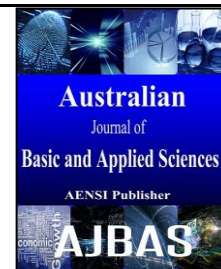




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Jatropha Based Microemulsion Efficiency Screening Study for Enhanced Oil Recovery

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ABSTRACT

Background: There is no secret that ultralow interfacial tension between formulated microemulsion and crude oil lead to high crude oil recovered from the reservoir. However, development of non edible triglyceride based microemulsion for enhanced oil recovery application is still considered scarce. **Objective:** This paper presents the determination of an aqueous phase composition of Jatropha based microemulsion in which the triglycerides constitute the whole oil-phase of the microemulsion. **Conclusion:** The measured interfacial tension between the optimum microemulsion and oil model, which is n-octane was 0.0015 mN/m. The corresponding maximum cumulative tertiary oil recovery is 78%, which was achieved after the injection of the optimum microemulsion formulation to a sand pack. The significant amount of oil recovered during cumulative tertiary oil recovery(78%) suggests the effectiveness of the optimum microemulsion formulation in enhanced oil recovery.

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INTRODUCTION

Based on conventional process, it is believed that about 30% of the original oil in place (OOIP) in the reservoir is recoverable. Apparently advanced methods are needed to attend the world demands since reserved oil through conventional methods start to decline. Enhanced oil recovery (EOR) method is one of the means to recover the remaining 70% of the oil originally in place (OOIP). Microemulsion flooding has been developed with the objective to maximize oil production thru tertiary recovery. Microemulsions are transparent, thermodynamically stable and isotropic liquid mixtures of oil, water and surfactant. Due to unique feature of microemulsion such as high stability, ultralow interfacial tension (IFT), large interfacial area and spontaneous formation, it has proven as efficient displacing fluid for EOR applications (Z Jeirani *et al.*, 2013b)

Microemulsion flooding reduces interfacial tensions between oil and water, thus increasing the displacement efficiency (Austad *et al.*, 1997). About additional 20% oil recovery was observed when the IFT of the formulated microemulsion is maintained

below 10-3 dyne cm⁻¹ (Levitt & Pope, 2008). However, works of Putz *et al.*, (1981) show opposite outcome. Oil recovery was surprisingly less than expected even at low interfacial tension. This is due to severe surfactant retention as slug deterioration occurs (Healy *et al.*, 1975). Surfactant retention issue was normally observed for anionic surfactants. It was shown that the addition of polyethylene glycol to microemulsion reduces the adsorption of anionic surfactants significantly (Osterloh & Jante Jr, 1992).

The efficiency of oil recovery depends on the composition of microemulsion. Few factors strongly influence the performance of microemulsion performance in oil recovery. Such factors include microemulsion water content, surfactant, co-surfactants, and co-oil. Microemulsion flooding with higher water content seems to be more effective at oil-bank formation (Osterloh & Jante Jr, 1992). Therefore oil-in-water microemulsion was favored as pre-prepared microemulsion in EOR application. Oil-in-water microemulsion interacts with reservoir oil to form middle phase microemulsion. Numerous researches show that middle phase microemulsion (Osterloh & Jante Jr, 1992) gives rise to the lowest IFT between crude oil and brine water.

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Thus, enhancing the efficiency of the microemulsion. However, work on oil-in-water microemulsion application in EOR application is scarce and limited. Prepared oil-in-water microemulsion are expected to form Winsor Type III microemulsion in-situ after hydrocarbon solubility (Jeirani *et al.*, 2014). Winsor type III microemulsion indicates lowest interfacial tension between formulated microemulsion and hydrocarbon. Ultralow IFT will lead to higher oil recovery. Santanna *et al.* (2009) and Zahra Jeirani *et al.* (2013) shows the significant effect of microemulsion viscosity on oil recovery. It was shown that higher viscosity of microemulsion results in higher oil recovery.

Triglycerides based oil in water microemulsion tends to show high potential in oil recovery due to its ultralow interfacial tension. Various sources of triglyceride oil based microemulsion have been studied such as Palm oil (Z Jeirani *et al.*, 2013a), Neemoil (Singla & Patanjali, 2013) and Pine oil (Santanna *et al.*, 2009). These oils have shown significant result in promoting type III microemulsion formation as well as high potential in EOR application. Numerous researchers have also studied the effect of oil polarity, oil chain length (Szekeres *et al.*, 2006; Warisnoicharoen *et al.*, 2000), co-oil (Szekeres *et al.*, 2006) type and concentration of surfactant and co-surfactant (Bera *et al.*, 2011; Hellweg, 2002) towards microemulsion phase inversion. Warisnoicharoen *et al.* (Warisnoicharoen *et al.*, 2000) found that C18 and longer carbon chain alkyl chain surfactant is able to solubilize large molecular volume oils. Thus, it increases the possibility of having long chain triglyceride oil to be soluble in water to form microemulsion.

The main objective of this paper is to formulate efficient non-edible triglyceride oil based microemulsion to improve oil production in a tertiary recovery process. Alkyl polyglycosides were used as the surfactant of the triglyceride microemulsion. The interfacial tension of surfactant-brine-alcohol-oil system is used as the main criteria in interpreting the performance of chemically EOR by microemulsion flooding. It is well known that IFT are established relationship in evaluating microemulsion performance in tertiary oil recovery. Therefore, measurement between formulated formulated microemulsions and model oil were conducted and cumulative tertiary oil recoveries were measured as screening criteria.

Materials, experimental, setup and procedures:

Materials:

Glucopon 650EC (APG) having an average alkyl chain length of 11, hydrophilic-lipophilic balance (HLB) of 11.9, and critical micelle concentration (CMC) of 0.073 g/L at 37 °C was used as a surfactant in this study (Jurado *et al.*, 2007). It was purchased from Cognis Malaysia Sdn. Bhd. Normal butyl alcohol (NBA), was used as co-surfactants for

screening purpose. These chemicals were supplied by BCI Sdn. Bhd. Sodium chloride, (NaCl) was purchased from LGC Scientific, Malaysia. Meanwhile, n-octane with purity of 99% was purchased from Sigma-Aldrich. Crude Jatropha oil was purchased from Bionas Malaysia Sdn. Bhd. All of the materials were used as delivered.

Microemulsion preparation:

Jatropha based microemulsion was prepared by mixing 230g of oil phase with 230 g of the aqueous phase. Oil phase was made of pure Jatropha oil while the aqueous phase consists of various compositions of 0.5, 1.0, 1.5 and 2 wt% of surfactant, 12, 14, 15 and 16wt% of co-surfactant, 1,2,3,4,5 and 6 wt% NaCl, and de-ionized water. After adding both the oil and aqueous phases, the mixture was shaken at low frequency of 100 rpm to avoid large oil droplet size formation. The mixture was then shaken for a day to ensure well-mixed and uniform mixture due to relatively large sample volume before transferred into a separating funnel and left to stand for about two weeks to reach equilibrium. The funnel was sealed with a tight lubricated Teflon stopper screw cap to avoid oil interaction with air leading to oil degradation. Type I microemulsion (oil-in-water microemulsion) was expected to form after the equilibrium. The synthesized Winsor type I microemulsions (lower phase) was collected by slowly separating Winsor type I microemulsion from excess oil in the separating funnel. It is to ensure that the desired formulated microemulsions were not mixed with excess oil phase while being separated from separating funnel.

IFT Measurement:

Prior to IFT measurements, the density of both n-octane and prepared microemulsion were determined. About 1.0 cm³ of sample was filled into the oscillating U-tube capillary of a Density/Specific Gravity Meter DMA 4500/5000 (Anton Paar, Austria) via a syringe at 30 °C. Measurement was repeated for all samples of microemulsion and n-octane. Following the general procedure, the light phase was injected into the rotating capillary filled with the heavy phase. During the injection, the capillary was rotated at the speed of 1000–2000 rpm. The rotation speed was increased to 4000rpm after the equilibrium was reached. The system reaches the equilibrium when the diameter of the droplet did not change with time (Drelich *et al.*, 2002). Since in all of the IFT measurements the system reached equilibrium after a very short time of about 10 seconds, the depletion of co-surfactant into the n-octane phase was considered negligible. Measurements were carried out in triplicate. The IFT was estimated using Vonnegut's equation, which relates IFT to the droplet radius of light phase, rotational velocity, and the densities of the two phases (Vonnegut, 1942).

Sand pack design and microemulsion flooding:

Sand pack flooding (Fig. 1 and Fig. 2) tests were conducted to measure the oil recovered after microemulsions flooding in oil recovery. The sand used in the experiments composed of SiO₂ and CaO with particle size of 100 mesh or 0.150 mm. Both sides were equipped with stainless steel sieves to prevent any sand from flowing. The sand pack was placed horizontally and flooded at atmospheric pressure and room temperature.

Specific steps were followed during the microemulsion flooding. They are described below. The flow rates of the injections were kept constant at 0.83 ml/min in order to mimic real field injection velocities (Iglauer *et al.*, 2010; Kumar & Mohanty, 2010).

1. Brine with 1wt% NaCl concentration was injected for about 3 pore volume (PVs).
2. 3 PVs of n-octane was then injected to reach connate water saturation.
3. 2 PVs of brine with 1wt% NaCl was again injected to reach connate water saturation.
4. The effluents from the sand pack were collected in sample tubes and the incremental oil recoveries were measured as secondary oil recovery.
5. 4 PV of formulated microemulsion was injected and the incremental oil recoveries were measured as cumulative tertiary oil recovery.

Summary of sand pack properties are summarized in Table 1.

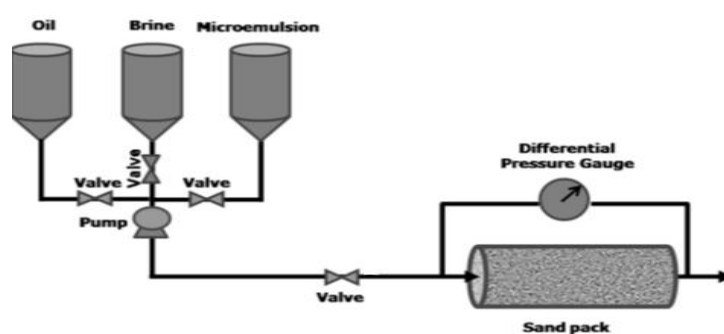


Fig. 1: Schematic of the experimental flooding system (Z Jeirani *et al.*, 2013b)



Fig. 2: Experimental Flooding System Set Up.

Table 1 Summary of Sand Pack Property

Parameter	Sand Pack
Length	45 cm
Diameter	5 cm
Porosity	32%
Permeability	26D
Pore volume (PV)	285ml
Connate water saturation	7%
Brine PV (secondary oil recovery)	2
Residual oil	121.83ml
Flow rate of injections	0.83 ml/min

The amount of recovered oil was quantitatively expressed in term of cumulative tertiary oil recovery, which is the percentage of the volume of the produced oil in the tertiary oil recovery step to the

volume of residual oil (remaining oil after secondary oil recovery step). The same methodologies of sand pack microemulsion flooding and IFT measurement were conducted for all of the microemulsion samples

to determine the optimum microemulsion formulation. The calculated cumulative oil recovery and IFT were used as the main criteria in selecting the most efficient microemulsion formulation.

Optimization of the co-surfactant concentration:

Microemulsion samples were prepared at various concentrations of co-surfactant ranging from 12,13,14,15 and 16wt% of the aqueous phase. The concentration of APG was fixed at 1 wt% of the aqueous phase. All of the samples were prepared at 3wt% salinity. Cumulative oil recovery and IFT results were obtained for all the Winsor Type I microemulsion formulations. The optimum concentration of the co-surfactant in the aqueous phase is the concentration at which its microemulsion yields the highest tertiary oil recovery and the lowest IFT.

Optimization of the surfactant concentration:

Microemulsion samples were prepared at various concentrations of surfactant ranging from 0.5,1,1.5 and 2 wt% of the aqueous phase. The concentrations of co-surfactant and NaCl in the aqueous phase were fixed at their optimum values at 15wt% and 1wt%. Cumulative oil recovery and IFT results were obtained for all the Winsor Type I microemulsion formulations. The optimum concentration of the surfactant in the aqueous phase is the concentration at which its microemulsion yields the highest tertiary oil recovery and the lowest IFT.

Optimization of salinity:

Microemulsion samples were prepared at various salinities ranging from 1 to 6 wt% of the aqueous phase. The concentration of APG (surfactant) and the selected co-surfactant was fixed at 1wt% and 15wt% of the aqueous phase. Cumulative tertiary oil recovery and IFT results were obtained for all the oil in water microemulsion formulations. The optimum concentration of NaCl in the aqueous phase is the salinity at which its microemulsion yields the highest tertiary oil recovery and the lowest IFT.

RESULT AND DISCUSSIONS

Results show that some surfactants do not sufficiently give ultralow interfacial tension at the oil/water interface to form microemulsions. Thus, co-surfactant molecules are added to lower the oil/water interfacial tension. Co-surfactants are molecules with weak surface-active properties that are combined with the surfactants to enhance their ability to reduce the interfacial tension and promote the formation of a microemulsion (Schick, 1987). For a co-surfactant-free triglyceride microemulsion sample, which contains only 1 wt% of APG as surfactant and 3 wt% NaCl in the aqueous phase, its measured IFT against n-octane is 5.3658 mN/m (Z Jeirani *et al.*, 2013a). Thus, to reduce the IFT to ultra-low values, normal butyl alcohol (NBA) was introduced as co-surfactant. Fig. 3 shows one photo of the transparent Winsor Type I triglyceride microemulsion samples in separating funnels.

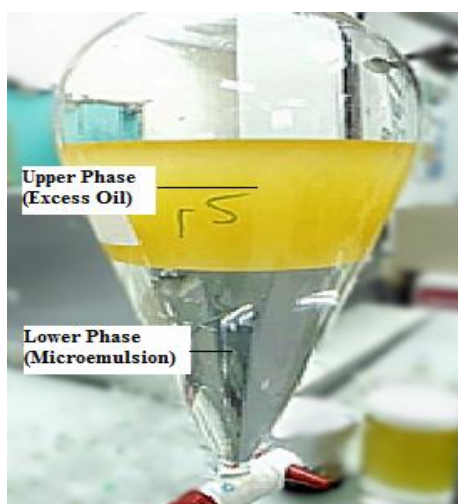


Fig. 3: Prepared Winsor Type I microemulsion.

Optimization of co-surfactant concentration:

Fig. 4 show the IFT variation of the microemulsion samples with the concentration of co-surfactants. The IFT between microemulsion samples and n-octane tend to reduce with increasing of co-surfactants concentration. In general, the observed reduction in IFT with an increase in the volume of

the co-surfactants shows good performance of the co-surfactant in reducing the IFT. The variation of IFT showed that the minimum IFT of 0.0015mN/m was achieved at 15 wt% of NBA concentration. However, it was also observed by increasing co-surfactant concentration to 16 wt%, only slight improvement demonstrated. Thus, 15wt% of NBA concentration

was selected as the optimum co-surfactant concentration in this formulation. It was supported by the formulated microemulsion flooding test as in Fig. 5. The optimum 15 wt% NBA concentration yield cumulative tertiary oil recovery of 78%. It was observed that the efficiency of microemulsion flooding increases with the co-surfactant concentration up to 15wt% NBA concentration. In contrast, above 15wt% of NBA concentration, no

further improvement was observed in term of cumulative oil recovery. This may be due to CMC formation where the co-surfactant tend to agglomerate at aqueous phase of microemulsion that accumulate at oil and water interface (Kamranfar & Jamialahmadi, 2014). Thus, higher interfacial tension was reached which lead to less efficient microemulsion flooding performance.

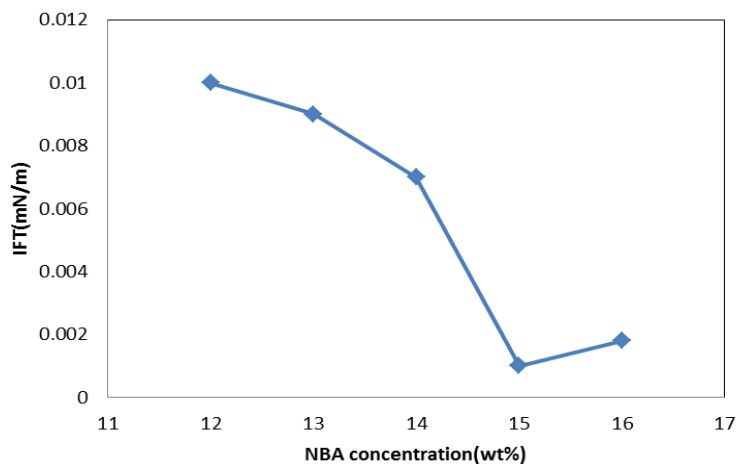


Fig. 4: Interfacial tension of microemulsion at 12, 13, 14 and 15 wt% of APG concentration. The concentrations of APG and NaCl are at 1wt% and 3wt% of the aqueous phase respectively.

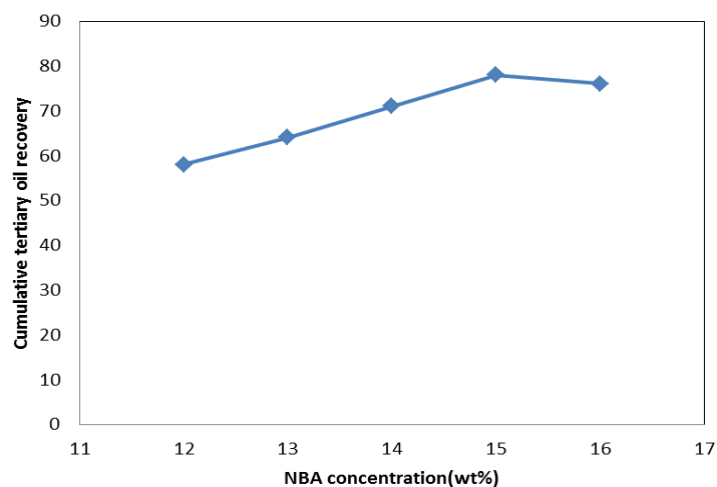


Fig. 5: Cumulative tertiary oil recoveries at 12, 13, 14, 15 and 16wt% NBA concentration. The concentrations of APG and NaCl are 1wt% and 3wt% of the aqueous phase respectively.

Optimization of surfactant concentration:

A total of four microemulsion samples were prepared at APG concentrations of 0.5, 1, 1.5, and 2 wt% of the aqueous phase. In all of the samples, the concentration of NBA and NaCl were kept constant at 15wt% (optimum concentration of co-surfactant) and 3 wt% (optimum salinity), respectively. Both the variation of IFT and cumulative tertiary oil recovery with the microemulsion samples at various APG concentrations are shown in and. When the concentration of APG in the aqueous phase of the

microemulsion exceeds 1wt%, the IFT starts to increase and the cumulative tertiary oil recovery starts to decrease continuously. It is obvious that the point at which the IFT and cumulative tertiary oil recovery yield their minimum and maximum values respectively indicates the optimum concentration of APG. Therefore, based on the collected experimental data, 1 wt% is selected as the optimum concentration of the surfactant in the aqueous phase of the microemulsion. shows the increases of cumulative

tertiary oil recovery at low APG concentration from 0.5 to 1 wt%.

Fig. 6 indicates the lowest IFT was achieved at 1.5wt%. However, cumulative tertiary oil recovery does not demonstrate further increment of

cumulative tertiary oil recovery. Thus, based on the collected experimental data, 1 wt% APG is selected as the optimum concentration of the surfactant in the formulated microemulsion.

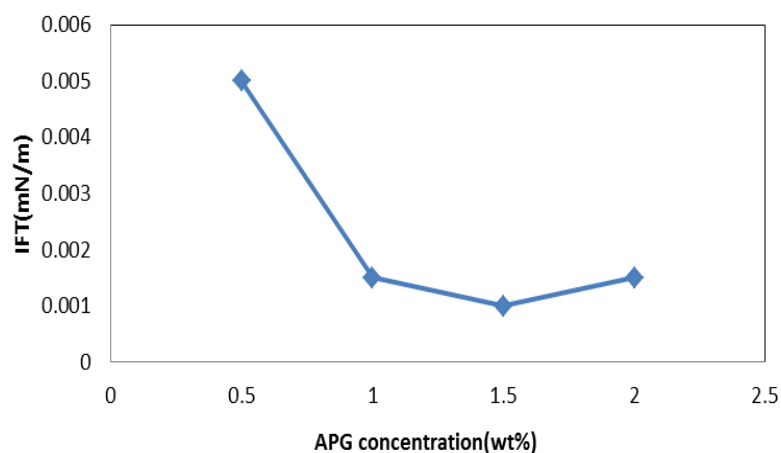


Fig. 6: Interfacial tension of microemulsion at 0.5, 1, 1.5 and 2 wt% APG concentration. The concentrations of NBA and NaCl are 15wt% and 3 wt% of the aqueous phase respectively.

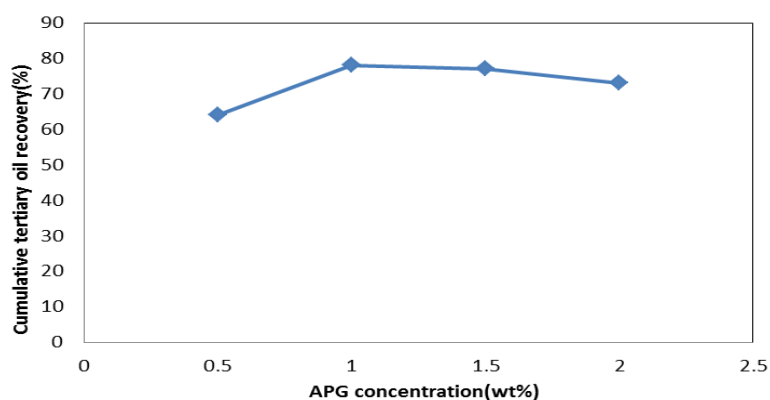


Fig. 7: Cumulative tertiary oil recovery at 0.5, 1.0, 1.5 and 2 wt% APG concentration. The concentrations of NBA and NaCl are 15wt% and 3 wt% of the aqueous phase respectively.

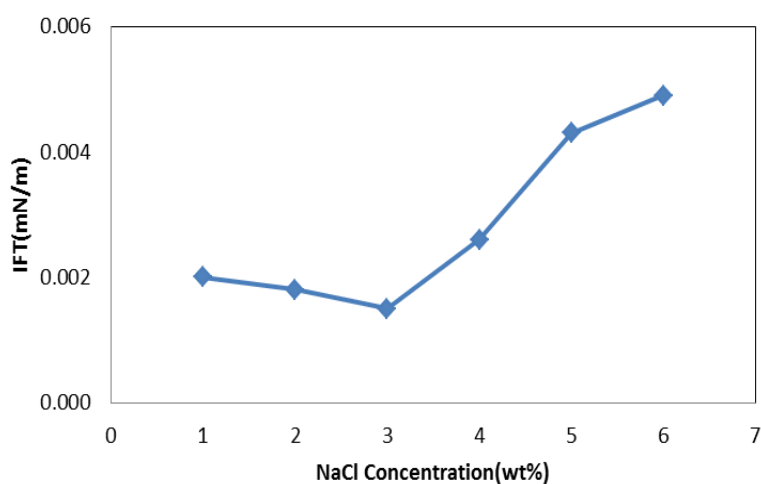


Fig. 7: Interfacial tension of microemulsion at 1, 2, 3, 4, 5, and 6wt% of NaCl concentration. The concentrations of NBA and APG are fixed at 15wt% and 1 wt% of the aqueous phase.

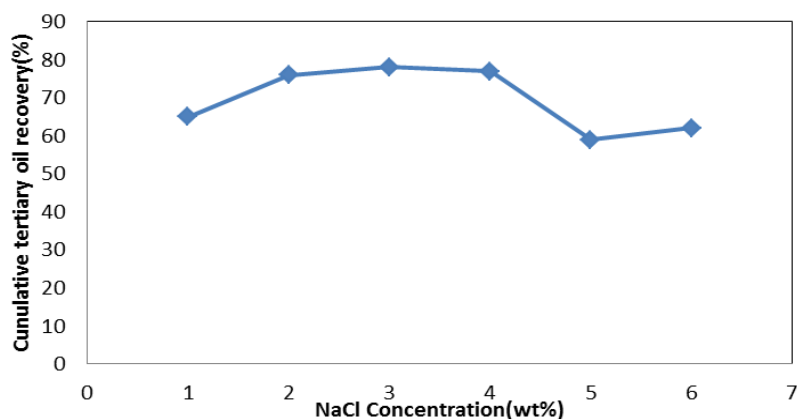


Fig. 8: Cumulative tertiary oil recovery at 1, 2, 3, 4, 5, and 6wt% of NaCl concentration. The concentrations of NBA and APG are fixed at 15wt% and 1 wt% of the aqueous phase respectively.

Optimization of salinity concentration:

A total of six microemulsion samples were prepared at various salinities of 1, 2, 3, 4, 5 and 6 wt% of the aqueous phase. In all of the samples, the concentration of APG and NBA was maintained at 1 and 15 wt%, respectively.

Fig. 7 demonstrates that the IFT of the microemulsion samples against n-octane is fairly constant at various NaCl concentrations up to 4wt% salinity. However, higher NaCl concentration above 4 wt% demonstrates a sudden increase of IFT. The increases of IFT lower the potential of the formulated microemulsion at specific salinity. This scenario suggested less efficiency of tertiary oil recovery that leads to significant reduction of tertiary oil recovery from 78% at 4wt% NaCl to 59% at 5wt% NaCl. This might be due to higher NBA concentration that partially ionized that interact with higher NaCl concentration (Bansal *et al.*, 1980). Thus, 3wt% salinity was selected as constant salinity in this formulation due to its lowest IFT as depicted in

Fig. 7. Lowest IFT indicates high solubilization of both oil and water in the microemulsion phase, thus leading to higher tertiary oil recovery efficiency. Based on Fig. 8 the cumulative tertiary oil recovery yield relatively similar value at lower salinity, which is at 1 to 3wt% salinity.

Conclusion:

The use of NBA as co-surfactant demonstrates high potential in lowering IFT as low as 0.0015mN/m. This can be achieved by increasing concentration up to 15wt% NBA. However, APG concentration showed inverse effects that increase the IFT between microemulsion and n-octane at concentration above 1wt%. The IFT of the formulated microemulsion was found to be almost independent of the salinity concentration at NaCl concentration below 4wt%. However, it shows significant increment of IFT between microemulsion and n-octane at salinity exceed 4wt% NaCl. This suggested less efficiency of tertiary oil recovery that leads to significant reduction of tertiary oil recovery

from 78% at 4wt% NaCl to 59% at 5wt% NaCl. Thus, the optimum composition of the aqueous phase was determined at 1 wt% APG, 3 wt% NaCl, 15 wt% NBA, and 81 wt% deionized water. The optimum formulation results in ultralow interfacial tension of 0.0015mN/m and cumulative tertiary oil recovery up to 78%.

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