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Separation Science and Technology
Publication details, including instructions for authors and subscription information:
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To cite this Article Weschayanwiwat, Punjaporn , Krutlert, Duanghathai and Scamehorn, John F.(2009) 'Effect of Electrolyte and Temperature on Volatile Organic Compounds Removal from Wastewater Using Aqueous Surfactant Two-Phase System of Cationic and Anionic Surfactant Mixtures', Separation Science and Technology, 44: 11, 2582 — 2597

To link to this Article: DOI: 10.1080/01496390903012130

URL: http://dx.doi.org/10.1080/01496390903012130

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Separation Science and Technology, 44: 2582–2597, 2009
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ISSN: 0149-6395 print/1520-5754 online
DOI: 10.1080/01496390903012130

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Effect of Electrolyte and Temperature on Volatile Organic Compounds Removal from Wastewater Using Aqueous Surfactant Two-Phase System of Cationic and Anionic Surfactant Mixtures

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Abstract: Benzene, toluene, ethylbenzene, and xylene are frequently observed contaminants in industrial wastewaters causing concerns about environmental and health effects. An aqueous surfactant two-phase (ASTP) extraction system using mixtures of cationic and anionic surfactants have been shown to be a promising surfactant-based separation technique to concentrate solutes such as proteins and dyes from aqueous solution. A phase separation of a surfactant solution occurs at certain surfactant compositions and concentrations, forming two isotropic phases. One is rich in surfactant aggregates (surfactant-rich phase) and the other is lean in surfactant aggregates (surfactant-dilute phase). Most of the organic contaminants tend to solubilize and concentrate in the surfactant-rich phase, leaving the surfactant-dilute phase containing only small amounts of contaminants as remediated water. The effect of NaCl addition on the critical micelle concentration (CMC) and the extraction ability of ASTP formed by mixtures of cationic surfactant (dodecyltrimethylammonium bromide; DTAB) and anionic surfactant (alkyl diphenyloxide disulfonate; DPDS) at 50 mM total surfactant concentration with a 2:1 molar ratio of DTAB:DPDS was investigated; the CMC of the mixture slightly decreases with increasing NaCl concentration. The extraction and preconcentration of benzene are greatly enhanced by added NaCl.

Received 22 May 2008; accepted 9 March 2009.

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The higher the degree of hydrophobicity of contaminants, the greater the extraction into the surfactant-rich phases. At 1.0 M NaCl addition, about 95% of xylene, 92% of ethylbenzene, 90% of toluene, and 79% of benzene are extracted into the surfactant-rich phase within a single stage extraction and the contaminant partition ratios can be as high as 395 for xylene, 273 for ethylbenzene, 206 for toluene, and 84 for benzene, which are greater than those obtained from the conventional ASTP extraction system using nonionic surfactants.

Keywords: Anionic surfactant, aqueous surfactant two-phase system, BTEX, cationic surfactant, extraction

INTRODUCTION

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Benzene, toluene, ethylbenzene, and xylene (BTEX) are volatile organic compounds (VOCs) and regulated as priority pollutants by the U.S. Environmental Protection Agency (EPA). They are commonly found in petroleum products such as gasoline, diesel fuel, and crude oil. The main sources of contamination result from spillage of fuels, solvents, or chemicals, causing several concerns including a direct health threat to humans and contribution to the formation of photochemical oxidants and ozone in the ambient air (1). These environmental concerns have led to U.S. State and Federal requirements to control VOC emissions from industries and at municipal wastewater treatment facilities. The average influent BTEX concentrations from 8 wastewater samples from Thai industry have been reported in the range of 15–45 ppm (2).

There are several procedures, which have been used to remove BTEX from wastewater such as carbon adsorption, steam/air stripping, biodegradation, and thermal destruction. Although these methods are highly effective, they are likely to suffer from limitations including recovery efficiency, volume of chemicals required, processing time, energy consumption, safety, and ease of use (3). The aqueous surfactant two-phase system (ASTP) is a promising surfactant-based separation technique used to extract and pre-concentrate organic pollutants of environmental concern. The phase separation of the surfactant solution into two isotropic aqueous phases is induced. One phase is lean in surfactant and called the surfactant-dilute phase. The other phase contains most of the surfactant in a concentrated form and is called the surfactant-rich phase or coacervate phase. The organic solutes tend to solubilize into the surfactant aggregates and accumulate in the surfactant-rich phase where they are predominantly present. The surfactant-dilute phase contains low pollutant concentration and can be treated water. The ratio of surfactant and solubilized organic solutes concentration in the surfactant-rich phase to those in the surfactant-dilute phase, called the partition ratio or distribution ratio, is very high (3–6).

A nonionic surfactant has been widely used in the ASTP extraction in a separation known as a cloud point extraction, where the temperature of the nonionic surfactant solution needs to be raised above the lower critical temperature (LCT) or cloud point of the surfactant to induce the phase separation. Beyond this critical temperature, the solution becomes turbid and separates into two isotropic phases, which are a nonionic micellar rich phase and a micellar-dilute phase (3). Thus, the operating temperature plays a critical role in governing the phase separation and distribution of organic solutes between phases (3,4,6,7). The higher the temperature above the cloud point, the greater the partition ratio and the extraction efficiency (4,6,8). This technique shows great potential for organic solutes separation from aqueous stream since it applies an environmentally-friendly surfactant with relatively low toxicity and flammability as the separating agent as compared to some conventional extraction techniques which employ toxic organic solvents. In addition, the surfactant concentration in the effluent (surfactant-dilute solution) is extremely low (on the order of 100 ppm for the system used here) because more than 99% of total surfactants are in the surfactant-rich solution. Thus, after the discharged effluent is mixed and diluted by water in the water body, the concentrations of these surfactants become negligible. The surfactants in the surfactant-rich solution have potential to be recovered for reuse (9,10) or the solution can be further treated or disposed of.

Mixtures of cationic and anionic surfactants can have superior properties in many applications compared to a single surfactant. For example, cationic and anionic surfactant mixtures often exhibit synergism in micelle formation as illustrated by a much lower critical micelle concentration (CMC) than the single surfactants involved. In addition, the cationic-anionic surfactant aggregates are more tightly packed than other types of aggregates due to the reduction in the repulsion force between oppositely charged head groups resulting in a smaller effective head group area (11). Therefore, surfactant aggregates called vesicles often form (12–16) where the packing of surfactant monomers is tightened, providing more preferable solubilization sites for organic solutes (17).

Mixtures of cationic and anionic surfactants can also phase separate, forming ASTP system at some specific surfactant compositions and concentrations, and have been applied for dyes concentration and protein extraction (18–21). The organic solutes can be simultaneously extracted and pre-concentrated in the concentrated solution of the surfactant-rich phase, resulting in a great reduction of the amount of waste requiring further treatment or disposal. The aggregates in the separated phases

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may exist in different forms, i.e., spherical micelles, rodlike micelles, and vesicles, depending on the surfactant concentration and molar composition.

The addition of some electrolytes can improve the performance of ASTP extractions for nonionic surfactant systems, increasing the fraction of solute extracted into the surfactant-rich phase (4,6,8). The effects of added electrolyte on the ASTP systems formed by mixtures of cationic and anionic surfactants have also been investigated (14,22,23). Nan et al. (23) studied the ASTP of cetyltrimethylammonium bromide (CTAB)/sodium dodecyl sulfonate (AS) and found that the phase separation occurs in the cationic-rich region and the anionic-rich region, but not at the neutral or stoichiometric surfactant composition. They reported that the addition of KCl into mixtures of CTAB/AS can enlarge the phase separation region. The same trend was also observed in the system of CTAB and sodium octyl sulfate (SOS) upon the addition of sodium bromide (NaBr) where the added electrolyte can promote the screening between charged head groups leading to a smaller effective area per head groups and aggregate structures becoming larger and flatter (22,24).

Therefore, this research aimed to apply the ASTP extraction technique using mixtures of cationic and anionic surfactants to remove BTEX from wastewater. The effects of operating temperature and added electrolyte concentration on the extraction ability were investigated.

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MATERIALS AND METHODS

A commercial grade twin-head monoalkyl diphenyloxide disulfonate (DPDS with trade name DOWFAX 8390) with 35% active obtained from Dow Chemical Company (USA) was used as an anionic surfactant. The DPDS is a surfactant comprised of mixtures of mono- and di-alkyl diphenyloxide disulfonates and alkyl diphenyloxide mono- and di-sulfonates. Rouse et al. (25) reported that 80% of the surfactant by weight is monoalkyldiphenyloxide disulfonate with a twin-head and a single tail. In this work, the same batch of DPDS was used throughout the work, although there are no general concerns about inconsistency in properties from batch to batch. Dodecyltrimethylammonium bromide (DTAB) with 99% purity purchased from Nanjing Robiot Company Limited (China) was used as a cationic surfactant. The molecular weights and oral LD50s for rats reported by the suppliers are 308.3 g/mol and 200–1,000 mg/kg, respectively for DTAB; and 642 g/mol and more than 5,000 mg/kg, respectively for DPDS. Benzene, toluene, ethylbenzene, and xylene (mixture of isomers) with purity higher than 99% were used as organic contaminants purchased from Carlo Erba Reagent (Italy), Carlo Erba Reagent (Italy), Acros Oganics (Belgium), and Scharlau Chemie (Spain), respectively. Sodium chloride (NaCl) with purity higher than 99% was used as the electrolyte purchased from Ajax chemical (Australia). All chemicals were used as received. Distilled and deionized water were used throughout the experiments.

The experimental procedure and sample analysis were adopted from a previous study (23) and can be summarized as follows: aqueous solutions containing cationic surfactant, anionic surfactant, and organic contaminant were prepared in a series of 22 mL crimped vials at full occupation at a total surfactant concentration of 50 mM at a 2:1 molar ratio of DTAB:DPDS and used as a base condition in this study. One pollutant was studied at a time and the concentration of pollutant was kept constant at 100 ppm because it has been reported by Kimchuwanit et al. (8) that the initial concentration of pollutant at low solute/surfactant molar ratio has no significant effect on the extraction efficiency. In addition, these BTEX concentrations are realistic levels for actual wastewaters from the petrochemical industry (2). The electrolyte was added into the solution at the desired concentration. The solutions were then homogenized using a magnetic stirrer for 15 min prior to transferring into several crimped vials with teflon coated rubber septa. The vials were equilibrated in an isothermal water bath at the designated temperature for 4 days until reaching an equilibrium condition. The volume of each phase was deduced from the measured solution height. The concentrations of surfactants and organic contaminant in both surfactant-rich and surfactant-dilute phases were analyzed using analytical methods and instruments detailed elsewhere (26). All experiments were done in triplicate. Material balances on DPDS and BTEX ensure that volatilization loss is negligible. The surface tensions were measured by a Wilhelmy plate tensiometer (DataPhysics; Model DCAT 11). The CMC values in the presence and absence of electrolyte were determined from the discontinuity in the slope of surface tension versus logarithm of surfactant concentration (27).

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RESULT AND DISCUSSION

Preliminary Study on CMC Determination and Phase Behavior

As shown in Fig. 1, the CMC of a mixture of DTAB:DPDS at molar ratio of 2:1 slightly decreases upon an addition of NaCl. At 1.0 M NaCl, the CMC is 0.017 mM as compared to 0.024 mM without electrolyte addition. To put this in perspective, the CMC of typical cationic or

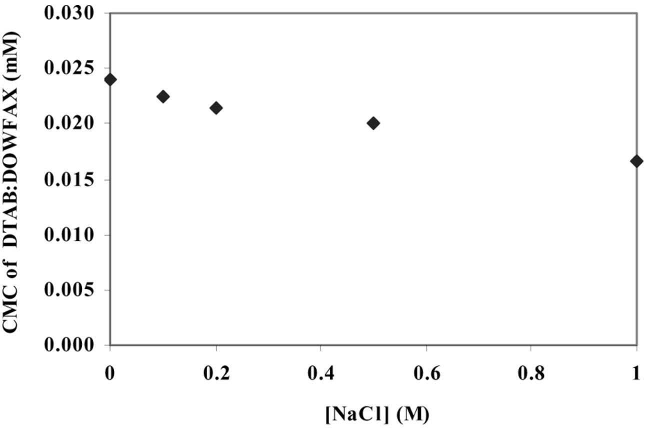

Figure 1. CMC of DTAB/DPDS solution as a function of NaCl concentration (System: 2:1 molar ratio of DTAB:DPDS, and temperature at 30°C).

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anionic surfactants as single components would decrease by one or two orders of magnitude with this salinity increase, whereas a nonionic surfactant only changes slightly with NaCl concentration. The effect of NaCl on the CMC implies that the mixed cationic-anionic surfactant aggregates are net neutrally or nearly neutrally charged. Since the anionic surfactant is a complex mixture of mono- and di-sulfonates, as well as mono- and di-alkyl surfactant, the stoichiometry is probably not exact at a DTAB:DPDS molar ratio of 2:1. So, the aggregates might have a small positive charge.

The cationic-anionic surfactant mixtures behave as pseudo-nonionic surfactants having the minimum cloud point around the neutralized surfactant molar ratio (28). The cloud points of cationic-anionic surfactant mixtures are very dependent upon the surfactant mixture composition, total surfactant concentration, and the presence of electrolyte; meanwhile the cloud point of nonionic surfactant greatly depends on the presence of electrolyte.

Effect of Temperature on the ASTP Extraction

There is a lack of literature studies on the LCT and upper consolute temperature (UCT) of mixtures of DTAB and DPDS. From a phase behavior study at a 2:1 molar ratio of DTAB:DPDS, the cloud point (above which two liquid phases coexist at equilibrium) is less than 4°C. The two phases are present between 4°C and 100°C, so the UCT at which the solution becomes one phase again is greater than 100°C. Figures 2a and 2b show the surfactant concentration and the benzene concentration in each of the two phases as a function of temperature, respectively. The effect of temperature on the partition ratio of the surfactant and of benzene is shown in Fig. 3a, where the partition ratio is defined as the ratio of the chemical concentration in the surfactant-rich phase to that in the surfactant-dilute phase. Figure 3b shows the fractional phase volume of the surfactant-rich phase and the fractional benzene removal as a function of temperature.

From Fig. 3b, only about 4.8% of the phase volume is occupied by the surfactant-rich phase. Since almost all the surfactant is present in the surfactant-rich phase, the surfactant concentration in this phase increases by a factor of about 20 from that in the feed (from 50 mM to

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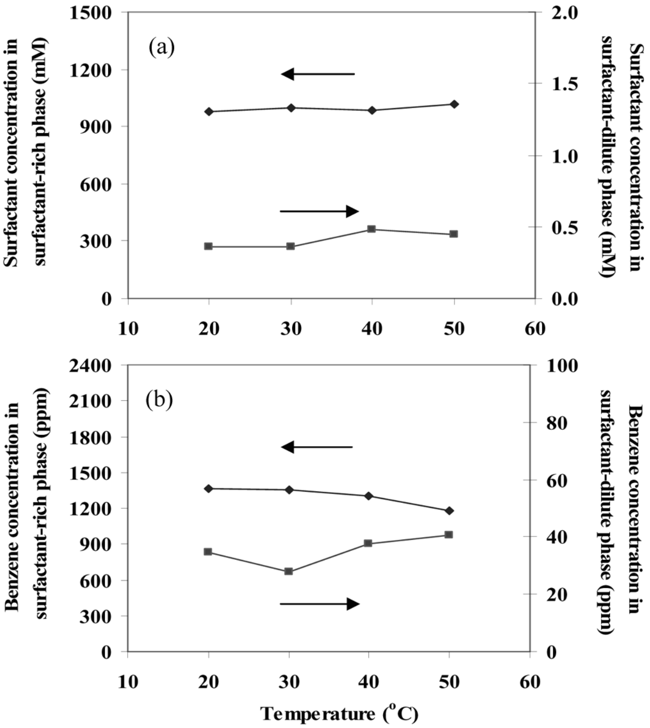

Figure 2. (a) Surfactant concentrations in the surfactant-rich and the surfactant-dilute phase; and (b) benzene concentrations in the surfactant-rich and the surfactant-dilute phase as a function of temperature (System: 2:1 molar ratio of DTAB:DPDS, 50 mM total surfactant concentration, 100 ppm benzene concentration, pH = 7 without electrolyte addition).

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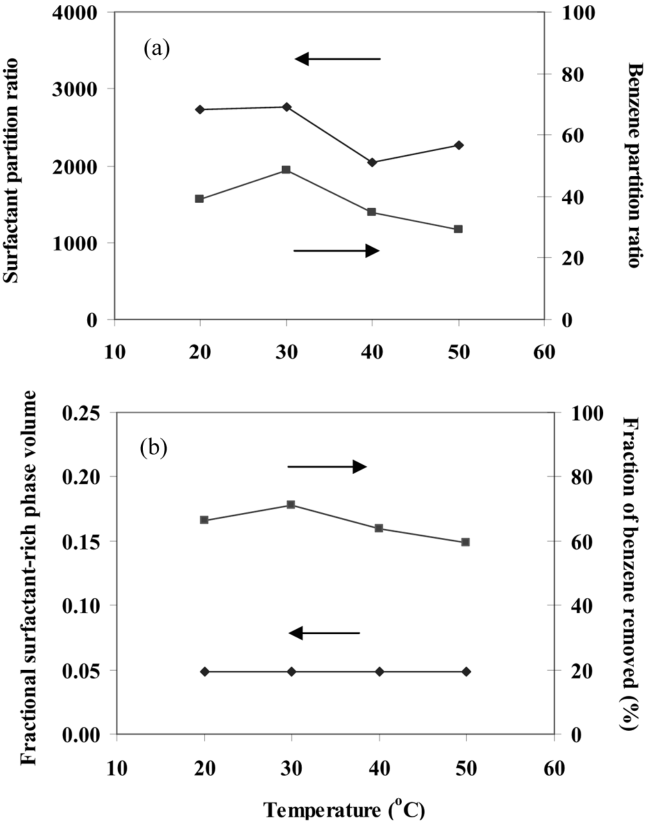

Figure 3. (a) Partition ratios of surfactant and benzene; and (b) fractional surfactant-rich volume and fraction of benzene removed (%) as a function of temperature (System: 2:1 molar ratio of DTAB:DPDS, 50 mM total surfactant concentration, 100 ppm benzene concentration, pH = 7 without electrolyte addition).

1,000 mM) as seen in Fig. 2a. Since the surfactant concentration in the dilute phase is quite low as shown in Fig. 2b, the surfactant partition ratio is very high (2,700 at 30°C). The fractional phase volume of the surfactant-rich phase was found to be 0.2 at 30°C and 0.08 at 50°C for a nonionic surfactant ASTP system (4) compared to 0.048 here. Since this stream is anticipated to be further processed (such as vacuum stripped) to separate pollutants from the surfactant solution to permit recycle of surfactant, this reduction in volume is economically valuable.

From Fig. 3a, the benzene partition ratio varies from 29 to 48, compared to a maximum value of 25 for nonionic surfactant (4), so the cationic-anionic surfactant mixture gives a better separation than the nonionic surfactant. From Fig. 3b, up to 71% of the benzene can be recovered in this single stage. The process can be properly scaled up to a multistage, continuous extractor (5,6,29) which can be used to treat a large volume of wastewater and achieve any desired removal of the benzene by adjusting the number of stages or transfer units.

The temperature does not have as much effect on the separation for a cationic-anionic surfactant mixture compared to a nonionic surfactant. For example, over a 30°C range, the partition ratio varies from 29 to 48 in this work, compared to 10 to 25 for nonionic surfactant. The analogous ranges for the surfactant partition ratio are 2000 to 2800 in this work and about 250 to 2300 for a nonionic surfactant. This low temperature dependence makes ASTP extraction using cationic-anionic surfactant mixtures more robust than when using nonionic surfactants.

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Effect of Electrolyte on the ASTP Extraction

The effect of NaCl addition on the ASTP extraction of cationic-anionic surfactant mixture for benzene removal was investigated at 30°C. Upon the addition of NaCl, the position of phases inverts due to density changes and the surfactant-rich phase is the top phase. As shown in Fig. 4a, the surfactant concentration in the surfactant-dilute phase at 0.2 M NaCl is obviously higher than those in an absence of NaCl and at other NaCl concentrations. Due to the fact that the addition of NaCl can change the phase behavior of the separated phases, i.e., invert phase positions, it is possible that when a small amount of electrolyte is added into the system, the size and shape of surfactant aggregates in the surfactant-rich phase are altered, resulting in a lower density of the surfactant-rich phase. At 0.2 M NaCl, the concentration of surfactant in the surfactant-dilute phase is very high and gradually declines with increasing NaCl concentration.

The surfactant concentrations in both phases at the highest NaCl concentration studied here (1 M) and those of in the absence of NaCl are comparable as shown in Fig. 4a. Therefore, the surfactant partition ratio, which is the ratio of surfactant concentration in the surfactant-rich phase to that of in the surfactant-dilute phase, remains almost constant upon the addition of NaCl except at 0.2 M NaCl where the concentration of the surfactant in the dilute phase is very high. These results are not in agreement with ASTP extraction of nonionic surfactants where the addition of NaCl can decrease the cloud point of the nonionic surfactant solution due to the salting-out effect, resulting in a greater temperature difference between the operating temperature and the cloud point. Thus, the surfactant partition ratio typically increases with increasing NaCl

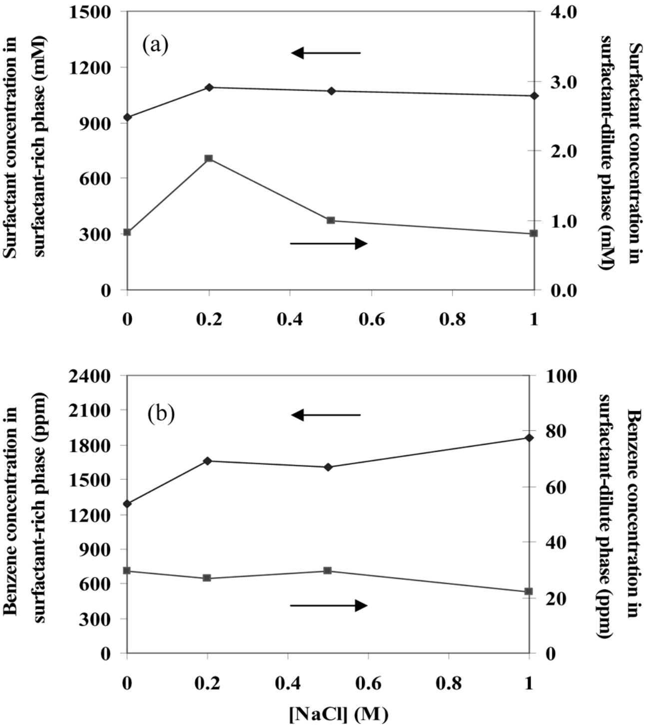

Figure 4. (a) Surfactant concentrations in the surfactant-rich and the surfactant-dilute phase; and (b) benzene concentrations in the surfactant-rich and the surfactant-dilute phase as a function of NaCl concentration (System: 2:1 molar ratio of DTAB:DPDS, 50 mM total surfactant concentration, 100 ppm benzene concentration, and temperature at 30°C).

concentration at constant temperature due to a dramatic increase in the surfactant concentration in the surfactant-rich phase and a decrease in the surfactant concentration in the surfactant-dilute phase (4,6).

Although the surfactant concentration in both phases are not as much affected by the addition of NaCl, the benzene concentration in the surfactant-rich phase increases while that of in the dilute phase slightly decreases as shown in Fig. 4b. The benzene partition ratio, which is the ratio of benzene concentration in the surfactant-rich phase to that of in the surfactant-dilute phase increases with the addition of NaCl and can be as high as 84 or approximately doubled upon the addition of 1.0 M NaCl as shown in Fig. 5a. It has been reported that the addition of electrolyte can increase the aggregation number of micelles causing a transformation of small micelles into larger and flatter structure, resulting in an

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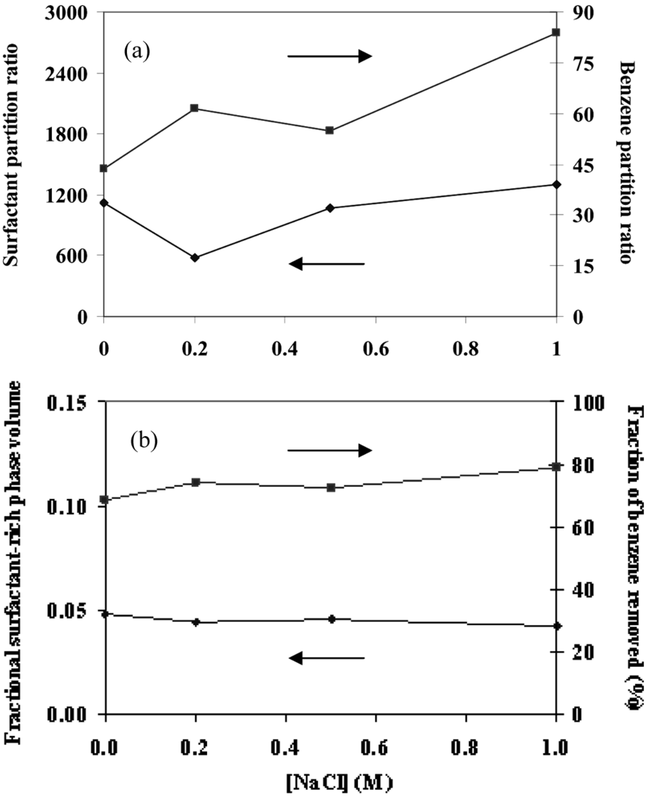

Figure 5. (a) Partition ratios of surfactant and benzene; and (b) fractional surfactant-rich volume and fraction of benzene removed (%) as a function of NaCl concentration (System: 2:1 molar ratio of DTAB:DPDS, 50 mM total surfactant concentration, 100 ppm benzene concentration, and temperature at 30°C).

extension of solute solubilization (30). Thus, the solubilization of organic solutes into the surfactant aggregates is enhanced. An increase in the benzene partition ratio is almost entirely due to an increase in the solubilization capacity of the surfactant aggregates in the surfactant-rich phase upon the addition of NaCl. The contribution from an increase in the surfactant concentration in the surfactant-rich phase and a decline of the surfactant concentration in the surfactant-dilute phase to the increased benzene partition ratio is very slight. Figure 5b shows the fractional surfactant-rich phase volume and the fractional benzene removed as a function of NaCl concentration. The fractional surfactant-rich phase volume slightly decreases upon the addition of NaCl. At 1.0 M NaCl, 79% of benzene is extracted into the surfactant-rich phase, which is 10% greater than without electrolyte

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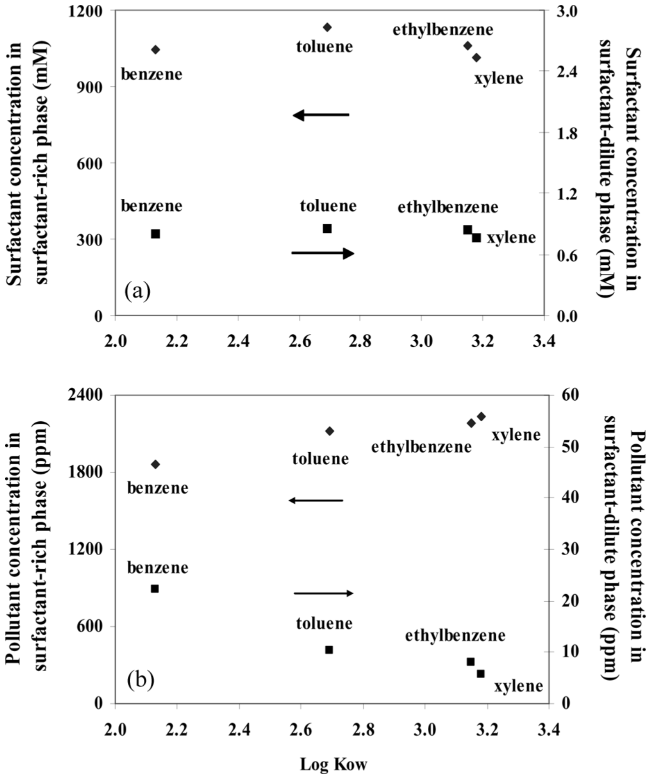

Figure 6. (a) Surfactant concentrations in the surfactant-rich and the surfactant-dilute phase; and (b) pollutant concentrations in the surfactant-rich and the surfactant-dilute phase as a function of log Kow (System: 2:1 molar ratio of DTAB:DPDS, 50 mM total surfactant concentration, 100 ppm pollutant concentration, 1.0 mM NaCl, and temperature at 30°C).

addition. Therefore, the presence of NaCl in the wastewater stream does not hinder the ASTP extraction process but enhance the pre-concentration and separation ability.

Effect of Degree of Hydrophobicity on the ASTP Extraction

The ASTP extraction system was applied to remove the volatile aromatic pollutants with a greater degree of hydrophobicity than benzene such as toluene, ethylbenzene, and mixed-xylene. A salinity of 1.0 M NaCl was used since a high benzene partition ratio was observed under this condition from Fig. 5a. The degree of hydrophobicity of these aromatic compounds can be represented by the $\log_{10}$ of the octanol-water partition coefficients (log Kow): 2.13 for benzene, 2.69 for toluene, 3.15 for ethylbenzene, and 3.12–3.20 for mixed-xylene as reported in the technical factsheet of U.S. EPA (31).

As shown in Fig. 6a, the surfactant concentrations in the surfactant-rich and the surfactant-dilute phases is not much affected by a different type of pollutants. However, the greater the degree of hydrophobicity of the pollutant (or higher log Kow), the greater the pollutant concentration in the surfactant-rich phase and lesser pollutant concentration in the surfactant-dilute phase as shown in Fig. 6b. Thus, the pollutant partition ratio increases regularly for this homologous series as log Kow increases as shown in Fig. 7a, even though the fractional

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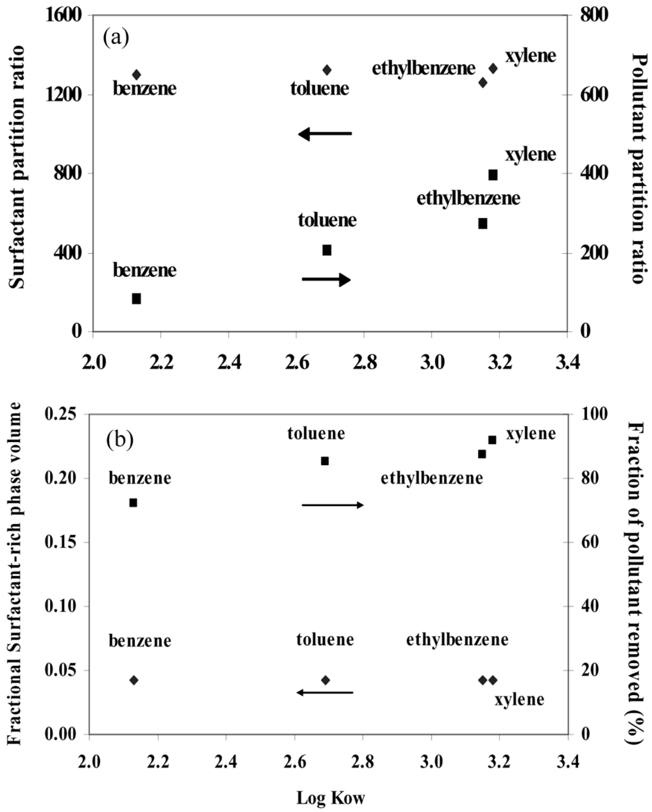

Figure 7. (a) Partition ratios of surfactant and pollutants; and (b) fractional surfactant-rich volume and fraction of pollutant removed (%) as a function of log Kow (System: 2:1 molar ratio of DTAB:DPDS, 50 mM total surfactant concentration, 100 ppm pollutant concentration, 1.0 mM NaCl, and temperature at 30°C).

surfactant-rich phase volume remains almost unaffected by a different type of pollutants as shown in Fig. 7b.

An increase in the pollutant partition ratio with log Kow of solutes in this cationic-anionic surfactant system is mainly due to an increase in the solubilization ability of solutes into the surfactant aggregates themselves, not from an altering of the surfactant concentrations in the surfactant-rich phase or in the surfactant-dilute phase since they remain almost constant or independent of pollutant structure. It has been reported that the solubilization in micelles correlates to Kow (32,33), but it does not necessarily correlate to structures outside of a homologous series.

Among these aromatic pollutants, xylene shows the highest partition ratio, followed by ethylbenzene, toluene, and benzene, respectively, which is consistent with their degree of hydrophobicity; this is in agreement with results from cloud point extraction using nonionic surfactants (4,6). Xylene and ethylbenzene have the highest degree of hydrophobicity or least water solubility, thus most prefer partitioning into the surfactant aggregates. However, ethylbenzene has a larger molecular structure as compared to xylene resulting in slightly less ability to partition into the surfactant aggregates in the surfactant-rich phase due to steric effects.

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ACKNOWLEDGEMENTS

This research was funded by the Thailand Research Fund (TRF) and the Commission on Higher Education (Grant ID 4880047). In addition, support was received from the industrial sponsors of the Institute for Applied Surfactant Research including Akzo Nobel, Clorox, Conoco/Phillips, Church and Dwight, Dow, Ecolab, Halliburton, Huntsman, Oxiteno, Proctor & Gamble, Sasol, SC Johnson, and Shell. Dr. Scamehorn holds the Asahi Glass Chair in the School of Chemical, Biological, and Materials Engineering at the University of Oklahoma.

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