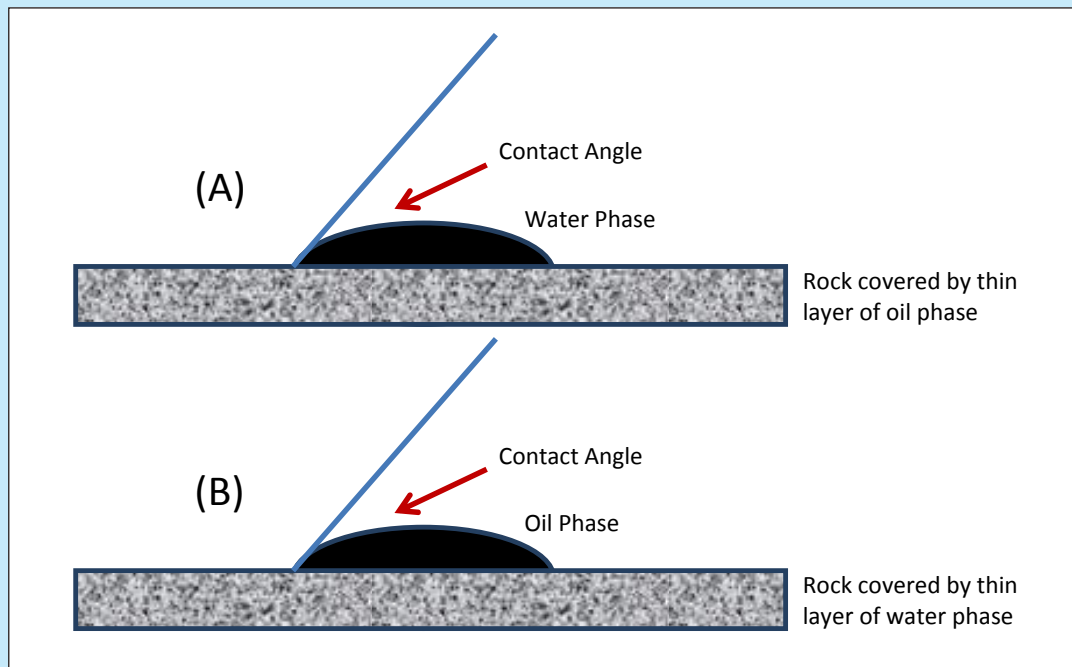


Figure 4
Contact Angle Measurement

Table 4
Recovery Factor of Spontaneous Imbibition Test

No Sample	Fluid	RF. (%)
Core-1	Water	41.69
Core-2	0.5% Surfactant	67.07
Core-3	1% Surfactant	92.25

Table 5
Results of Wettability Measurement

Ageing Fluid	Droplet	Contact Angle	Wettability
Brine	Oil	0	Mixed wet
Oil	Brine	27.73	Mixed wet
0.5% Surfactant	Oil	0	Mixed wet
1.0% Surfactant	Oil	0	Mixed wet

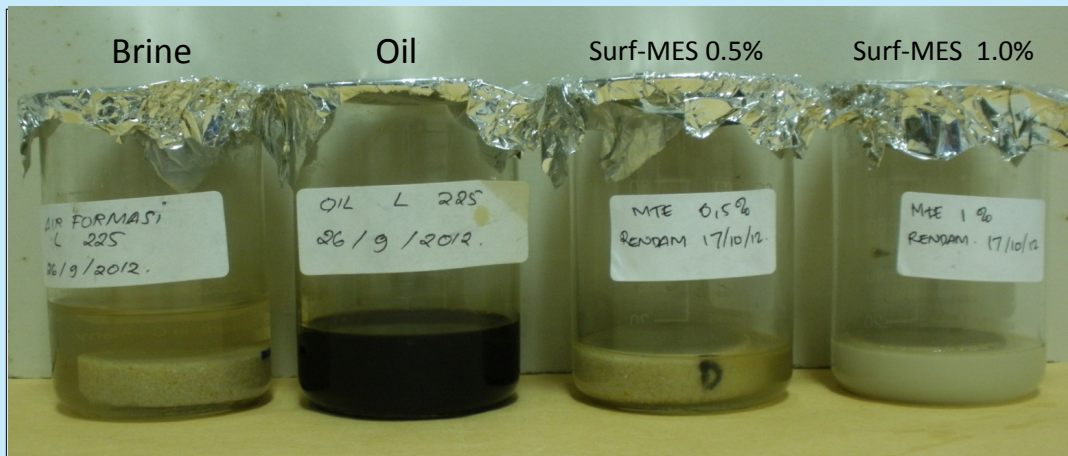


Figure 5
Ageing Cores for Several Days

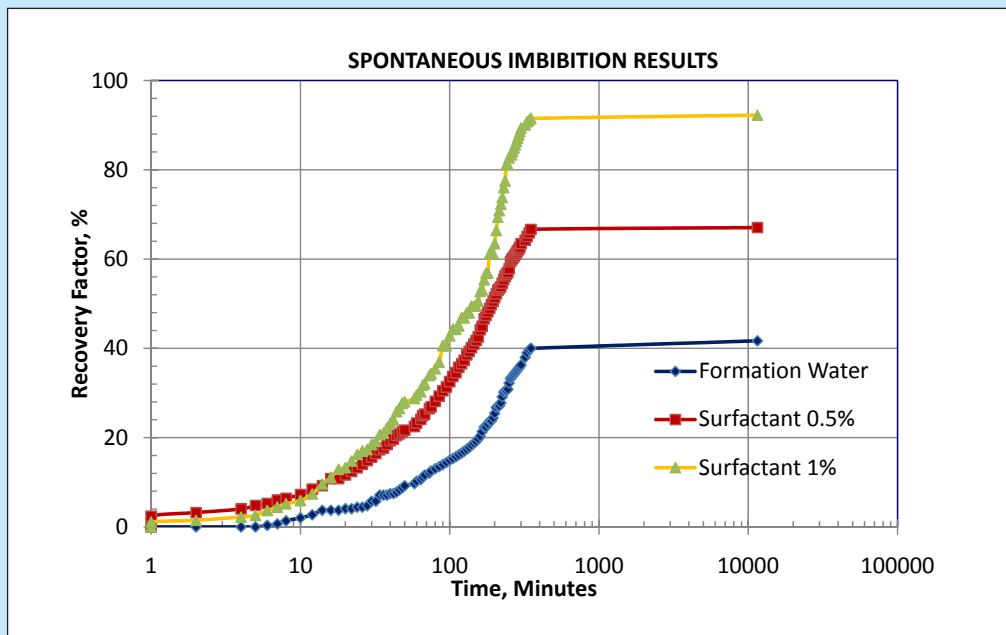


Figure 6
Recovery Factor of Spontaneous Imbibition vs. Time

illustrates contact angle measurement. The droplet can be water phase or oil phase. If the droplet is water phase then contact angle above 90° will be more oil wet and below 90° is more water wet (A). In the contrary, if the droplet oil phase then contact angle above 90° will be more water wet and below

90° is more oil preferent (B).

Four pieces of rocks each is aged using the fluid such as brine, oil, Surfactant-MES 0.5%, and 1.0%, see Figure 5. Leave the aging time up to 40 days to evaluate wettability alteration. After that all the cores were subjected to measure the wettability and

evaluation should be done if there were any changes in wettability characteristics.

IV. RESULTS AND DISCUSSIONS

A. Formulated Surfactant-MES

Surfactant-MES formulations has been done in the lab and having properties very low interfacial tension i.e. 10^{-3} dyne/cm and Winsor type-I of phase behavior. In addition this surfactant has also very good properties that fulfill the criteria for enhanced oil recovery proposal. This formulated surfactant MES has been used for further experiment such as wettability alteration and imbibition test as part of tests for oil recovery enhancement screening.

Surfactant-MES produced from palm oil will be very potential for substitusing surfactant-crude oil based normally used in oil industries, and available abundance in Indonesia. The price of Surfactant-MES is a very cheap compare to surfactant-crude oil based.

B. Imbibition Results

Table 4 shows the spontaneous imbibition results and graphically can be seen in Figure 6. The results indicate that surfactant solutions could improve the recovery factor of oil by imbibition processes compare to the control, immersing in brine. Optimum oil recovery factor by brine imbibition is only 41.69%. On the other hand imbibition of surfactant solutions could produce oil recovery as much as 67.07% with 0.5% surfactant and 92.25% for 1.0% surfactant solutions.

Visual observation indicates that the imbibition processes were created by emulsification at low IFT rather than wettability alteration mechanism. It has been stated in the introduction that spontaneous imbibition processes could be created by wettability changes and low interfacial tension in which emulsification may happen. This results indicated that Surfactant-MES has a capability to create spontaneous imbibition processes which is normally happen in the oil production mechanism during flooding on EOR projects.

C. Wettability Changes

The result is exhibited in Table 5 indicating that all experiments did not change wettability of rocks. The rocks are still having wettability characteristics similar to the control test (brine). The rocks have

wetting tendency with all of fluids such brine, oil and surfactant. This phenomenon can be classified as mixed wet.

As the targets of these experiments are firstly not only to get spontaneous imbibition working properly but secondly also to create wettability changes to more water wet. Therefore this Surfactant-MES need to further improving to fullfill one of the targets. To get more appropriate surfactant for wettability alteration, therefore, HLB (Hydrophilic-Lipophilic Balance) could be a little bit improved. Hopefully, if the wettability alteration agent can be create from Surfactant-MES, the imbibition processes will work intensively.

V. CONCLUSION AND RECOMMENDATION

1. A formulated surfactant-MES can be developed to match the need of low interfacial tension surfactant for enhanced oil recovery at mature fields.
2. Imbibition experiments indicated that this formulated Surfactant-MES has a capability to imbibe water into the cores and displacing oil
3. However, this formulated surfactant-MES has not a capability yet to alter the wettability characteristic of quartz rocks to become more water wet from originally mixed wet.
4. The imbibition processes is not caused by wettability alteration but rather caused by emulsification created by low interfacial tension phenomena.
4. Further formulation should be improved to find surfactant mixture compositions that has a capability to change the rock wettability to be more water wet.

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