Characterization of vesicle for Enhanced Oil Recovery

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A systematic study has been performed employing the use of a cationic surfactant didodecyldimethylammonium bromide (DDAB), an anionic surfactant sodium lignosulfonate (SLS) and water for Enhanced Oil Recovery (EOR) from the underground porous media of the depleting oil field of Naharkotiya, Duliajan, in the Upper Assam basin, India. The strong electrostatic interaction between the oppositely charged head groups present in cationic/anionic surfactant mixtures, the geometric packing ratio can effectively be tailored by varying the ratio of cationic and anionic surfactants. Here the behaviour of aqueous mixtures of the surfactants, SLS and DDAB with reference to Interfacial Tension (IFT) and EOR has also been explored. The zeta ($\zeta$)-potentials are positive at DDAB-rich regions and negative at SLS-rich regions. The vesicles appear to be stable, supported by large absolute values of $\zeta$-potentials and little change in UV absorbance for several months. These vesicles can recover 46.45% oil from water flooded porous media compared to micelles of SLS and DDAB, which can recover 16.76% and 36.80% of oil respectively.

Keywords: DDAB, Sodium lignosulfonate, Vesicle, Interfacial tension, Enhanced oil recovery

The interaction between the positively charged cationic and negatively charged anionic surfactants has received much attention because of the special properties of the complex system in biology, chemistry and oil-recovery, etc. The interaction between cationic and anionic surfactants causes the formation of catanionic vesicles\(^1\text{--}\text{3}\). They are attracting much interest because they are formed spontaneously and can be obtained from a variety of surfactants. Kaler et al.\(^4,5\) were first to report the vesicle formation from mixed cationic and anionic surfactants. Phase behaviour for the mixed aqueous surfactant systems of cationic octadecyl trimethyl ammonium chloride (OTAC)/anionic ammonium dodecyl sulfate (ADS)/water was also examined\(^6\). In surfactants mixtures exhibit significant synergism\(^7\). Synergistic effects in mixtures of a cationic and an anionic surfactant have been theoretically investigated by Bergstrom\(^8\). Vesicles and micelles represent two of the important classes of self-assembled structures that can be formed by amphiphiles in dilute or semidilute solutions. Vesicles constitute an interesting morphology formed by self-aggregating amphiphilic molecules. They exhibit a rich structural variety and are of interest both from a fundamental point of view (for studying closed bilayer systems) and from a practical point of view\(^9\). Vesicles are hollow spheres enclosed by a bilayer of the amphiphiles and are commonly used to encapsulate labile hydrophilic molecules within their interior. Micelle tend to occur in a range of morphologies, including spherical, ellipsoidal, and cylindrical structures\(^10\). SLS with one unbranched alkyl tail that contain 12 carbon atoms often forms micelle above the critical micelle concentration (CMC)\(^11\) while DDAB with two alkyl tails often forms vesicles above the critical vesicle concentration (CVC). On the other hand, vesicles formed in cationic/anionic surfactant mixtures are believed to be nanoparticles which are thermodynamically stable, and their size, charge, or permeability can be readily adjusted by varying the relative amounts and/or chain lengths of the two surfactants. Surface-modified nanoparticles in fluids are used to recover hydrocarbon from underground formations. The use of surface-modified nanoparticles in such fluids provides particles that are stable under pressure\(^12,13\). The work reports the results of experimental studies conducted with respect to the following:

(i) IFT of aqueous phase (SLS and DDAB) and oil phase
(ii) On phase behaviour of dilute (below 1% wt.) aqueous DDAB solutions
(iii) Relating IFT and EOR from conventional core samples
In spite of numerous works, concerned to phase behaviour of binary and ternary water-DDAB-hydrocarbon systems, there are only a few data on such important properties of DDAB aqueous solution as CMC, IFT and peculiarity of aggregation process\textsuperscript{14,15}. It was observed that the vesicles, prepared by usual sonication or solvent extraction methods are unstable and eventually revert to liquid crystalline state. This led to the conclusion that all vesicles, unlike micelles or liquid crystals, are in peculiar nonequilibrium state of matter to which the usual theories of self-assembly based on statistical mechanics cannot be applied. However, the spontaneous vesicle formation in DDAB hydroxide solutions was also reported. According to the theory\textsuperscript{16} under certain conditions it should be possible to produce stable vesicle systems. It has been shown that DDAB forms stable vesicles in diluted solutions of 10\textsuperscript{-4} moles/kg. It is further found that the process of spontaneous vesiculation in DDAB solution occurs with increase of surfactant concentration and is accompanied by a sharp fall of IFT. These observations lead to the conclusion that the study deals with the new kind of water/oil interfacial instability stipulated by phase transition both on interface and in bulk solutions.

Present work concentrates on the recovery of residual oil left after the secondary recovery. The residual oil can be treated as thin oil films covering the surface of the rock grains and the clay particles. If the surface area is quite big, the content of the oil film could contain a large part of the residual oil in the reservoir\textsuperscript{17}. The aim of the EOR is to withdraw the residual oil. There are two interfacial interactions: liquid (flooding solution)/liquid (oil) and liquid (oil)/solid (rock, sand or clay). So far much effort has been done for the former. Surfactants have synergistic enhancement in lowering IFT, leading to the formation of O/W emulsion\textsuperscript{18}. Basically lower the IFT, the easier the recovery of residual oil, and more efficient is the EOR process\textsuperscript{17,19}. The understanding of the mechanisms of EOR using surfactant and microemulsion flooding has been attempted\textsuperscript{20,21}. Roughly 20% of the otherwise unrecoverable underground oil can be obtained by the EOR process. The oil remains trapped in the reservoir because of high IFT (about 20–25 mN/m) between the crude oil and reservoir brine. If the IFT can be reduced to around 10\textsuperscript{-3} mN/m, a substantial fraction of the residual oil in the porous media in which it is trapped can be mobilized. Low interfacial viscosity of the system also plays an advantageous role. Surfactants will simultaneously form three coexisting liquid phases when mixed with oil and water within well defined salinity and temperature interval. The middle phase which contains the greatest concentration of the surfactant mixture, called a microemulsion reduces the IFT between oil and water thus allowing the oil-rich (top) phase to be pushed through porous media by the aqueous (bottom) phase, and the three liquid phases flow as macroemulsions.

Experimental Procedure

Materials

Pure cationic surfactant DDAB was supplied by Focus Synthesis LLC San Diego, CA, USA and used as received. Double distilled and deionized water was used. Hydrocarbons were distilled and purified from polar impurities by passing through aluminum oxide, incandesced at 973°K. The surface tension of treated water was 71.81 mN/m and electrical conductivity was (1.05±0.1) ×10\textsuperscript{-4} s/m. The molecular weight of DDAB [CH\textsubscript{2}N\textsuperscript{Br}(CH\textsubscript{3})\textsubscript{2}(CH\textsubscript{2})\textsubscript{9}CH\textsubscript{3}] was 0.494 kg mol\textsuperscript{-1}. DDAB is a double-chained surfactant and thus it tends to self-assemble in water into bilayer structures of vesicles and lamellar phases. The paraffin oil of viscosity (μ\textsubscript{o}) 113.51×10\textsuperscript{-3} Pa-s and density (ρ\textsubscript{o}) 0.920 kg/m\textsuperscript{3} was collected from Digboi refinery, Assam. Crude oil is not used because it is very viscous at normal temperature at which the flooding experiments were conducted in the laboratory. Paraffin oil is taken as a standard to generate data, which will be substantiated for the actual study. The brine solution was 3000 ppm of NaCl in distilled water (DW) having viscosity (μ\textsubscript{w}) of 9.67×10\textsuperscript{-3} Pa-s and density (ρ\textsubscript{w}) of 1 kg/m\textsuperscript{3}. Analytical grade NaCl was procured from BDH (Mumbai, India). Black liquor (BL), a waste was collected from Nowgong Paper Mills of Jagiroad, Assam. It is an anionic surfactant whose main constituent is SLS. BL was subjected to vacuum evaporation and freeze drying to remove the water, sulphur-di-oxide and other volatiles. The dried spent sulfite liquor solids (SSLS) so obtained contains 98.82% of SLS which is taken for the study. The porous media for flooding experiment was the conventional core sample obtained from the oil bearing formation at a depth of 3856-3859 m from Naharkotiya oil field belonging to Oil India Limited, Duliajan, Assam. The molecular weight of SLS (C\textsubscript{10}H\textsubscript{14}N\textsubscript{2}O\textsubscript{2}H\textsubscript{2}O) was 0.535 kg mol\textsuperscript{-1}.

Materials

Experimental Procedure
Interfacial tension measurements

The oil and aqueous surfactant solutions (both cationic and anionic) were contacted with minimum disturbance at the interface, in a volumetric oil-water ratio of 1:2\textsuperscript{22,23}, after about 7200 s (2 days) of equilibration the IFT's were measured using a spinning drop tensiometer (BOSS Engg Ltd. Chicago, USA, Model 500).

Particle size measurements

The diameter of vesicles was determined by DLS (Dynamic Light Scattering measurement), which was made with a spectrometer of standard design (CILAS 1180 Model) and a Laser deflection particle size analyzer.

UV-Vis measurements

The UV-Vis spectra were recorded on a Spektralphotometer SPECORD 205 P, Analytic Jena AG calibrated at 190 to 300 ηm wave-lengths at room temperature of 298 K. The vesicles were dissolved in water, their concentration being in the range of 7×10\textsuperscript{-4} M (M=moles/L).

ζ -Potential determination

The ζ-potentials of the mixed micelles were obtained by measuring their electrophoretic mobilities with a ZetaSizer 3000 (Malvern Instruments Ltd.) and by applying a Smoluchowski equation in which the viscosity and dielectric constant of water of the measured temperature were used. Marian Smoluchowski was the first to properly derive an equation to calculate the zeta potential from electrokinetic mobility:

\[ \mu = \zeta \varepsilon / \eta \]  

where ζ = the zeta-potentials; \mu = the electrophoretic mobility; \varepsilon = the electric permittivity of the liquid and η = the viscosity. Here the SI system has been used, in old literature CGS units were used and all equations relating to electricity have a different form. The external DC voltage applied for the mobility measurements was kept constant at 100 V. Each sample was injected to the cell by the syringe, so as to avoid the generation of bubbles in the cell\textsuperscript{24}. The stationary levels in the ζ-potential cell were found from a Van Gils plots. The ζ-potential measurements were performed at room temperature at the upper stationary level. The arithmetic mean was taken from several ζ-potential measurements of the same sample after the highest and lowest values were deleted. The averaged ζ-potential was corrected for the retardation of the electrical double layer by Henry’s function\textsuperscript{25}. The pH of the solutions were measured with a pH meter. The samples of SLS/DDAB mixtures with the SLS mole fraction of 0, 0.2, 0.4, 0.6, 0.8 and 1 were prepared using the total surfactant concentrations slightly above the CMCs, so that there were enough particles, not to cause multiple scattering. Also the samples with dilutions were used for measurements. Each sample was injected to the cell by a syringe and at this time care was taken so that no bubbles were produced in the cell. If there are bubbles in the cell, their movement would be seen as the movement of particles and would change the result.

First the solutions were prepared by taking the CMC of DDAB and SLS in water. Say the solutions A and B are made. Then A and B solutions were combined with water at different molar concentrations in test tubes (say a, b, c etc), shook adequately and immersed in a temperature controlled bath at 308 K for 25200 s (7 days). Samples a, b, c etc were withdrawn to determine the phase diagram. The solution and gel phases of the samples a, b, c etc which cannot be separated by shaking or stirring were then separated by centrifugation for 30 min. The centrifuged solution samples (a, b, c etc), were allowed to equilibrate without stirring for 7 days. Thereafter, the phases were separated by careful decantation of the solution. The dry content of the phases was determined by weighing. The amount of DDAB was determined by chemical analysis. The amount of SLS was calculated as the difference between the amount of DDAB and the total amount of dry matter in the samples. The composition and amount of SLS in the supernatant phase was determined by standard gas chromatographic method.

Permeability test and oil recovery

The permeability test was conducted in a cylindrical section of 0.3048 m length and diameter 0.0381 m, operated vertically in the experimental setup described elsewhere\textsuperscript{28,29} (Fig. 1). The setup essentially comprises a cylinder packed with conventional core sample, pressure gauges, sample reservoir, sample collector, stirrer and a pump all connected by pipes of 0.0127 and 0.022225 m (1.27 and 2.2225 cm) outside diameter. Flooding solutions were stirred in the reservoir and injected at the bottom of the cylindrical section at a constant volumetric flow rate of 2×10\textsuperscript{-8} m\textsuperscript{3}/s by self priming monoblock 186.425 W (0.25 HP) pump supplied by Telco, Coimbatore, India. The inlet and outlet pressure of the
cylindrical section was recorded from pressure gauges. A constant superficial velocity of $7.3 \times 10^{-5} \text{m/s} + 1.62 \times 10^{-6} \text{m/s}$ and $3.472 \times 10^{-6} \text{m/s}$ was maintained during the brine and surfactant flooding steps respectively. Experiments were conducted at room temperature of 298ºK.

The absolute permeability ($K$) of the porous medium to brine measured during initial brine flooding was found to be 86.14 md when both flow rate and pressure were constant. The pore volume of the porous medium as calculated by saturation method is 29.43 mL. Paraffin oil was injected into the test sample that was saturated with brine and brine was displaced by oil until the irreducible brine saturation ($S_{wc}$) was reached. The relative permeability ($K_{ro}$) of oil at $S_{wc}$ was calculated and the oil in the test sample was the initial oil saturation ($S_{oi}$). The test sample which was saturated with $S_{wc}$ and $S_{oi}$ was flooded by secondary brine flooding. During secondary brine flooding the breakpoint i.e. the point at which the first drop of brine comes out was noted and the brine with oil that flows out from the test sample was collected in graduated test tubes. The difference in pressure was noted at about $5 \times 10^{-6}, 10 \times 10^{-6}, 20 \times 10^{-6}, 40 \times 10^{-6}, 80 \times 10^{-6}, 130 \times 10^{-6}$ and $260 \times 10^{-6} \text{m}^3$ effluent of brine and oil collection. Simultaneously the relative permeabilities of the test sample to oil and water were also calculated. Oil was separated by adding deoiler Catflo (Catalytic polyelectrolyte) supplied by Thermax, Pune, India, with constant agitation by means of a stirrer. Secondary brine flooding was continued till residual oil saturation ($S_{ro}$) was reached. When no further oil was recovered by brine flooding and the core was saturated with residual oil saturation ($S_{ro}$), surfactant slug was used for enhanced oil recovery.

**Results and Discussion**

**Critical micelle concentration (CMC) of DDAB/SLS/Water systems**

The results of the IFT between the aqueous solution and paraffin oil are plotted in Fig. 2. The aqueous solution contains DDAB or SLS or a surfactant mixture (DDAB+SLS). It is seen that the CMC and IFT are minimum when the aqueous solution contains DDAB+SLS and maximum for SLS. It is observed that an IFT as low as $0.08 - 0.1 \text{mN/m}$ is obtained at DDAB+SLS concentration of $3.68 \times 10^{-4}\% \text{ g/L}$. Rosen et al. observed that at concentrations below 0.01 wt%, when mixtures of certain surfactants are used at the proper ratio, ultra low IFTs were recorded. The IFT decreased with increasing surfactant concentration as was observed by Taft et al., and each IFT curve had a break point, which was taken as the CMC. The CMC of the surfactant mixture i.e., the vesicle is much lower than the individual surfactants because the double hydrocarbon chain of DDAB together with single hydrocarbon chain of SLS produces strong hydrophobicity, hence the whole molecule becomes hydrophobic and vesicle will form at lower solution concentration, i.e. the CMC decreases. The CMC of mixed surfactant system at 298ºK and 308ºK by taking several mole fractions of SLS ($\alpha_{SLS}$) is shown in Fig. 3.

**Particle size**

The mixing of a negatively charged hydrophilic SLS with a positively charged hydrophobic macromolecule such as DDAB will lead to formation of nanometer particles with desirable properties. SLS
loaded DDAB nanometer particles were prepared by the spontaneous addition of DDAB to SLS. A solution of SLS (molecular weight = 0.535 kg.mol⁻¹) was added dropwise to a solution of DDAB (molecular weight = 0.494 kg.mol⁻¹) and vortexed for 30 s at room temperature. Figure 4a and 4b show the particle size distribution of unilamellar vesicle (ULV) of the surfactant mixture, mixed in the ratio 33 of SLS: DDAB = 6:4.

The surfactant mixture of 0.01 M after stirring for 1209.6 s (0.014 days), is found to be a monodispersed system (Fig. 4a). But the surfactant mixture > 0.015 M is a polydispersed system with two distribution peaks (Fig. 4b). It indicates that to form monodispersed vesicles a proper mole concentration of the surfactant mixture is necessary. Results in Fig. 4a and 4b show that vesicles exhibit nanometer size particles.

ζ-Potential of vesicles

ζ-Potentials of the mixed micelles of six different samples (α_{SLS} = 0, 0.2, 0.4, 0.6, 0.8 and 1) were measured and the results are presented in Fig. 5. The ζ-Potentials are large in magnitude. For pure DDAB and SLS micelles, the ζ-Potentials are 97 and -87 mV respectively. The ζ-Potentials of the mixed surfactant micelles lie between the pure components. As α_{SLS} increased ζ-Potentials decreased and became negative beyond α_{SLS} = 0.57. It is interesting to observe that ζ-Potentials α_{SLS} = 0.6 and α_{SLS} = 0.8 are negative. There are virtually no micelles in the region between α_{SLS} = 0.4 and α_{SLS} = 0.6 values (Fig. 5). Hence, ζ-Potentials of the mixed micelles could not be measured between these α_{SLS} values. The ζ-Potentials
at $\alpha_{\text{SLS}} = 0.4$ and $\alpha_{\text{SLS}} = 0.6$ were those of the vesicles because they were measured in the vesicular region slightly above the phase boundary.

At the SLS/DDAB ratios between 4/6 and 6/4, the point of zero zeta potential (pzz) is achieved for the air-water interface. This signifies that the isoelectric point (the point where the pH=7) is located somewhere between these ratios.

Stability of vesicles

Figure 6 depicts the UV-Vis spectra of vesicles just after their formation: Series 1; after 864000 s (10 days); Series 2; after 1728000 s (20 days); Series 3 and Series 4 after 2592000 s (30 days), which were measured to examine the stability of the vesicles. The absorption maximum of vesicles were situated between 165 and 180 nm. The absorbance remains same for 5184000 s (2 months) indicating that the vesicles are quite stable during the period. Also the high absolute value of $\zeta$-Potentials in Fig. 5 indicates that strong repulsive forces exist amongst the vesicles and the stability of the vesicles can be manifested. Anionic surfactant SLS is responsible for the thermodynamic stabilization of vesicles as well as for the change of vesicle sizes.

General phase behaviour of DDAB/SLS/Water systems

Figure 7 shows the general phase behaviour of DDAB/SLS/Water systems studied. The isotropic phase is of relatively low viscosity and is transparent. For $0 < \alpha_{\text{SLS}} < 0.2$ and $0.8 < \alpha_{\text{SLS}} < 1.0$, the large micellar regions (M) are formed. Meanwhile, for $0.2 < \alpha_{\text{SLS}} < 0.8$, micellar fluid channel is closely connected with the vesicular channel (Fig. 6). The vesicle solution channel was found to be turbid and the turbidity increased with increasing total surfactant concentration. At the fixed total surfactant concentration the turbidity increased with increasing $\alpha_{\text{SLS}}$. From right to left of the phase diagram, the molecular solution (I) appears first and as the concentration of DDAB increases with the decrease of SLS concentration the micelles (M) are formed. At low water concentrations and higher DDAB concentration and almost constant SLS concentration the mixture of micelles and vesicles (M+V) starts to form. A phase transition is confirmed by turbidity and dispersion intensity of light from a vesicle. When a membrane of vesicle is changed from a gel state to a liquid crystalline state, turbidity is decreased due to difference of a density of two states and molecular packing. On continuously increasing the concentration of SLS, the white precipitate disappears and transforms to gel is located in the bottom of the test tube (gel in the bottom). With the further increase of SLS concentration the gel is collected in the test tube (gel in the top). The apparent viscosity of the gel phase decreases with increasing the ratio of DDAB:SLS. In lower SLS concentration phase separation reveals precipitation and the precipitate formed in the samples do not transform into transparent gel for a long equilibrium. Only when the SLS concentration reaches a certain value, gel is phase appears. The reason for this difference of behaviour is that Na$^+$ ion contributes to the ionic strength of the sample. The interaction between the electrolyte and cationic
surfactant (DDAB) is therefore more screened at a high SLS concentration, and less dense concentration phase results.

Permeability test and oil recovery

The results of permeability experiment when the porous media NH$_2$ was flooded by brine and surfactant are included in Table 1. The permeability tests were conducted from the test data using JBN method in unsteady - state displacements. It is seen from Table 2 that the initial water saturation ($S_{wi}$) was 9.28% PV and initial oil saturation ($S_{oi}$) was 90.72% PV in NH$_2$, and oil recovery was 49.91% PV (Table 2 and Fig. 9).

Relative permeability is a direct consequence of the different proportions of each different fluid present in the porous medium and as such is directly dependent upon the percentage saturation of the wetting and the non-wetting phase. The lower curve of Fig. 8 shows imbibition where water displaces the oil, also describes how oil relative permeability ($K_{ro}$) reduces from a maximum value of $S_{oi}$ to $S_{or}$. The value of maximum water permeability, and the shape of $K_{rw}$ and $K_{rw}$ curves, depends on a number of factors. In these cases $K_{ro}$ remains high and $K_{rw}$ remains low as water saturation increases. This means that water velocity remains lower than oil velocity until the point at which $K_{rw}$ and $K_{rw}$ become equal at crossover. Hence the ability of water to bypass oil was suppressed, as water breakthrough during permeability experiment was delayed. As the cross over point was achieved at water saturation of less than 50% in NH$_2$ it was assumed to be oil-wet porous medium. The theoretical plot of relative permeability against water saturation as shown in Fig. 8 is the same as in literature.

It has been demonstrated that mobilization of oil can be achieved by lowering the IFT sufficiently. However, cationic/anionic surfactant mixtures exhibit the largest synergistic effects such as reduction in CMC and IFT. There is physically a simple explanation for enhanced synergism in such mixed charge system. When anionic surfactant with longer alkyl chain forms precipitates with cationic surfactant DDAB the precipitates formed by two surfactants will have more hydrophobicity than the pure surfactants. This will make the precipitates diffuse easily and increase their adsorption onto the water surface. As a result, CMC of the mixed micelle will be lowered. Under such circumstances the oil may be trapped by capillary forces. The additional oil recovery by using DDAB+SLS, DDAB and SLS from the $S_{or}$ (residual oil saturation left after brine flooding) is shown in Fig. 10; the highest recovery from $S_{or}$ by mixed surfactants (DDAB+SLS) was 18.98% PV with a water cut of 68.91% PV, followed by DDAB and SLS as shown in Table 2. It is observed that lower the IFT (Fig. 2) better is the EOR. In this case DDAB+SLS have the lowest IFT and hence the oil recovery is the maximum, followed by DDAB and SLS flooding. Obviously, the vesicles formed by DDAB play an important role in EOR process. Vesicles display interesting behaviour, such as lower critical concentrations and larger IFT reduction (Fig. 2). Vesicles exhibit better surface-active properties compared to conventional surfactants like SLS, better
solubility in water, and good oil solubilization properties compared to SLS alone.

Conclusion

These vesicle (DDAB+SLS) have many unique properties such as lowering the IFT between oil and the aqueous phase in which the surfactants are mixed. This lowering of IFT leads to the recovery of the trapped crude oil from the water flooded natural porous media. Mixtures of cationic (DDAB) and anionic (SLS) surfactants form vesicles which are nano size particles and are very stable. At larger concentrations of either surfactant, the vesicles appear to be stable due to large absolute zeta potentials. The absorbance remains unchanged for two months, implying that the vesicles are quite stable for a long time and the absorption maximum of vesicles lies between 165 and 220 nm. The phase behaviour of the DDAB/SLS/water system was investigated. When water is very rich and the total surfactant concentration is below 0.4 wt%, four distinct phase regions of isotropic molecular solution (I), micellar solution (M), vesicles (V), and a mixture of micelles and vesicles (M+V) have been observed. Each of the phase regions was examined by visual inspection. In the vesicular region, the (DDAB+SLS) mixed surfactant vesicles were formed spontaneously. So it can be concluded that the vesicle (DDAB+SLS) are efficient in enhanced oil recovery from water flooded porous media mainly because vesicles drastically lower the IFT between the aqueous phase and oil and also it is due to its stability for a long period.

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