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REMOVAL OF VOLATILE AROMATIC CONTAMINANTS FROM WASTEWATER BY CLOUD POINT EXTRACTION

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ABSTRACT

Removal of the aromatic contaminants benzene, toluene, and ethylbenzene from wastewater was investigated using cloud point extraction (CPE). A nonionic surfactant, *t*-octylphenolpolyethoxylate, was utilized as the separating agent. When the nonionic surfactant solution is heated above the cloud point temperature, phase separation is induced. The micellar-rich phase or coacervate phase and the micellar-dilute phase are formed. The aromatic contaminants tend to solubilize into the micelles and concentrate in the coacervate phase. The concentration of the solutes in the coacervate increases as temperature, added electrolyte concentration, and degree of alkylation of the aromatic solutes increase.

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INTRODUCTION

A novel class of separation processes utilizing a surface-active agent are known as surfactant-based separations (1,2). These are used increasingly in process engineering (3). Processes such as froth flotation and micellar-enhanced ultrafiltration can be effective in environmental clean up (1,4). One surfactant-based separation of interest is cloud point extraction (CPE), which has been shown to be an effective technique to remove dissolved organic contaminants from water. This research focuses on cleaning up wastewater containing volatile aromatic pollutants benzene, toluene, and ethylbenzene, which can originate from gasoline tank leakage.

From an economic perspective, the surfactants, which serve as solvents in the extraction processes, have to be recovered. Since, these aromatic solutes have high volatility, they can be released from the surfactant solution by vacuum stripping, leaving a solute-free surfactant stream available for reuse (5–7). There have been literature studies of less volatile compounds using CPE such as phenolics (8–10). While these compounds can show excellent separation efficiency, there is no demonstrated efficient way to separate the solute from surfactant for surfactant reuse. In addition, as it is quite difficult to study experimentally the types of systems used here due to loss of solute by volatilization, previous investigations have tended to avoid these contaminants despite their importance.

BACKGROUND

Cloud point extraction is a separation technology using the benign polyethoxylate nonionic surfactant as a separating agent (8–19). It has been shown to be an alternative to traditional liquid–liquid extraction because of efficiency, cost effectiveness, and environmental friendliness without any usage of toxic and flammable organic solvents (11,16). This CPE is a specific example of aqueous biphasic extractions (20). When the aqueous nonionic surfactant solution is at a temperature higher than a certain temperature known as cloud point, phase separation is induced, forming two isotropic aqueous phases (8–19,21,22). The phase rich in surfactant micelles is called micellar-rich phase or coacervate phase. The other phase, which is lean in surfactant micelles and has the concentration of surfactant approximately 2–20 times the critical micelle concentration (CMC), is called dilute phase. As the phase separation is reversible, both phases can merge into a single phase upon cooling (11). Dissolved organic solutes will tend to solubilize in surfactant aggregates like micelles and thus concentrate in the coacervate phase, which contains surfactant in concentrated form. The cloud point temperature is sometimes defined at a surfactant concentration of 1 wt% (23), but is not highly concentration dependent (13,22–24). The minimum cloud point occurs at the lower consolute temperature or lower critical temperature (LCT) at the critical surfactant concentration (22).

To accomplish the phase separation, the temperature of the nonionic surfactant solution must be above the cloud point. The total surfactant concentration must be above the surfactant concentration existing in the dilute phase above the cloud point. Either the solution can be heated or the cloud point of nonionic surfactant reduced below the operating temperature. The CPE can be a low-energy separation process since a surfactant can be chosen with a cloud point below the wastewater operating temperature. Lowering the degree of polymerization of ethylene oxide or lengthening the hydrocarbon chain of the hydrophobic moiety of the nonionic surfactant can depress the cloud point (11,25,26). The addition of polar organic solutes, such as fatty acids, aliphatic alcohols, and phenol, generally lowers the cloud point (27). Added electrolyte can affect the cloud point with some anions, such as chloride, sulfate, and carbonate, depressing the cloud point due to the salting-out effect (24,28). On the other hand, some ions, such as thiocyanate, iodides, and nitrates increase the cloud point due to the salting-in effect (29). Adding anionic surfactant increases the cloud point (24,30,31). The effect of electrolytes on the cloud point of a pure nonionic surfactant and a mixed ionic–nonionic surfactant system has been discussed in the literature (24,30,32).

Studies of both the microstructure and macroscopic thermodynamic properties of the coacervate have given insight into its nature. Hoffmann et al. (33) studied the kinetics of aqueous nonionic surfactant solutions at the cloud point and found the formation of a new phase at a temperature higher than the cloud point. They stated that the existence of the new phase is controlled by nucleation phenomenon. Turro and Kao (34) proved the presence of micelles in that phase by using three types of fluorescence probes as the indicator. Kato et al. (35,36) studied the microstructure of nonionic surfactant in semidilute solutions of nonionic surfactant including a system at a temperature higher than the cloud point via various techniques. They proposed that below the cloud point, the micelles form entangled networks. When temperature increases, the extent of cross-linking increases, forming the multiconnected network as determined by the self-diffusion technique (36). The comparison between the solubilization of organic solute into surfactant aggregates in a coacervate phase and the solubilization into surfactant micelles showed that the thermodynamic solubilization equilibrium constant for each of the aggregates is similar for similar surfactants and solutes (15). The nonideality of mixing of anionic and nonionic surfactants in the coacervate aggregates was shown to be similar to that in micelles existing below the cloud point (31). The last two studies support the hypothesis that the surfactant aggregates in the coacervate are micellelike in structure in that they have a hydrophobic region and a hydrophilic region where head groups interact in a similar fashion as normal micelles.

Many researchers have studied the CPE of organic contaminants, but few works have dealt with the volatile organic compounds (VOCs) of great environmental concern (16). We believe that this is due to the experimental difficulty of making accurate measurements on these systems since leakage of these species is difficult to overcome. It is economically worthwhile to study removal of these pollutants from water because these solutes have high vapor pressures, permitting them to be stripped off from the coacervate phase, leaving this phase solute-free for reuse.

EXPERIMENTAL

Materials

A polydisperse commercial, $OP(OE)_7$, with an average of 7 moles of ethylene oxide per mole of octylphenol (trade name Igepal CA-620) contributed by Rhodia (Cranbury, USA) was used as the nonionic surfactant in this study. Reagent grade benzene from Labscan Co, Ltd. (Bangkok, Thailand) with purity of 99.7%, toluene from J. T. Baker (Phillipsburg, USA) with purity of 99.8%, ethylbenzene from Fluka (Buchs, Switzerland) with purity of 98%, and NaCl from AJAX chemical (Auburn, Australia) with purity of 99.9% were purchased. All chemicals were used as received. The water was distilled and deionized.

Methods

A solution, containing nonionic surfactant, aromatic solute, and water with and without added electrolyte, was transferred into several identical vials. To prevent headspace loss, the solution must occupy almost all of the vial volume (22 mL) to neglect vapor volume. The rubber septa coated with polytetrafluoroethylene (PTFE) were used to seal these vials to make sure that no leakage occurred. The vials were placed in an isothermal water bath and the phase separation occurred immediately because of the density difference between the two phases. When the equilibrium was reached, which is defined as the condition where no further change in coacervate volume is observed, the relative phase volumes of each phase were measured by the solution height. The concentrations of nonionic surfactant and aromatic solute in both coacervate and dilute phases were measured.

The concentrations of $OP(EO)_7$ and aromatic solutes were measured by using a CE 2000 series UV-spectrophotometer (Cecil Instrument Limited, Cambridge, UK) at 224 nm and a gas chromatography with a flame ionization detector (Perkin Elmer, Inc., Shelton, USA), respectively. Because of the high volatility of aromatic solutes, static headspace sampling was used as the sample injection technique with no interference of a high molecular weight nonionic surfactant. The conditions used for determination of the aromatic solute concentrations were as follows: column, Supelcowax 10; carrier, ultra-pure nitrogen with the flow rate of 20 mL/min; oven temperature, 100°C isothermal; injector temperature, 150°C; and detector temperature, 250°C. The external standard quantitative calibrations were obtained for the analysis of surfactant and aromatic solutes in both phases. Closure of the material balance is taken as evidence that leakage of the volatile solute is negligible.

RESULTS AND DISCUSSION

In the reported data, surfactant concentrations are reported in m*M*, but solute concentrations are in ppm because wastewater pollutant concentrations are often designated in these weight-based units. There was no significant effect on the extraction due to changes in an initial concentration of organic solute (trichloroethylene) at low solute/surfactant molar ratio, as shown by Kimchuwanit et al. (14). To illustrate the relative magnitude of these concentrations, at our base case of 70 m*M* surfactant and 100 ppm solute initial concentrations. The solute/surfactant molar ratio is 0.0183 for benzene, 0.0155 for toluene, and 0.0135 for ethylbenzene.

Effect of Total Surfactant Concentration on Cloud Point Temperature of Benzene

Although the CPE is surfactant concentration dependent, under the conditions used here (30–110 m*M*), the cloud points of $OP(EO)_7$ are fairly constant at 22°C. The addition of 100 ppm benzene can lower the cloud point by 5°C, as shown in Table 1. Figure 1 shows that as the total surfactant concentration increases, the surfactant concentration in the coacervate phase remains essentially unchanged. The fractional coacervate volume increases with total surfactant concentration, as required from material balance considerations, as shown in Fig. 2. The ratio of surfactant concentration in the coacervate phase to that in the dilute phase (surfactant partition ratio) also remains constant as shown in Fig. 3. In addition, the benzene partition ratio, which is the ratio of benzene concentration in the coacervate phase to that in the dilute phase, is not much affected by increasing total surfactant concentration as shown in Fig. 4. There is a higher concentration of micelles in the coacervate phase, leading to a higher micellar solubilization capacity for aromatic solutes. Hence, the percentage of benzene extracted increases. The CPE of polycyclic aromatic hydrocarbons (PAHs), studied by Sirimanne et al., follows the

Table 1. Cloud Points (°C) of 70 m*M* *t*-Octylphenolpolyethoxylate System

	Solute Concentration (ppm)	
System	0	100
Benzene	22	17
Toluene	22	14
Ethylbenzene	22	11

same trend (17). From Fig. 5, at the lowest temperature studied here (30°C), at the total surfactant concentration of 110 m*M*, 86% of the benzene is extracted into the coacervate phase in a single stage.

Effect of Temperature on Cloud Point Extraction of Benzene

As temperature increases, the system is further away from the cloud point causing the nonionic surfactant micelles to be less water soluble. The dehydration of the hydrophilic polyethoxylate groups in the surfactant increases the inter-surfactant attraction and hence, inter-micellar attraction, which makes the coacervate more concentrated and with lower volume as the temperature is

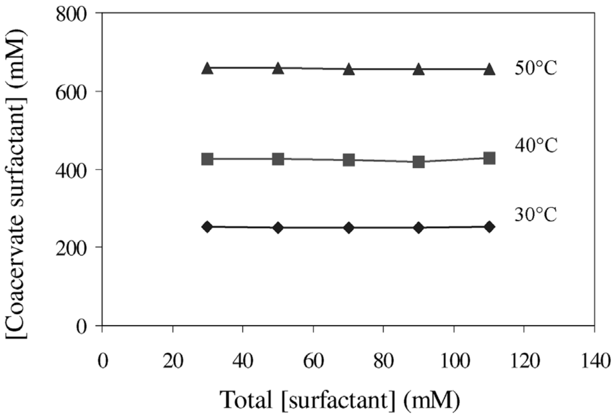

Figure 1. Surfactant concentration in coacervate phase as a function of total surfactant concentration and temperature (system: 100 ppm benzene without added electrolyte).

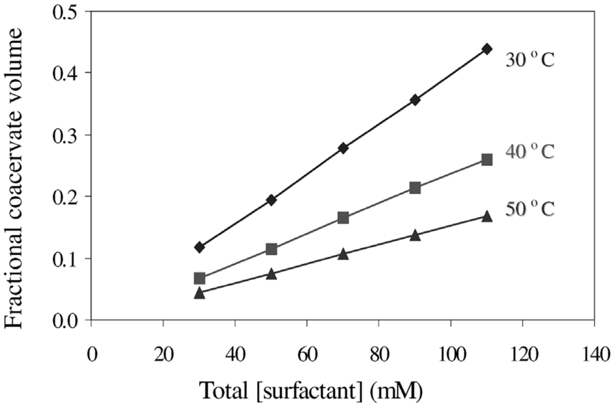

Figure 2. Fractional coacervate volume as a function of total surfactant concentration and temperature (system: 100 ppm benzene without added electrolyte).

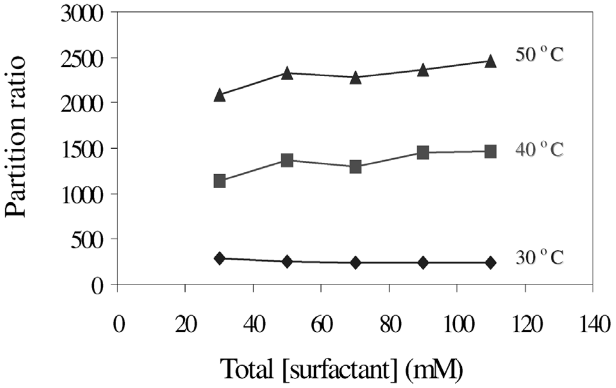

Figure 3. Surfactant partition ratio as a function of total surfactant concentration and temperature (system: 100 ppm benzene without added electrolyte).

increased above the cloud point. As the temperature increases, both surfactant and benzene partition ratio increase substantially, as shown in Figs. 3 and 4, respectively. At 50°C, a surfactant partition ratio exceeding 2000 and a benzene partition ratio exceeding 30 are observed. It is very beneficial to increase the temperature because it gives a very high surfactant partition ratio, which makes surfactant recovery more economical. Nevertheless, there are limitations on increasing temperature. The upper critical temperature can be reached, above which the phase separation does not occur (22). Since raising the operating

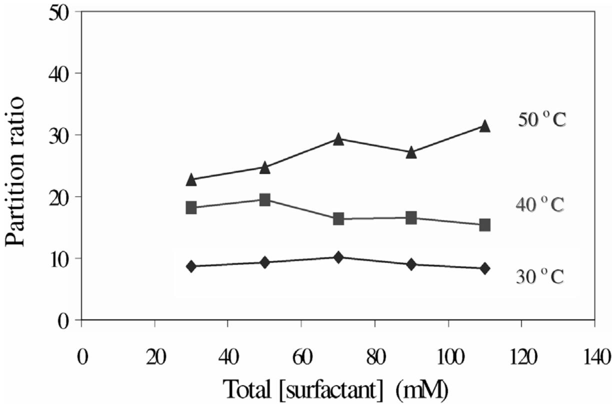

Figure 4. Benzene partition ratio as a function of total surfactant concentration and temperature (system: 100 ppm benzene without added electrolyte).

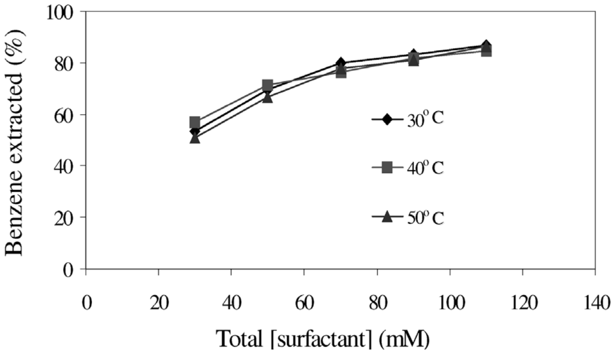

Figure 5. Percentage of benzene extracted in coacervate phase as a function of total surfactant concentration and temperature (system: 100 ppm benzene without added electrolyte).

temperature is energy intensive, the alternative of adjusting surfactant structure and other solution conditions so the cloud point is substantially below the operating temperature is desirable. However, an increase in temperature does not affect the fraction of benzene extracted into the coacervate phase substantially, as shown in Fig. 5. Although the concentration of benzene in the coacervate phase increases substantially as the temperature is raised, the fractional coacervate volume decreases. Therefore, these opposing effects result in the fraction of benzene extracted remaining nearly unchanged. However, higher temperature definitely has advantages in that it results in a lower solute concentration in the dilute phase and a higher solute concentration in the coacervate and a resulting lower coacervate phase volume with reduced processing costs downstream in treatment of the coacervate for surfactant recovery.

Effect of Added Electrolyte on Cloud Point Extraction of Benzene

The addition of NaCl to the micellar solution of OP(EO)$_7$ can depress the cloud point due to the salting-out effect (14,24,31). Therefore, it is analogous to an increase in the operating temperature. It has been reported that the lowering of the cloud point is related directly to an increase in added electrolyte concentration. The effect of electrolyte concentration on benzene partition ratio and fraction of benzene extracted at a total surfactant concentration of 70 m*M* and 30°C is shown in Fig. 6. The result demonstrates that the fractional coacervate volume decreases slightly with increasing salt concentration. The benzene partition ratio substantially increases with increasing NaCl concentration. This added electrolyte effect agrees with previous studies by several groups (12,14). An increase in NaCl concentration up to 0.6 *M* at 30°C can increase the benzene partition ratio a few fold. This salinity effect is approximately equivalent to the effect of a 20°C (from 30 to 50°C) temperature increase while increasing the benzene partition ratio. Nevertheless, the fraction of benzene extracted is not much affected by increasing the NaCl concentration, which is similar to the result shown in Fig. 5, where an increase in operating temperature has little effect on fraction of benzene extracted into the coacervate phase.

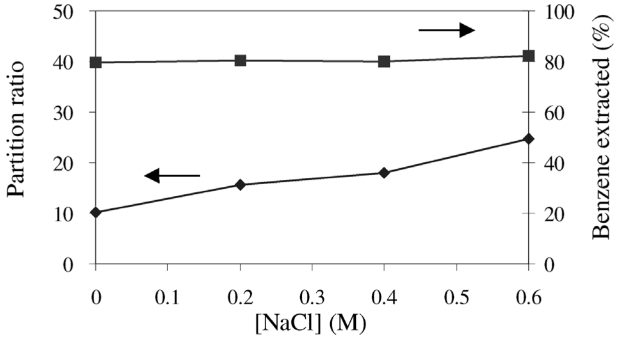

Figure 6. Benzene partition ratio and percentage of benzene extracted in coacervate phase as a function of NaCl concentration (system: 100 ppm benzene, 70 m*M* surfactant, and 30°C).

Effect of Degree of Alkylation of Aromatic Solutes on Cloud Point Extraction

A series of VOC aromatic solutes with varying degrees of alkylation (benzene, toluene, and ethylbenzene) was studied. A higher degree of alkylation of the solutes within a homologous series results in a greater partition ratio, as shown for ethylbenzene, toluene, and benzene in Fig. 7. This is in agreement with the solubilization study of organic solutes in aqueous solutions of nonionic surfactant. The higher the degree of alkylation (or lower the water solubility) of a homologous series of solutes, the higher the solubilization constant generally is (37). A secondary effect is that the addition of ethylbenzene can depress the cloud point of the system more than the other two solutes, as shown in Table 1. Thus, it gives the highest temperature difference between cloud point and operating temperature, which is analogous to increasing the temperature. The same trend with water solubility has been observed in systems where degree of chlorination was varied except at high degrees of chlorination where anomalies are sometimes seen (10,15). In addition, the fraction of aromatic solutes extracted into the coacervate phase depends on the degree of alkylation of the solutes. At the highest operating temperature studied (50°C) and the total surfactant concentration of 70 m*M*, up to 95, 89, and 78% of ethylbenzene, toluene, and benzene are extracted within a single stage, respectively.

Scale-Up of Cloud Point Extraction

Essentially all reported CPEs were carried out in a batch experiment on a laboratory scale (4,8–19). In order for this technology to become commercia-

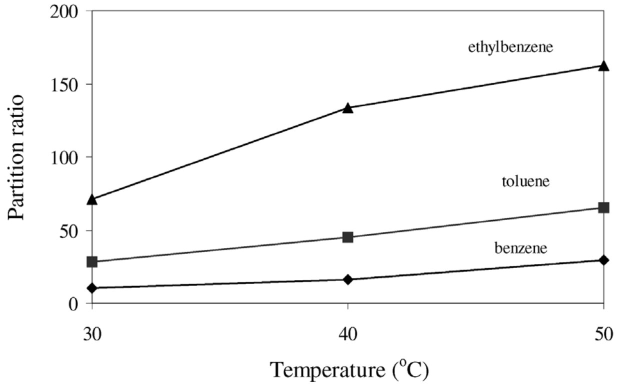

Figure 7. Partition ratio of several aromatic solutes as a function of temperature (system: 100 ppm aromatic solutes, 70 m*M* surfactant without added electrolyte).

lized, scale-up to continuous, multi-stage units will be necessary. In addition, recovery and reuse of the surfactant from the coacervate is crucial for economical operation; hence, the emphasis here is on volatile solutes, which can be stripped away. These two engineering problems are far from trivial; the viscous coacervate phase may cause plugging of extractors or strippers and efficient liquid–liquid contact may be difficult to attain in an extractor due to stickiness and viscous nature of the coacervate phase. Efficient thermodynamic extraction behavior (high partition ratio) is not a sufficient criterion for an efficient integrated separation scheme.

The principles of CPE are analogous to that of a conventional liquid–liquid extraction, except that the solvent can be completely miscible with the feed solution. Figure 8 shows the integrated flow diagram of the multistage CPE process including a surfactant recovery unit. The contaminated feed water and a concentrated surfactant solution are fed to a temperature-controlled extractor where two streams are mixed mechanically at the temperature above the cloud point. As a result, phase separation takes place. The heavy coacervate phase, which contains the majority of the solutes, settles down at the bottom of the extractor as an extract phase due to a density difference. The dilute phase, which

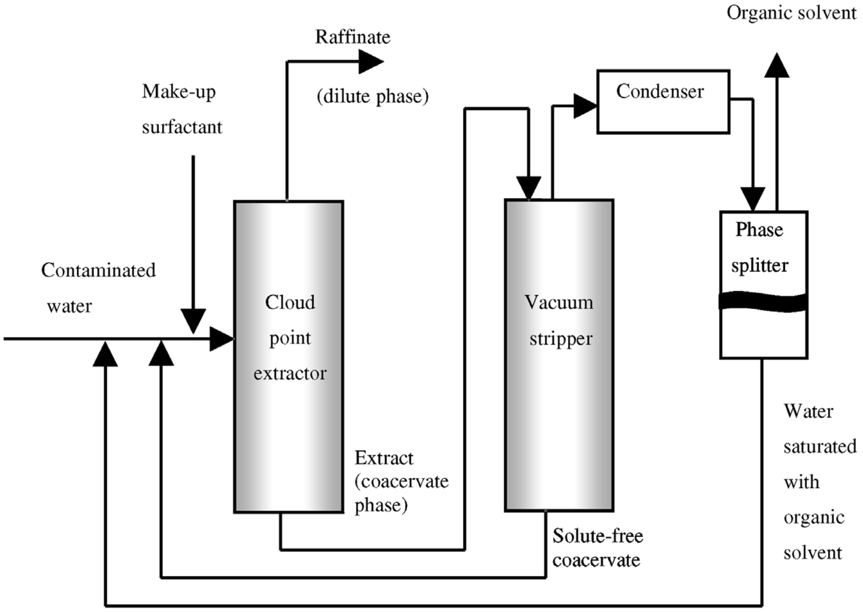

Figure 8. Schematic of integrated process including a multistage cloud point extractor and vacuum stripper.

is lighter, will rise up to the top of the extractor as a raffinate phase, which will be clean enough hopefully to be returned to the environment. Moreover, a vacuum stripper can strip the aromatic solutes, which have high volatility, from the coacervate phase, so that this resulting surfactant-rich phase can be recycled for reuse. Current work includes design, construction, and operation of a continuous, steady-state, multistage trayed liquid–liquid extractor and a continuous, steady-state, packed-column vacuum stripper for scale-up of this process.

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