

Composition of *n*-dedocyl polyglucoside based middle phase microemulsions in surfactant-enhanced aquifer remediation

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The composition and solubilization power of the middle phase microemulsion formed by *n*-dodecyl polyglucoside (APG)/*n*-butanol/water/non-aqueous phase liquids (NAPLs) quaternary system have been studied. The hydrophile-lipophile balanced (HLB) plane equation for the quaternary system has been deduced. Some physicochemical parameter, such as the mass fraction of *n*-butanol in the HLB interfacial layer, the coordinates of the start and end points of the middle phase microemulsion, the mass fractions of APG and *n*-butanol in the total system, and the solubilities of APG and *n*-butanol in oil phase are calculated. The washing liquids prepared according to the physicochemical parameters of the *n*-dodecyl polyglucoside based middle-phase microemulsion have been used to wash the NAPLs adsorbed on soil. The results indicate that the APG based middle phase microemulsion is suitable in surfactant-enhanced aquifer remediation.

Keywords: Microemulsions, Polyglucoside microemulsions, Middle phase microemulsions, Solubilization power, Surfactant enhanced aquifer remediation

Soil contamination from the non-aqueous phase liquids (NAPLs) is one of the main cause of pollution¹. In order to repair the soils, various techniques have been developed among which surfactant-enhanced aquifer remediation (SEAR) has gained more and more attention in recent years²⁻¹¹. SEAR is an excellent alternative technology compared with the traditional pump-and-treat technology in removing NAPLs^{12,13}. Surfactant-based technologies are ascribed to two different mechanisms in enhancing remediation. One is the surfactant-enhanced solubilization whereby hydrophobic contaminants partition into the micellar cores. Another is surfactant-enhanced mobilization whereby contaminants and surfactant solutions form middle-phase microemulsions with ultralow

interfacial tensions (IFTs). This ultralow IFT causes bulk oil displacement, leading to more NAPLs being solubilized in middle phase microemulsions. Surfactant-enhanced solubilization can increase remedial efficiency by one to two order(s) of magnitude while surfactant-enhanced mobilization achieves even greater efficiencies¹⁴. Increasing NAPLs solubility by using surfactants can potentially reduce remediation time¹⁵. Microemulsion has largest solubilization power and the ultra low IFT between oil and water, therefore, it is the most ideal or impactful technology in SEAR¹⁶⁻¹⁹.

Alkyl polyglucosides (APG or C_iG_j) are of importance in microemulsion research due to their excellent biodegradability, good surface-activity and renewability²⁰⁻²². One potential use for APG is to form microemulsion, which is less sensitive to temperature²³. Among the various methods for study of microemulsions, the Winsor phase diagram is commonly used²⁴⁻²⁶. From the Winsor phase diagram, the phase types and phase volumes can be visually observed, and the salinity (or alcohol) window and the optimum salinity (or alcohol) concentration can be calculated. However, the composition and the solubilization power of the middle-phase microemulsion cannot be obtained precisely. That is, it is difficult to use the middle-phase microemulsion with their optimum properties. We have proposed a novel method in studying the microemulsions, viz., ϵ - β fishlike phase diagram, from which the composition and solubilization power could be obtained²⁷⁻²⁹.

On the present study, the microemulsion formed by *n*-dodecyl polyglucoside (APG)/*n*-butanol/water/non-aqueous phase liquids (NAPLs) quaternary system was studied with ϵ - β fishlike phase diagrams. The composition and the solubilization power of the microemulsions were investigated. The washing liquid was prepared according to the composition of the middle-phase microemulsion, and used to remove the NAPLs from the contaminated soils. The work will provide good insights into the role of microemulsion in SEAR.

Experimental

The NAPLs used in the experiments were *o*-dichlorobenzene, carbon tetrachloride, methylene

chloride, *n*-hexane and toluene (Sinopharm Group Chemical Reagent Co. Ltd, AR grade), and were used without further purification. The surfactant used in the experiments was *n*-dodecyl polyglucoside (APG or $C_{12}G_j$), which was synthesized by our research group and purified twice before experiments²³. The composition was determined by gas chromatography GC-9A with an FID detector, an HP 19091 J-413 capillary column (30.0 m×320 m×0.25 m) and 5% phenyl methyl siloxane as stationary phase at 310 °C (12 °C/min). The experiments were carried out under N_2 at 0.8 mL/min N_2 and split ratio of 50:1. The results showed that the $C_{12}G_j$ was $C_{12}G_{1.46}$ (monoglucoside 70.71%, diglucoside 17.42%, triglucoside 6.45% and tetraglucoside or more 5.42%).

Soil was collected from the campus of Shandong Polytechnic University, dried in an electric blastdrying oven (DHG-9620A, Shanghai Yiheng Scientific Instruments Limited Company) at 150 °C for 12 h, and screened through a 2 mm sieve to remove the coarse fragments. The soil was separated into 5 groups (about 5.0 g per group) and mixed with the five NAPLs (about 2.0 mL) mentioned above. Washing liquids to wash the contaminated soils were prepared according to the compositions of β_E and ε_E .

Water used in all experiments was doubly distilled by SZ-93A water distiller (Shanghai Yarong Biochemical Instrument Company). An FA1104 electron balance and a 501 super thermostat were used in these experiments.

Based on the tetrahedral phase diagram at constant temperature and pressure, in a quaternary system of surfactant (S)/alcohol (A)/oil (O)/water (W), the mass fractions of surfactant (β), alcohol () in the system, and the mass fraction of oil to water and oil (α) are defined by Eqs. (1)-(3), respectively.

$$\beta = \frac{W_s}{W_s + W_A + W_O + W_W} \quad \dots (1)$$

$$\alpha = \frac{W_A}{W_s + W_A + W_O + W_W} \quad \dots (2)$$

$$\alpha = \frac{W_O}{W_O + W_W} \quad \dots (3)$$

where W_s , W_A , W_O and W_W are the mass of surfactant, alcohol, oil and water, respectively.

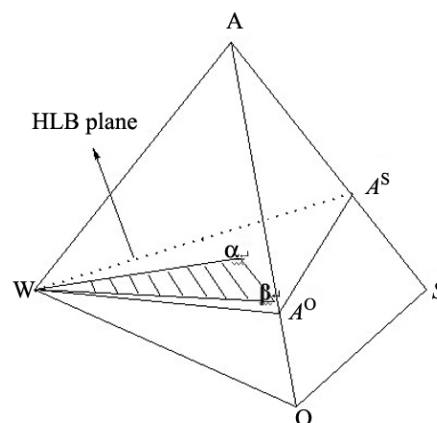


Fig. 1 — HLB plane in a tetrahedral phase diagram for surfactant (S)/alcohol (A)/oil (O)/water (W) quaternary system; the shaded area means a three phase triangle in the midst of a three phase region. A^O : solubility of alcohol in oil, A^S : the mass fraction of alcohol in the interfacial layer. [Reproduced from Ref. 29 with permission from Elsevier].

At constant temperature, pressure and mass ratio of oil-to-water, the phase behavior in a cross profile of the tetrahedral phase diagram is a plane (Fig. 1)²⁹.

The surfactant solutions were prepared by weighing certain amount of APG into a large number of teflon-sealed glass tubes and diluted to the desired concentrations with distilled water. Then, the NAPL equal to the amount of APG and water was weighed into the tubes, and the total volume of the solution was fixed at 10 mL. *n*-Butanol was injected into the samples monotonically while the APG concentrations were fixed at varying values. All samples were allowed to equilibrate at room temperature (25 °C) in a water bath for 10 – 14 days after being shaken for 2 min, in order to bring the water, NAPLs and APG in sufficient contact with each other. The phase equilibrium was determined by visual inspections of the samples with different overall compositions. The volumes of the three different phases were recorded and the ε - β fishlike phase diagram was plotted.

If α (0.50), temperature and pressure (normal atmosphere) are held constant, and is plotted horizontally and is plotted vertically, the - phase diagram can be obtained (Fig. 2).

Results and discussion

The - phase diagrams of microemulsions formed by APG/*n*-butanol/NAPLs/water quaternary systems (Fig. 2) at 25 °C and $\alpha = 0.5$ look like a fish, called - fishlike phase diagram²⁹. The “fish head” (point B) is downward, which is the beginning of the middle-phase microemulsion, and the “fish tail” (point E) is upward,

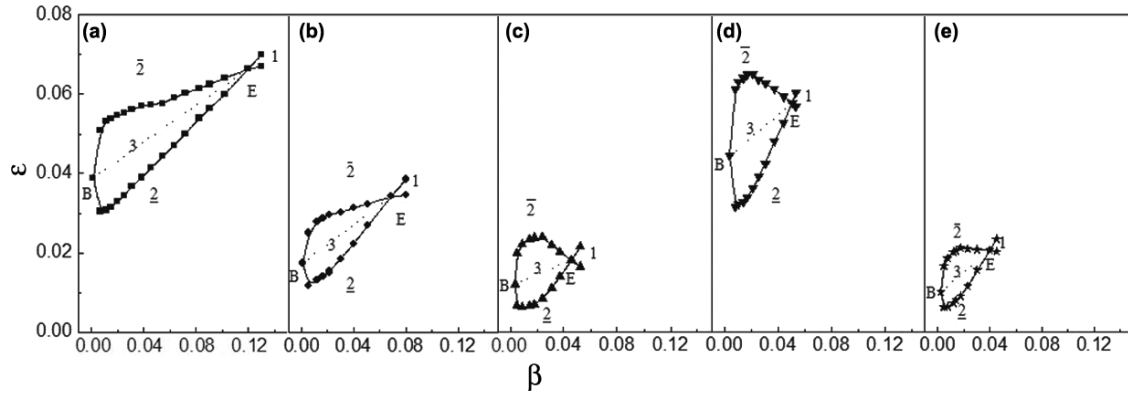


Fig. 2 — The fishlike phase diagram for the APG/*n*-butanol/NAPLs/water quaternary system at 25 °C and $\alpha = 0.5$. [(a) *n*-hexane; (b) toluene; (c) carbon tetrachloride; (d) *o*-dichlorobenzene; (e) methylene chloride].

Table 1 — Values of β_B , ε_B , β_E , ε_E , S^O , A^O , C_S , C_A , A^S_{eff} , A^S and R_{mol} for quaternary system APG/*n*-butanol/NAPLs/water

NAPLs	β_B	ε_B	β_E	ε_E	S^O	A^O	C_S	C_A	A^S_{eff}	A^S	R_{mol}
<i>n</i> -Hexane	0.001	0.038	0.120	0.066	0.002	0.075	0.119	0.033	0.219	0.219	1:1.585
	($\pm$)0.0001	($\pm$)0.004	($\pm$)0.02	($\pm$)0.007	($\pm$)0.0002	($\pm$)0.008	($\pm$)0.02	($\pm$)0.004	($\pm$)0.03	($\pm$)0.03	
Toluene	0.001	0.017	0.069	0.034	0.002	0.034	0.068	0.018	0.213	0.216	1:1.513
	($\pm$)0.0001	($\pm$)0.002	($\pm$)0.007	($\pm$)0.004	($\pm$)0.0002	($\pm$)0.003	($\pm$)0.007	($\pm$)0.002	($\pm$)0.03	($\pm$)0.03	
<i>o</i> -Dichlorobenzene	0.003	0.045	0.049	0.059	0.006	0.086	0.046	0.017	0.270	0.270	1:2.11
	($\pm$)0.0003	($\pm$)0.005	($\pm$)0.005	($\pm$)0.006	($\pm$)0.0006	($\pm$)0.009	($\pm$)0.005	($\pm$)0.002	($\pm$)0.03	($\pm$)0.03	
Carbon tetrachloride	0.004	0.011	0.046	0.017	0.007	0.023	0.042	0.006	0.133	0.133	1:0.816
	($\pm$)0.0003	($\pm$)0.002	($\pm$)0.005	($\pm$)0.002	($\pm$)0.0007	($\pm$)0.003	($\pm$)0.005	($\pm$)0.001	($\pm$)0.02	($\pm$)0.02	
Methylene chloride	0.002	0.009	0.040	0.020	0.005	0.019	0.037	0.011	0.227	0.227	1:1.699
	($\pm$)0.0002	($\pm$)0.001	($\pm$)0.004	($\pm$)0.002	($\pm$)0.0005	($\pm$)0.002	($\pm$)0.004	($\pm$)0.002	($\pm$)0.03	($\pm$)0.03	

which is the intersection of the three and single-phase regions. At fixed β , the increase of ε causes a series of phase inversions Winsor I ($\underline{2}$) $\rightarrow$ III ($\underline{3}$) $\rightarrow$ II ($\bar{2}$). The difference of ε values from the beginning to the end of the middle-phase microemulsion at constant β is called the alcohol window, and with the increase of APG concentration, the alcohol window initially increases and then decreases (Table 1).

A section through a phase tetrahedron at $\alpha = 0.5$ is a particular three-phase tie triangle including the microemulsion in the midst of the Winsor III region (Fig. 1). The plane WA^SA^O is called a hydrophile-lipophile balanced (HLB) plane³⁰. The hydrophile-lipophile property of the mixed amphiphilic layer in the quaternary system is just balanced in the midst line of the middle-phase region.

The composition of the microemulsion in the midst line of the Winsor III region obeys Eq. (4)²⁹,

$$\bar{\varepsilon} = \frac{A^S - F\alpha}{1 - A^S + F\alpha} \beta + \frac{F\alpha}{1 - A^S + F\alpha};$$

$$F = \frac{A^O S^S - S^O A^S}{1 - S^O - A^O} \quad \dots (4)$$

where S^S and A^S are, respectively, the mass fractions of surfactant and alcohol in the amphiphilic layer that is composed of surfactant and alcohol, and S^O and A^O are respectively the solubilities of surfactant and alcohol in NAPLs. For given values of α and β , a plot of the mid-point of ε in the three-phase region ($\bar{\varepsilon}$) versus β is linear (the broken line in Fig. 2). The linear equations for different NAPLs are given below, where the values of standard errors (R^2) are approximate to 1, supporting the linear relationship between ε and β .

$$\bar{\varepsilon} = 0.0386 + 0.2313 \quad (\textit{n-hexane}), (R^2 = 0.9934) \quad \dots (5a)$$

$$\bar{\varepsilon} = 0.0171 + 0.2534 \quad (\textit{toluene}), (R^2 = 0.9942) \quad \dots (5b)$$

$$\bar{\varepsilon} = 0.0111 + 0.1408 \quad (\textit{carbon tetrachloride}), (R^2 = 0.9839) \quad \dots (5c)$$

$$\bar{\varepsilon} = 0.0431 + 0.2837 \quad (\textit{o-dichlorobenzene}), (R^2 = 0.9994) \quad \dots (5d)$$

$$\bar{\varepsilon} = 0.0089 + 0.2817 \quad (\textit{methylene chloride}), (R^2 = 0.9993) \quad \dots (5e)$$

If the slope and intercept of Eq. (4) are K and I , respectively, then A^S is obtained by Eq. (6).

$$A^S = \frac{K + I}{1 + K} \quad \dots (6)$$

The molar ratio (R_{mol}) of surfactant to alcohol in the balanced interfacial layer can be calculated from A^S . The values of A^S and R_{mol} are shown in Table 1.

The point B in Fig. 2 indicates the formation of middle-phase microemulsion, while the point E denotes the disappearing of middle-phase microemulsion and the beginning of the single phase microemulsion. The values of β_E and ε_E indicate the minimum concentrations of APG and alcohol, respectively, for getting a single phase microemulsion while the ratio of water to oil is 1. Smaller the values of β_E and ε_E , smaller are the amounts of surfactant and alcohol required to form the single phase microemulsion, and the larger the solubilization of the system.

The volume fraction (Φ) of the middle-phase at the mid-point $\bar{\beta}$ was measured for a series of β values and plotted as a function of β (Fig. 3). Extrapolation of this line to $\Phi=0$ and to $\Phi=1$ yields β_B and β_E , respectively. Then, the values of ε at these two points (ε_B and ε_E) can be obtained by the HLB plane equation (Eq. (5)) (Table 1).

The solubilities of surfactant (S^0) and alcohol (A^0) in NAPLs can be calculated by Eq. (7).

$$S^0 = \frac{B}{(1 - \beta)(\beta + B)} \quad \dots (7)$$

$$A^0 = \frac{B}{(1 - \beta)(\beta + B)}$$

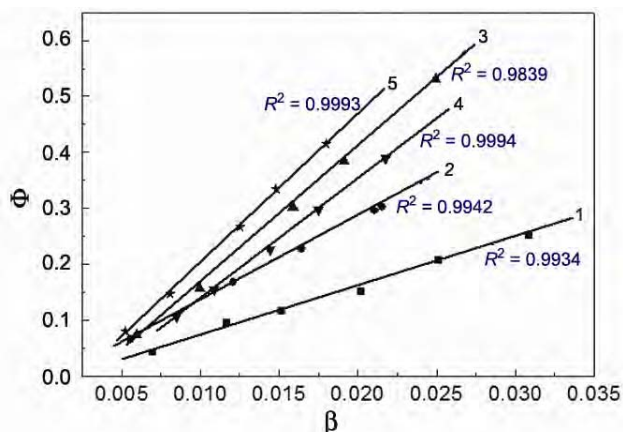


Fig. 3 — Volume fraction (Φ) of the middle-phase at the mid-point of the middle-phase region as a function of β . [1, *n*-hexane; 2, toluene; 3, carbon tetrachloride; 4, *o*-dichlorobenzene; 5, methylene chloride].

The mass fractions of surfactant (C_S) and alcohol (C_A) in the interfacial layer can be calculated by using the simple mass balance equation (Eq. (8)).

$$C_S = \frac{B(1 - \beta_E - \beta)}{(1 - \beta_B - \beta)} \quad \dots (8)$$

$$C_A = \frac{B(1 - \beta_E - \beta)}{(1 - \beta_B - \beta)}$$

In Eq. (7), the solubilities of surfactant and alcohol in the water phase are neglected. This approximation does not significantly influence the values of C_S and C_A due to their lower solubilities³¹. Equation (7) can be used to calculate the relative proportions of surfactant and alcohol in the interfacial layer not only for point E, but also for other points along the trajectory β and $\bar{\beta}$ of the mid-points in the three phase region. The mass fraction of alcohol in the interfacial layer can also be expressed as Eq. (9).

$$A_{\text{eff}}^S = \frac{C_A}{C_A + C_S} \quad \dots (9)$$

The values of S^0 , A^0 , C_S , C_A and A_{eff}^S are listed in Table 1.

From Table 1 it can be seen that the values of β_E and C_S are approximately equal, while C_A is smaller than ε_E . The surfactant molecules mainly dissolve in the interfacial layer, while alcohol dissolves in NAPLs as cosolvent and is also adsorbed on the interfacial layer as cosurfactant. The values of A^S and A_{eff}^S are approximately equal.

The values of β_E , ε_E for toluene are smaller than those for *n*-hexane, which means that the solubilization power of the APG based middle-phase microemulsion for toluene is larger than that for *n*-hexane. This is due to the penetrating ability of NAPL molecules in the surfactant palisade layer. The molecular volume of toluene³² (0.163 nm³) is smaller than that of *n*-hexane³¹ (0.187 nm³), and therefore, the toluene molecule tends to penetrate the surfactant palisade layer to a greater extent. This favors the change of the curvature of the amphiphile layer, consequently, less surfactant and alcohol are needed to balance the hydrophile-lipophile property of the amphiphilic layer²⁹. The molecular volumes of other NAPLs are *o*-dichlorobenzene³³ (0.181 nm³), carbon tetrachloride³⁴ (0.142 nm³) and methylene chloride³⁵ (0.094 nm³). According to this viewpoint, the order of

the amount of surfactant or alcohol for other three NAPLs is *o*-dichlorobenzene > methylene chloride > carbon tetrachloride. However, the values of β_E for *o*-dichlorobenzene, carbon tetrachloride and methylene chloride are approximately equal, while the order of ε_E values is *o*-dichlorobenzene > methylene chloride $\approx$ carbon tetrachloride, which is because the solubility of alcohol in *o*-dichlorobenzene ($A^O = 0.086$) is larger than that in methylene chloride and carbon tetrachloride. The reason may be that the *o*-dichlorobenzene molecule is more polar, and therefore, more alcohol is solubilized. If the mass fractions of alcohol in interfacial layer, C_A , for different chlorocarbon systems are taken into account, the difference will vanish. The orders for β_E and C_A are nearly the same, that is, *o*-dichlorobenzene $\approx$ methylene chloride $\approx$ carbon tetrachloride. From this viewpoint, the influence of volume of chlorocarbon for the solubilization power of the middle-phase microemulsion is small.

The β_E value for toluene is larger than that for *o*-dichlorobenzene, however, the ε_E value is smaller than that for *o*-dichlorobenzene. The molecular polarity and volume of *o*-dichlorobenzene are larger than those of toluene, therefore the solubility of nonionic surfactant APG in *o*-dichlorobenzene is smaller than that in toluene, which is consistent with the values of C_S . Hence, less alcohol is required to balance the hydrophile-lipophilic layer, which is agreement with the values of C_A . As a polar molecule, the solubility of alcohol in *o*-dichlorobenzene is larger than that in toluene.

The order of the values of both β_E and C_S is uniform and slightly different from the values of ε_E . If the solubilities of alcohol in NAPLs (A^O) are taken into account and abstracted from ε_E , the difference disappears. That is, the order of the C_A values is the same as those of β_E and C_S values.

Thus, the order of the solubilization power is: methylene chloride $\approx$ carbon tetrachloride $\approx$ *o*-dichlorobenzene > toluene > *n*-hexane, which means that chlorocarbons are easier to be solubilized in APG based middle-phase microemulsions than hydrocarbons. This may be because the electronegative chlorine atoms easily penetrate the amphiphilic palisade and facilitate the formation of single phase.

The values of molar ratio of surfactant to alcohol (R_{mol}) of the hydrophilic-lipophilic layer, is depended not only on the value of A^S , but also on the NAPL

Table 2 — Recovery rate of the NAPLs with washing liquids with and without APG and *n*-butanol

NAPLs	Recovery rate (%)	
	Washing liquid with APG and <i>n</i> -butanol	Distilled water
<i>n</i> -Hexane	98.7	2.6
Toluene	96.8	0.90
<i>o</i> -Dichlorobenzene	94.3	0.92
Carbon tetrachloride	95.5	1.06
Methylene chloride	94.8	1.21

^aThe concentrations of APG and *n*-butanol for different NAPLs were consistent with those at fish tails (β_E, ε_E).

molecular weight. The smaller the ratio, the smaller the amount of surfactant and, larger the amount of the alcohol on the amphiphilic layer.

For APG based microemulsion used in SEAR, the washing liquids can be prepared according to the component of the single phase microemulsion (β_E, ε_E), that is, the ε - β fishlike phase diagram is a very convenient method in the utilization of microemulsion.

In order to demonstrate the practical use of the APG based microemulsions in SEAR, the contaminated soils were washed by the washing liquids. The washing liquids were prepared by dissolving certain amounts of APG and *n*-butanol in distilled water, according to the component of the single phase microemulsion (β_E, ε_E) for different NAPLs. The soils contaminated with various NAPLs were added into the washing liquids and shaken in the washing liquid. Each soil sample was 5 g. After 2 weeks at 25 °C, the liquid was removed and analyzed by GC method. Control experiments, in which the washing liquid was distilled water and not APG and *n*-butanol solutions, are also performed. The recovery rates of NAPLs are listed in Table 2, which demonstrates the feasibility of APG based middle-phase microemulsion in SEAR.

The utilization of the APG based middle-phase microemulsion method can reduce the required amount of surfactant and cosurfactant in preparing the washing liquids and the emission of waste water. More importantly, APG is biodegradable and renewable, therefore, the APG based microemulsion method is an important method in SEAR.

In the present work, solubilization power and the composition of *n*-dodecyl polyglucoside based microemulsions have been studied with the ε - β fishlike phase diagram. The order of the solubilization power is: methylene chloride $\approx$ carbon tetrachloride

≈ *o*-dichlorobenzene > toluene > *n*-hexane. The *n*-dodecyl polyglucoside molecules are mainly incorporated into the interfacial layer. However, the *n*-butanol molecules dissolve in the aqueous or NAPLs phase, besides entering into the interfacial layer. The *n*-dodecyl polyglucoside based microemulsion shows high efficiency in remediation of NAPLs contaminated soil.

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