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Research Article

# IONIC LIQUID AND EUTECTIC-BASED IONIC LIQUID AS NOVEL ADDITIVES FOR FOAM STABILIZATION IN POROUS MEDIA HYDROCARBON-RICH ENVIRONMENT

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## ABSTRACT

*Further maintain of foam stability in foam flooding process is a major challenge for oil industry. In this work, capability of the common ionic liquid (IL) and newly developed eutectic-based IL or widely known as Deep Eutectic solvent (DES) were evaluated to assess their use as co-surfactant in stabilizing foam in the presence of oil. The novelty of the new chemicals in EOR application is in its ability to improve the surfactant performance in foam stability while being cheap, biodegradable and easy to produce for bulk application. The optimum amount of eutectic- and imidazolium-based ILs used as additives to a fixed concentration of an in-house-surfactant, MFOMAX (M) in the presence of oil was determined. The physicochemical properties measurement of the mixtures and foam stability test in a bulk column test were conducted. Core flood experiments were conducted to estimate gas breakthrough, mobility reduction factor (MRF) and incremental oil recovery. The addition of ILs in surfactant solution were found to enhance foam stability. Furthermore, it was found that addition of additives increased the interfacial tension of M/IL solution against crude oil which improved foam stability. However, foam stability increased with decreased surface tension of M/IL solution against N2 gas. The results in core flooding experiments exhibit the advantages of ILs at their optimum formulation in delaying the gas breakthrough time and increment in MRF value. The additional oil recovery was slightly higher with the addition of additives in surfactant solution. The recommended optimum surfactant/IL mass ratio to obtain the highest bulk foam stability of imidazolium-based ILs and eutectic-based ILs is at 90:10 and 80:20, respectively. The common IL requires lower concentration as compared to eutectic-based IL in order to perform well which is encouraging as common ILs are normally more expensive.*

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## INTRODUCTION

Foam flooding is an enhanced oil recovery (EOR) technique to overcome gas channelling and gravity overriding issues leading to low recovery, hence the sweep efficiency can be improved (1, 2). The foam effectiveness is evaluated in its MRF (3). Foam displacement has convinced to be a possible solution to those mentioned issues, enhancing the efficiency of oil production (4, 5). The main focuses in foam displacement for EOR are foamability and foam stability under reservoir conditions (e.g., salinity, temperature, pressure).

As the sensitivity of surfactant towards harsh reservoirs conditions are more pronounced, hence several studies on compatible chemicals for applications were encouraged (6). Recently, several studies have started to explore the utilization of ILs as a substitute to organic surfactants in EOR applications. ILs have received attention and have been evaluated on a wider scale because of their special chemical and physical properties. Density, melting point, viscosity, surface tension, thermal stability and vapor pressure are some of their physicochemical properties which reliant on the combination of cations and anions (7, 8). More recently, a new kind of ILs have been developed and the concern is more on their properties. DESs are considered as a new class of ILs. The availability, ease of synthesis and biodegradability of the components make the DESs cheaper and versatile alternative to ILs (9).

In this research, the bulk foam stability experiment using Foamscan instrument was carried out to determine the best surfactant/IL formulation prior to core flood experiments under reservoir conditions. The interfacial and surface tensions of different ILs content in surfactant solution were investigated. The focus of this research is to determine the capability of two groups of ILs, common ILs and eutectic-based ILs, as additive in enhancing surfactant performance on foam stabilization. This study is also important to discover and compare the potential of these chemicals to control gas mobility and enhance oil recovery. Previous work by Hanamertani et al. (2018) was only limited to gas mobility control without oil to independently understand the mechanism of chemical stabilization and propagation of foam without further investigation on oil recovery (10). Therefore the ability of ILs-foam in improving oil recovery in this work is the ultimate objective in order to determine whether first, ILs is capable to overcome the well-known limitation of foam; destruction of foam as a result of contact with oil, and secondly to establish whether a stable foam is the main factor to improve oil recovery.

## LITERATURE REVIEW

### FOAM IN POROUS MEDIA

Among the primary challenges of injecting foam into porous media are (1) foamability and (2) foam stability. Foamability is described as surfactant's foam-generating power. As for foam stability, it is described as the difference in foam volume or height against time, after creation of foam (11). However, works of literature have reported that the focus of a foam displacement process relies mainly on the stability of the lamellae (2, 12) thus the selection of surfactants as a foaming agent is important (13). Other researchers have raised out concerns about foam instability when dealing with oil, and due to this complex foam/oil interaction, studies on foam stability are more crucial in the porous media environment (1, 2, 4, 14, 15). Hanamertani et al. (2018) examined the effect of ILs in influencing the surface properties of foam particularly in the lamella. The interaction between the surfactant synthesized with these chemicals proven beneficial in strengthening the lamella hence enhancing the surface properties of foam thus its stability in the absence of oil. A pseudo-emulsion film is the thin liquid film between the gas phase and oil droplet. When this film formed between the gas phase and oil droplet is stable, oil spreading is suppressed, and it will stay in the lamella. Oil disperse or bridge liquid/gas interface when the pseudo-emulsion film ruptures, and break the foam (2, 16).

## IONIC LIQUIDS AS ADDITIVES

Eutectic- and imidazolium-based ILs could increase the foam half-life in bulk foam test at ambient and high temperature conditions in the absence of oil (17). Despite the capability of ionic liquid-based additives (i.e. ILs and DESs) to stabilize foams through their ability to strengthen the surface properties of the foam formed, the potential applications of ILs and eutectic-based ILs for improving the stability of foams have not been fully investigated. Moreover, in other studies, MRF values calculated from core flood experiments were only examined in absent of oil (10). The MRF can determine the capability of foam in gas mobility control in porous media. Therefore, this research is essential to figure out the performance of ILs on bulk stability of surfactant-foam with oil and the effect on oil recovery.

Foams are less stable with the presence of oil hence additives could minimize the destabilizing effect of oil (16, 18). ILs have been classified as organic salts that are completely ionic in nature and usually comprised of both anionic and cationic species which have a melting point less than 100°C. ILs are normally categorized according to their cationic part like imidazolium, ammonium, pyridinium and phosphonium (19). A DES is a fluid comprised of two or more mixtures of safe and cheap components that are able to associate with each other, often via hydrogen bond interactions, to form a eutectic mixture with a melting point lower than that of each individual component. As DESs are considered as electrolytes, they have high possibility to create electrostatic interactions or cationic-anionic interactions with surfactant resulting to the reduction in surfactant head group repulsion at the interface (20). The existence of salts or electrolytes is supposed to affect the surfactant foam formation and stability by suppressing the bubble coalescence through its effect on enhancing disjoining pressure in the liquid films and influencing the electrostatic stabilization at the interface (21). Electrolytes induce the cationic-anionic type interaction with anionic surfactants which leads to the reduction of surfactant head group repulsion at the interface (22). Several studies pointed out the fact that the addition of certain amount and type of salts at optimum surfactant concentration will give the positive effect on generation of foam, bubble coalescence and stability of the foam lamellae (8, 23-26).

# MATERIALS AND METHODS

## MATERIALS

MFOMAX (M), an in-house-developed surfactant, with an active content of 20%, provided by PETRONAS Research Sdn. Bhd. (PRSB), was utilized as the foaming agent. It is comprised of amphoteric and anionic surfactants. In order to evaluate the application of IL as additives and to predict their optimum formulation for foam stability enhancement, two types of ILs with three surfactants: IL formulations were investigated. 1-butyl-3-methylimidazolium trifluoromethane sulfonate indicated by IL4 represents as common IL whereas Choline Chloride - glycerol (1:2) indicated by IL11 represents as DES.

The mixtures were prepared in the presence of 3.3 wt.% salinity of synthetic brine solution. A fixed surfactant concentration which is 0.5 wt.% was utilized in this investigation as base case solution, while the concentrations of the additives were varied. The selected formulations ranged from 90:10 to 60:40 of surfactant/IL mass ratio and the light Malaysian crude oil 39.4° API was from Baronia field, Malaysia as provided by PRSB. $N_2$ gas was employed for foam in all the experiments. In the core flooding experiments, Berea sandstones cores (diameter 1.5 inches and length 6 inches) were used.

## METHODS

### *Bulk foam experiment*

The bulk foam stability experiments were performed using the Foamscan instrument (27). In this study, two factors were considered which are foaming time and foam half-life. This is to evaluate the capability of the mixture solutions in generating foam and maintaining its stability. The amount of mixture solutions (60 +/- 1 ml), and 10% of oil (6 ml) were mixed by a fixed $N_2$ gas flow rate (50 ml/min) till reaching the pre-set foam volume (150 ml) which represents foamability. These variables were kept constant for all the tests at 90°C and 3000 mBar (43.5 psi). The bulk foam stability experiments were performed in both conditions, with and without the additives. M was formulated at three different concentrations of ILs to assess the optimum amount of ILs as additive for use in the core flooding experiments after conducting this bulk foam stability test as the screening tool.

### *Surface and interfacial tension measurements*

Prior to core flooding experiment, physicochemical properties of the selected formulation of surfactant/additive were determined on the IFT700 equipment manufactured by Vinci Technologies to measure the surface and interfacial tensions for optimum formulation. Both experiments were operated at 90°C and 1800 psi to represent reservoir temperature and pressure. The results obtained under this condition were subsequently used to evaluate the core flooding experimental results.

### *Core flooding experiment*

In this study, HTHP Core Flooding System from Sanchez Technologies (France) was used to represent field application and to determine the consistency of the foam stability between bulk and porous media test. Three runs had been performed in this research where the base case experiment was only on M (the solution without additive). This experiment is necessary to investigate the additives capability to influence foam stability, the breakthrough time and oil recovery. This experiment was performed at 90$^{o}$C with a backpressure of 1800 psi at the end of the holder to simulate the formation pressure.

Initially, the pore volume (PV) and absolute brine permeability of each core were measured. Next, in the first stage, the brine saturated cores were flooded by Baronia oil to find initial water saturation (Swi). After the drainage process, the oil-saturated core was aging. Then, the core was injected with brine until reach almost 100% water cut to indicate residual oil saturation after water flooding (Sor). Next, the first $N_2$ gas was injected. From the total amount of oil produced in the effluent during the water and gas flood, the oil recovery was then calculated. The chemical was injected, then followed by the final step which was the second gas injection to represent the foam flooding. Several PV of injected fluids were injected to ensure residual oil saturation (Sor) is achieved and each step was ceased after reach a steady state where the pressure drop value remained stable. During gas injection, the cumulative volume of the gas using gasometer were measured to determine the gas breakthrough time. The pressure drop obtained from the first and second gas injection were used to measure MRF. A higher MRF represents a stronger foam that can stabilize the gas front and delay the gas breakthrough (28, 29). For the oil recovery after foam flooding, it was classified as additional oil recovery (AOR). The constant flow rate at 0.2 cc/min. was applied for all injections.

# RESULTS AND FINDINGS

## BULK FOAM EXPERIMENT

### *Effect of additive concentrations*

The effect of different concentrations of ILs as additives to M on foam stability test can be analysed using foam half-life indication plot as presented in Fig. 1. The dashed line representing half-life of M solution without the presence of additive. The graph shows that the addition of additives generally was able to enhance surfactant's performance in stabilizing the foam except for M/IL4 mixture solution at 60:40 mass ratio. It can be indicated that this additional amount of IL cannot resist the force to counteract film rupture thus reduce the performance of M. The capability of the surface to modify and reinstate itself relies on the properties of the mixture solutions and their concentrations which could be the reason why the different observation can be seen for each sample. The 60:40 formulation was observed to be the poorest formulation for both type of additives. In M/IL4, the relationship between the IL concentration used and foam half-life is inversely proportional. The half-life of foam gradually decreases with increasing IL contents from 90:10 to 60:40. At 90:10 ratio, the highest half-life was achieved, which is 66.92 minutes. It was the suggested optimum IL contents to obtain highest foam stability for M/IL4 (imidazolium-based IL) which probably due to sufficient electrostatic attraction among oppositely charged head groups and hydrophobic interactions among surfactant-IL tail groups (17, 30). This interaction engages the cationic part of IL4, creating synergetic effect with M surfactant at the interface to retain thin film stability (10).

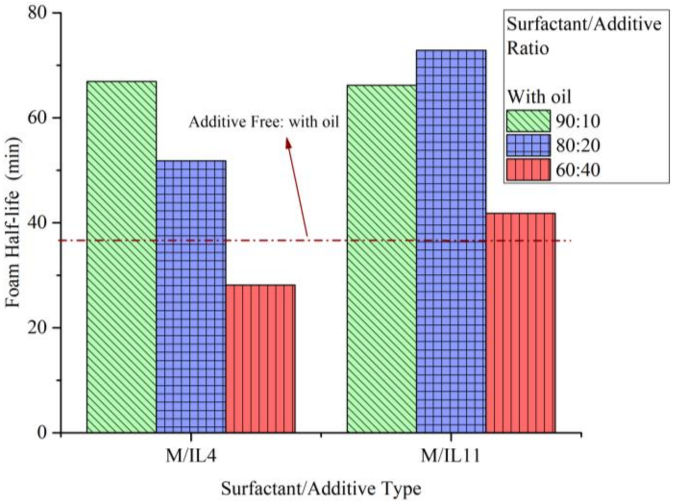

**Fig. 1:** Foam half-life for different type of additives at different ratios with Baronia oil.

For M/IL11 (eutectic-based IL), the foam stability appears to increase with increasing IL contents in the mixture solution (from 90:10 to 80:20) and decreases thereafter with increasing IL contents (60:40). The stabilized lamellae at a slightly higher concentration could be associated with the eutectic-based IL's potential to induce surfactant molecules redistribution at the interface by creating additional and strong cohesive bonding through hydrogen bonding network (10). Therefore, the delay for the foam film to rupture might be caused by the effect of this intermolecular hydrogen bonding which happened between neighbouring molecule, surfactant and eutectic-based IL in this case, as this attractive interaction will create tighter packing of the adsorbed molecules and hinder the mobility of the surfactant while providing an additional foam film elasticity to counteract film rupture (17, 31, 32). From the graph, the optimum surfactant/IL mass ratio to obtain highest bulk foam stability of M/IL4 and M/IL11 is at 90:10 and 80:20, respectively.

## PHYSICOCHEMICAL PROPERTIES MEASUREMENT

### *Interfacial tension*

The variation of foam half-life and foaming time for all the mixture solutions with IFT relationship is shown in Fig. 2 for correlation. It was found that in the absence of ILs, with lower IFT value, M 100:0 was able to achieve the shortest foaming time of 1.78 minutes only as compared to solutions containing ILs. By having a lower interfacial tension could increase the interfacial area with a lower energy. This will then influence the foamability by reducing the foaming time, whereas shorter the foam half-life. In the presence of oil where foam-oil interaction occurs, the addition of these type of ILs reduce the capability of surfactant in generating foam. With their higher IFT values than M alone, this might be the reason why the coalescence of the dispersed droplets will be slower hence longer the foaming time and half-life. The oil has the tendency to encroach into the lamella and be adsorbed at the gas/liquid interface and displace foam-promoting surfactant thus inhibit foam. On the other hand, it has been claimed that the presence of additive in oil can also act as an emulsifying agent. This emulsifying agent in the oil might create a very stable emulsion. Hence, by having ILs and surfactant molecules at the interface could act as electrostatic or steric barriers against droplet coalescence thereby increasing the emulsion stability (33). The presence of stable emulsion could stay in the lamellae and will create stable pseudo-emulsion film.

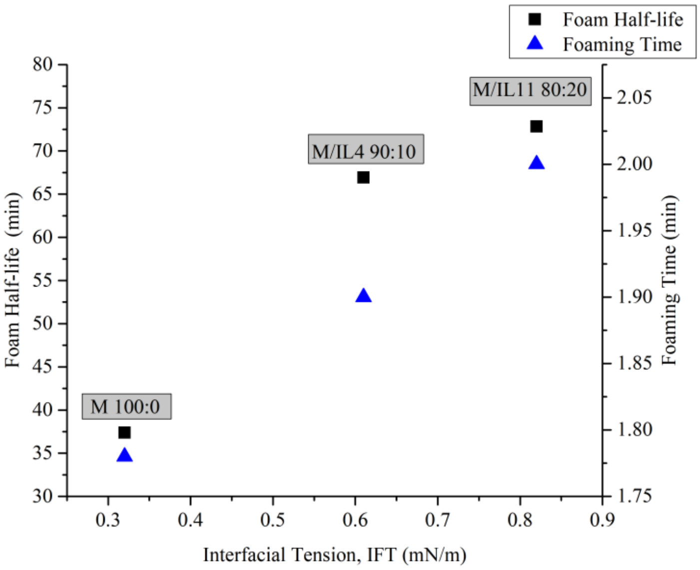

**Fig. 2:** Foam half-life and foaming time vs IFT measurement

In this research, the addition of ILs in surfactant solution could enhance foam stability even though there is a slight delay in generating foam. This suggests that the solution that took longer time to generate foam will create a more stable foam. Based on the experiments we conducted, it can be summarized that the stability of foam increased with increasing IFT values. With the increment in IFT values, the stability of foam improved by combining ILs with M solution. The highest IFT value was observed for M/IL11 80:20 mixture solution corresponds to the longest half-life. This revealed the capability of eutectic-based IL to enhance foam stability by having the highest IFT value among other solutions. Some researchers reported the same findings by predicting that the presence of higher IFT values among the mixture solutions gave significant stabilization of the foam films (27, 34, 35). Our results also indicate that the generated foam is destabilized by the oil phase as addressed by other researchers (14). It was claimed that foam stability influences by the oil phase composition where the presence of light components is detrimental to foam stability.

### *Surface tension*

Fig. 3 presents the relationship between foam half-life and foaming time versus ST measurement. In the other study (17), it was reported that ILs could reduce the surface tension. On top of that, eutectic-based IL at optimum ratio gave a slightly higher reduction of ST as compared to common IL. The results presented that the presence of additives could lower the ST between mixture solution and $N_2$ gas. It is expected that the presence of IL in binary system might alter the ST of individual surfactant solution as suggested by others (36). From other point of view, there is a relationship between foamability and ST. Although M/IL mixture solutions produced lower ST than M however they took longer foaming time representing a lower foamability as more energy is required to generate new surface. Nevertheless, the presence of ILs shows a prominent effect on foam half-life. With this reduction, the foam half-life was found higher than in the solution of M alone. This observation of good foam stability usually infers poor foamability has also been claimed by other studies (37, 38). The results suggest that when ILs could reduce the ST, it apparently reflects the increment of surfactant molecules per unit area at solution/gas interface.

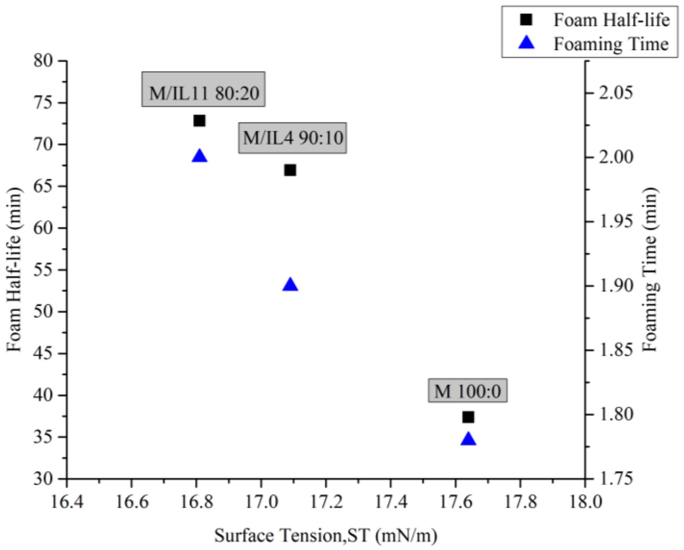

**Fig. 3.** Foam half-life and foaming time vs ST measurement

## CORE FLOODING EXPERIMENT

### *Breakthrough time*

Considering the optimum formulation for each IL, further test had been conducted to examine their performance in porous media. The prediction for gas breakthrough time could be provided by measurement of the cumulative volume of the gas produced in the effluent of different solutions as demonstrated in Fig. 4(a), and Fig. 4(b) illustrated the recovered oil from core M 100:0 (IL-free case) using core 1 after gas breakthrough.

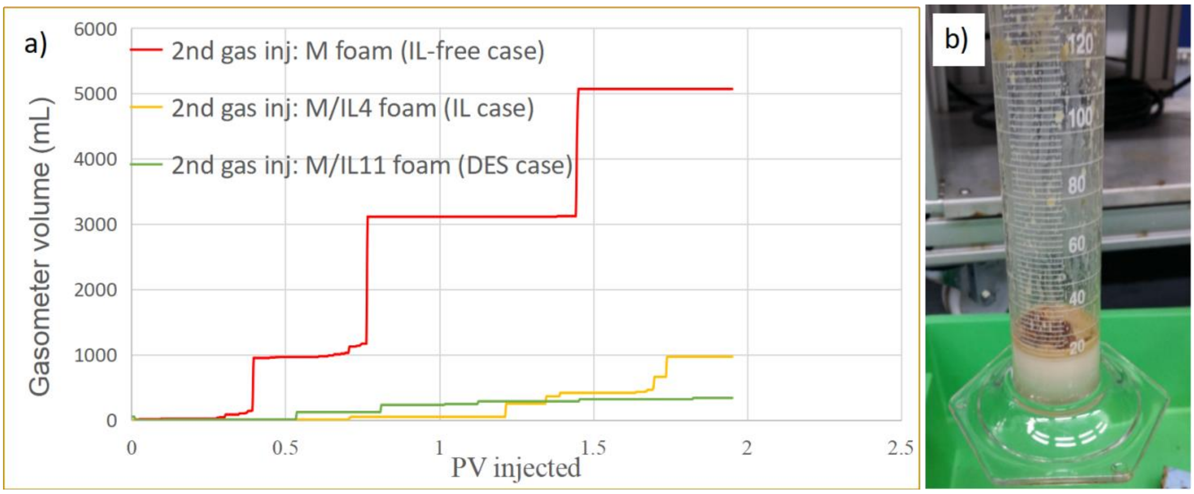

**Fig. 4.** **(a)** Gas breakthrough graph; **(b)** Picture of the effluent of core 1 (IL-free case) after gas breakthrough

Our results showed that the significant delayed in gas breakthrough occurred when ILs were present as co-surfactant. This observation has proved that ILs improved the surfactant performance during foam mobility control application based on differential pressure and MRF values as reported in the other work (10). For the first run, M 100:0 (IL-free case) using core 1, an estimated early gas breakthrough can be detected at 70 minutes after about 0.35PV injected. M/IL4 90:10 was able to hold the gas longer and the channelling of the gas was observed after 1.21PV injected (242 minutes after gas injection). A potentially equivalent capability of M/IL11 80:20 to delay the gas breakthrough can also be noted from the graph, however the performance is less than M/IL4 90:10. The gas breakthrough occurred after 0.70PV injected (140 minutes after gas injection). The time the gas breakthrough was observed indicates the initial stage of gas release due to the ruptures or collapse of foam formed. For all cases, at certain times and PV injected, a rapid increase of gas breakthrough was observed, followed by a continuous increment of gas volume produced in the effluent. Nevertheless, although the gas breakthrough of M/IL11 occurred before M/IL4, the increment of gas volume produced showed a significant reduction over PV injected as compared to M/IL4. Production of stable foam as observed with the addition of recommended additives would be expected to enhance oil recovery as compared to using M alone.

### *MRF and Oil Recovery*

The evaluation on the chemicals performance in affecting oil recovery has been done and could be highlighted as the new outcomes of this research. The effect of IL types at their optimum formulation on the oil recovery efficiency were determined after performing foam flooding process. From the results, the AOR increased with the presence of additives. This corresponded to the increment of oil saturation reduction. After the chemical and second gas injection, the oil saturation reduction during test 1, 2 and 3 were about 5.36, 6.11 and 6.02%, respectively, giving the residual oil saturation after foam flooding to be 23.94, 23.20 and 25.80%, respectively. The highest reduction of oil saturation during run 2 (IL-case) could be due to generation of progressively stronger foam that will slower the gas mobility leading to enhancement of sweep efficiency. This is convincing since the addition of IL4 in the surfactant solution could delay the gas breakthrough longer than the others as illustrated in Fig. 4(a), and it is the one that reduced the oil saturation the most, as supported by other researchers (39). This prediction can be made from the cumulative amount of gas collected in the effluent as presented in Fig. 4(a). Furthermore, as can be seen, the addition of small amount of common IL which is 556ppm of IL4 in the M solution gave AOR up to 10.62% after foam flooding. Meanwhile, the higher amount of eutectic-based IL is necessary to be comparable with the expensive imidazolium-based IL which is about 1250ppm of IL11, the optimum amount determined in bulk experiment earlier, leading to increment of oil recovery up to 9.72% of original oil in place (OOIP). This could be attributed to the effects of oil transporting properties of the foam and formation of stable foam in the core by having some surface interactions such as the hydrophobic interactions, hydrogen bonding formation and electrostatic stabilization as discussed earlier in section 4.1.1 and can improve oil recovery efficiency. As for M individually, the increment of oil recovery was up to 8.62% of OOIP after foam flooding.

From the results obtained, it showed that the oil recovery may be controlled by sustainability of the good mobility ratio rather than the high value of reduction achieved as illustrated in Fig. 5. The graph showed that during beginning of $N_2$ injection, the increment in MRF can be seen with the addition of ILs in surfactant solution as compared to the MRF for the base case (M). The presence of optimum IL11 contents in M solution gave the highest MRF value of about 13.65 at ~0.36 PV injected which suggest that the mobility of $N_2$ was lowered upon its contact with formulated surfactant solution, whereas the maximum MRF reached for M/IL4 was about 11.90 after ~0.47 PV injected. Although the maximum MRF for M/IL4 was lower than that of M/IL11, the early increment in MRF and its sustainability has led to comparable effect in improved oil recovery with M/IL11.

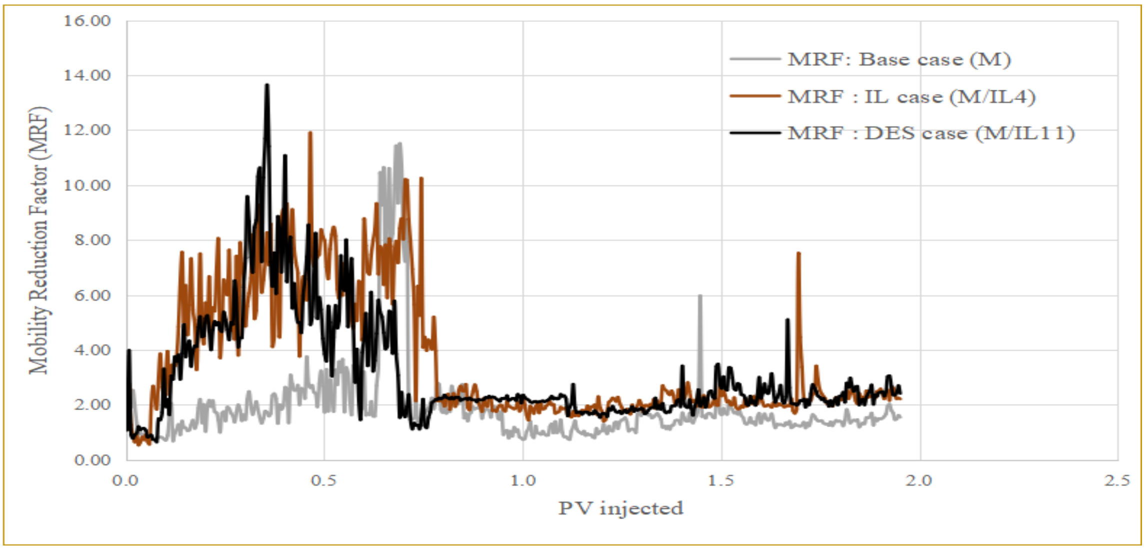

**Fig. 5.** Comparison of MRF profiles during foam generation using M (Base case), M/IL4 (IL case) and M/IL11 (DES case)

# CONCLUSION

The influence of imidazolium- and eutectic-based ILs additives on the bulk stability of M foam in presence of oil and enhanced oil recovery in core flood has been evaluated in this work. It was also developed to compare and relate the bulk foam experimental findings to that of the porous media. Through this investigation, it was shown that optimum IL contents is necessary to obtain higher foam stability but depending on surfactant/additive type and that the presence of oil had the greatest influence on foam stability. The suggested optimum surfactant/IL mass ratio to obtain highest bulk foam stability of M/IL4 and M/IL11 is at 90:10 and 80:20, respectively. The common IL requires lower concentration as compared to eutectic-based IL in order to perform well in all conditions (foamability, foam stability and oil recovery) which is encouraging as ILs are normally more expensive. The results on foam stability according to the foam half-life (bulk foam test) and additional oil recovery (core flooding experiment) can be ranked as the following:

Bulk foam test: M/IL11 80:20 > M/IL4 90:10 > M 100:0

Core flooding: M/IL4 90:10 > M/IL11 80:20 > M 100:0

M /IL4 90:10 was found to be more effective in the core flood experiment which was able to delay the gas breakthrough longer than others thus supporting improved oil recovery. However, the capability of eutectic-based IL as a new cost-effective IL was comparable to that common IL as co-surfactant. It should also be noted that the slight draw back in DES effectiveness could be compensated by the fact that DES is biodegradable, cheap and can be synthesized from mainly naturally existing compounds. In conclusion, further core flooding experiments to represent field application could be carried out to explore new cost-effective ILs as additive to surfactant which are more compatible with oil types and reservoir conditions using same surfactant/IL formulation that is beneficial to a greater understanding on the effect of oil recovery enhancement.

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