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Separation Science and Technology

Publication details, including instructions for authors and subscription information:

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Online publication date: 08 July 2010

To cite this Article Trakultamupatam, Punjaborn, Scamehorn, John F. and Osuwan, Somchai(2005) 'Scaling Up Cloud Point Extraction of Aromatic Contaminants from Wastewater in a Continuous Rotating Disk Contactor. II. Effect of Operating Temperature and Added Electrolyte', Separation Science and Technology, 39: 3, 501 – 516

To link to this Article: DOI: 10.1081/SS-120027992

URL: <http://dx.doi.org/10.1081/SS-120027992>

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**Scaling Up Cloud Point Extraction of
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Effect of Operating Temperature and
Added Electrolyte**

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ABSTRACT

When a nonionic surfactant solution is heated above the cloud point, phase separation occurs. Two isotropic phases are formed: a micellar-rich or coacervate phase and a micellar-dilute phase. The organic contaminant dissolved in the wastewater tends to solubilize into the micelles and concentrate in the coacervate phase, resulting in a low concentration of

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contaminant in the dilute phase. In this work, this cloud point extraction (CPE) was scaled up from batch to continuous operation in a multistage rotating disk contactor to remove toluene from wastewater. The surfactant partition ratio, toluene partition ratio, and the concentration of surfactant and toluene in the coacervate phase increase with increasing temperature. The addition of NaCl shows corresponding results to an increase in temperature since NaCl can depress the cloud point. The overall volumetric mass transfer coefficient and the number of transfer unit increase, whereas the height of transfer unit decreases when temperature is raised or NaCl is added to solution.

Key Words: Aromatic contaminants; Cloud point extraction; Rotating disk contactor; Wastewater; Nonionic surfactants.

INTRODUCTION

Cloud point extraction (CPE) has been successfully demonstrated to remove and preconcentrate the organic pollutants from an aqueous wastewater with high separation factors in batch experiments for decades.^[1–11] It occurs when an aqueous solution of nonionic surfactants is heated above a certain temperature known as the cloud point. The solution separates into two isotropic phases: a micellar-rich or coacervate phase and a micellar-dilute phase.^[1–14] The coacervate phase is a viscous phase, which is denser than the dilute phase and can be very concentrated in surfactant, sometimes exceeding 50 wt%. The surfactant concentration in the dilute phase is much more dilute than the coacervate; often approximately 2–20 times the critical micellar concentration (CMC). The pollutants dissolved in the wastewater will solubilize into the surfactant micelles and concentrate in the coacervate phase, leading to a low concentration of organic pollutants presence in the dilute phase.^[8–11] If a single-stage extraction results in insufficient purification, multiple stages can be used as a traditional liquid–liquid extraction, as investigated in this work. The dilute phase, which contains a low concentration of organic pollutants, can be discharged to the environment. If the volatile organic solutes are removed from the coacervate phase by vacuum stripping, the solute-free coacervate stream is available for reuse. When the organic solutes are nonvolatile organic compounds or in low-volume applications where regeneration of the coacervate is not worthwhile, the coacervate phase, which contains a high solute concentration in small volume, can be disposed or incinerated. From our previous work, we have shown in batch experiments that CPE is a promising technique to remove chlorinated hydrocarbon and aromatic compounds from aqueous wastewater by concentrating them in the coacervate phase.^[9–11] These chemicals are common pollutants of great environmental concern originating from industrial effluents. The objective



of this work was to scale up the CPE technique to remove volatile aromatic contaminants from wastewater continuously in a differential rotating disk contactor (RDC). The effect of column temperature and added electrolyte on the CPE of toluene were investigated.

BACKGROUND

In previous batch studies of CPE of volatile organics from water, the equilibrium solute and surfactant partition ratios were measured for benzene, toluene, ethylbenzene, trichloroethylene, and di-, tri-, and tetrachloroethane.^[9–11] In Part I of this series^[15] on scaling up CPE, a continuous extraction column was shown to separate toluene from water with a height of transfer unit of approximately 30 cm. Operational problems only occurred if a linear velocity of the polluted feed stream flowing upward in the column is too high, which can cause entrainment of the coacervate stream in the overhead.

Most studies of liquid–liquid extraction in a RDC have focused on the hydrodynamics and mass transport aspects. There are many attempts to improve the extraction performance by considering the effect of the rotation of the rotor disk on the dispersed phase drop size, characteristic velocity of drops, etc.^[16–19] Adjustment of temperature and addition of a foreign substance do not generally have a dramatic effect on the extraction efficiency in these customary systems since two contacted phases are only slightly soluble. In contrast, in CPE, the solubility (or relative phase volumes) of dissimilar phases can be highly dependent on temperature or added electrolyte. In this, Part II of the series on RDC for CPE, the effect of temperature and added electrolyte (NaCl) was investigated.

A phase separation of the nonionic surfactant solution can be induced by a change in temperature or electrolyte concentration. When temperature increases, the micelles grow with higher aggregation numbers to form larger entities,^[20,21] which can increase the solubilization capacity for solubilizates. Glatter et al.^[22] reported that a sphere-to-rod micelle shape transition for *n*-alkyl polyglycol ether (CiEj) occurs as temperature increases. In addition, the cloud points of nonionic surfactants can be influenced by the addition of electrolyte due to the salting-in and the salting-out effect depending on the type of ions. The ions that are water structure formers, i.e., SO_4^{2-} , OH^- , F^- , and Cl^- , depress the cloud point by reducing the availability of free water molecules to hydrate the polyethylene oxide head groups. Meanwhile, the water structure breaker ions, i.e., I^- and SCN^- , raise the cloud point by enhancing the number of free water molecules available to interact with the polyethylene oxide head group.^[1,12,23,24]



EXPERIMENTAL

Materials

Reagent grade toluene from J. T. Baker (Phillipsburg, NJ) with a purity of 99.8% and NaCl from AJAX chemical (Auburn, Australia) with a purity of 99.9% were used. A polydisperse, commercial branched *t*-octylphenolpolyethoxylate, OP(EO)₇ with an average of 7 mol of ethylene oxide per mole of octylphenol (trade name Triton X-114) purchased from Dow Chemical Inc. (South Charleston, WV) was used as the nonionic surfactant. All chemicals were used as received. The water was distilled.

Apparatus: Rotating Disk Contactor

Figure 1 shows a schematic diagram of the CPE pilot plant. A cylindrical column made of Pyrex glass with 29.2 mm ID has an acrylic water jacket with 49.2 mm ID, through which temperature-controlled water can be circulated. The extractor column has a mixing zone in the middle and a settling or empty zone at either end of the column. To increase the residence time of the raffinate

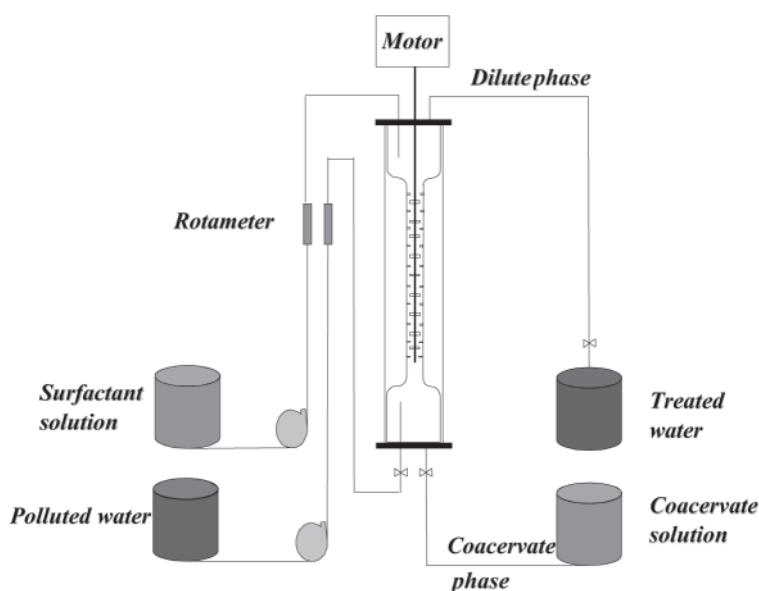


Figure 1. A schematic diagram of the CPE unit.



(micellar-dilute phase) and the extract phase (coacervate phase) before leaving the column, the diameter of the settling zone (100 mm ID) needs to be substantially larger than that of the mixing zone (29.2 mm ID). The heights of the settling zone and each mixing zone are 150 and 700 mm, respectively. In the mixing zone, there are 32 horizontal rotor disks of 17.52 mm in diameter and 1 mm in thickness mounted on a speed adjustable, vertical shaft at the center of the column. In addition, there are 33 annular stator rings with the outer and inner diameter of 29.2 and 20.44 mm, respectively, and 1 mm in thickness. The opening of the stator rings is larger than the rotor disk diameter. The compartment spacing between stators is 22 mm. The rotor disks, stators, and shaft are made of 316 stainless steel.

Procedures

As detailed in the Part I, the surfactant solvent solution and the contaminated feed water were fed into the extractor counter-currently at defined flowrates regulated by the rotameters. The system reached steady state prior to the 4-hour sampling time. After 4 hr of operation, the samples were collected from the effluent dilute phase and the coacervate phase (see Fig. 1) over a measured time interval to determine the flow rate and the concentration of nonionic surfactant and toluene for each phase.^[15]

The concentration of toluene was measured by a gas chromatograph with a flame ionization detector (Perkin Elmer, Inc., Shelton). Because of the high volatility of aromatic solutes, static headspace sampling was used as the sample injection technique, which eliminated interference of the high-molecular weight nonionic surfactant. The concentrations of nonionic surfactant, OP(EO)₇, was determined by using a CE 2000 series UV-spectrophotometer (Cecil Instrument Limited, Cambridge, England) at 224 nm, in which the absorptivity was in a range of 0–1 absorbance units (AU). The operating conditions of the gas chromatograph were described in our previous article.^[15] The external standard quantitative calibrations were obtained for the analysis of surfactant and toluene in both phases. The experiments were reproduced at least twice. Closure of the material balance was taken as evidence that leakage of the volatile solute is negligible.

The RDC operating variables and conditions were as follows: operating temperatures were 40°C, 45°C, and 50°C; concentration of the surfactant solution was 300 mM; concentration of solute in the wastewater was 100 ppm; concentrations of NaCl were varied from 0 to 0.6 M; agitator speed was 150 rpm; wastewater to surfactant solution flow rate ratio (feed to solvent flow rate ratio) was 6.9/1; solute was toluene.



RESULTS AND DISCUSSION

Effect of Temperature on Cloud Point Extraction of Toluene

When temperature increases, the system is further away from the cloud point, causing the nonionic surfactant to be less water soluble. The dehydration of the hydrophilic polyethoxylate groups of the surfactant enhances the intermolecular attraction, which makes the coacervate more concentrated and with lower volume as temperature is raised above the cloud point. The surfactant concentration in the coacervate phase increases substantially, while that in the dilute phase slightly declines with increasing temperature, as shown in Fig. 2. The flow rate of the coacervate phase decreases when temperature is raised as required from material balance considerations. The micellar size and aggregation number of the nonionic surfactant micelles increase with increasing temperature, especially beyond the cloud point.^[20–22] The increased surfactant concentration in the coacervate with increasing temperature results in a higher toluene concentration in the coacervate phase, as shown in Fig. 3. In addition, the concentration of toluene in the effluent dilute phase, decreases from 12.4 ppm at 40°C to 7.7 ppm at 50°C.

The ratio of toluene or surfactant concentration in the coacervate phase to that in the dilute phase is the partition ratio. Figure 4 shows a substantial

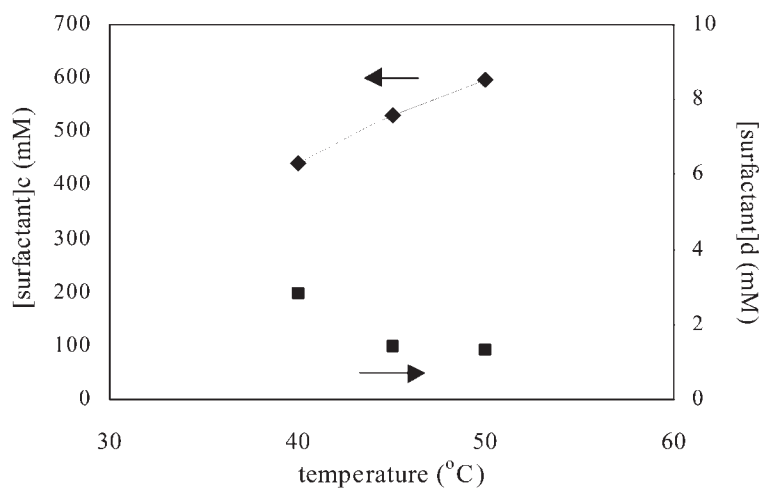


Figure 2. Surfactant concentration in coacervate stream (c) and dilute phase stream (d) as a function of temperature (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio and 150 rpm agitator speed).



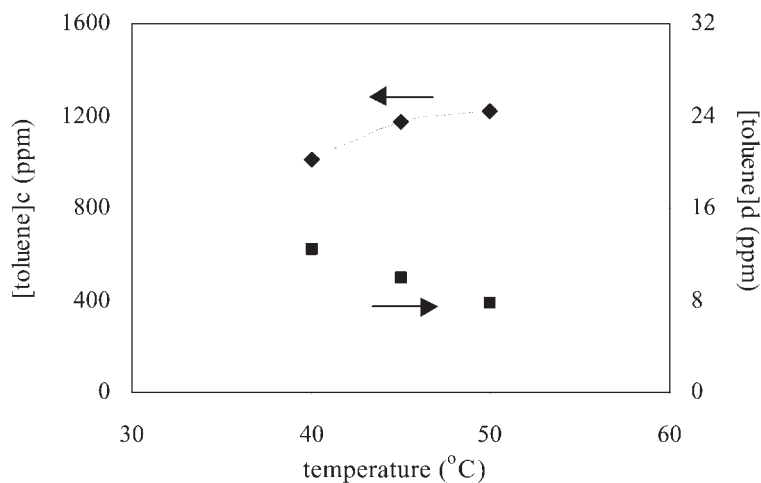


Figure 3. Toluene concentration in coacervate stream (c) and dilute phase stream (d) as a function of temperature (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio and 150 rpm agitator speed).

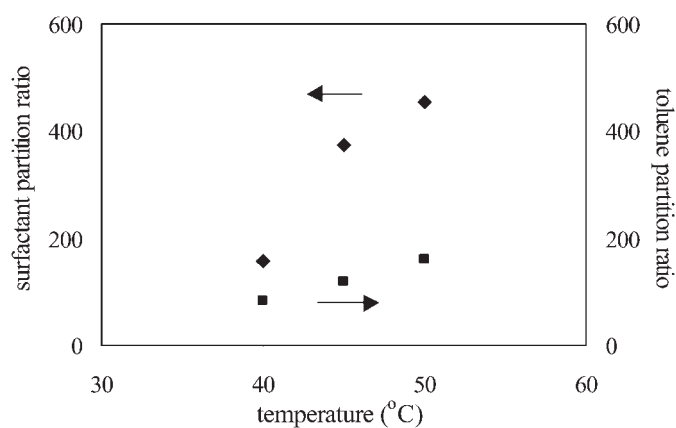


Figure 4. Surfactant and toluene partition ratio as a function of temperature (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio and 150 rpm agitator speed).



increase in surfactant and toluene partition ratio (about threefold and twofold, respectively) as temperature increases 10°C from 40°C to 50°C. The concentration of toluene in the coacervate is 159 times as concentrated as in the dilute phase stream, while the concentration of surfactant in the coacervate is 454 times as concentrated as in the dilute phase stream at 50°C. These trends correspond to previous results in batch experiments.^[9–11] However, the surfactant partition ratio obtained from the extractor is less than that obtained from a single-stage batch experiments. For example, the surfactant partition ratio in the continuous RDC is 156 compared to approximately 1300 in batch experiment at 40°C.^[11] This is due to an entrainment of fine coacervate droplets to the top of the column with the dilute phase, resulting in a higher surfactant concentration in the effluent dilute phase and a lower surfactant partition ratio. However, as much as 93% of surfactant is present in the coacervate stream at the lowest temperature studied here and 87.5% of toluene is extracted in the coacervate stream, as demonstrated in Fig. 5. The fraction of surfactant and toluene present in the coacervate phase slightly increases with increasing temperature.

It is very beneficial to increase the temperature since it results in higher surfactant and toluene partition ratios and a lower toluene concentration in the dilute phase. Also, the coacervate volumetric flow rate decreases when temperature is raised, which causes the treatment of the coacervate downstream for surfactant recovery to be less expensive. Nevertheless, there are theoretical limitations on the operating temperature of CPE. Upon increasing temperature, the upper critical temperature or upper consolute solution temperature can be reached, above which the phase separation does not take place.^[13] The upper consolute solution temperature is rarely reached for practical nonionic surfactant systems since it often exceeds 100°C (needs a pressurized system). Upon decreasing temperature, a point is reached where the coacervate droplets cannot flow downward and settle down at the bottom of the column since a density difference between the coacervate and the dilute phase is too small to accomplish a countercurrent operation in an agitated extractor. Flooding occurs at this temperature, which corresponds to the appearance of a cloudy surfactant solution at a certain location in the mixing zone. Below that position, there is no existence of the coacervate droplets. This particular operational problem occurs only in the agitated extractor at operating temperatures below 40°C. In batch experiments, the CPE showed good phase separation, even at 30°C because the separated phases were not disturbed while separating and settling, as detailed in a previous article.^[11] This is dissimilar to RDC where the separated phases are dispersed and coalesced along the column. This flooding effect is yet another incentive to operate at higher temperatures.



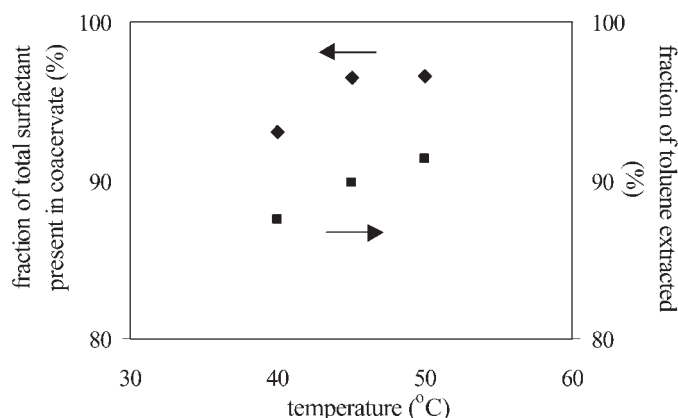


Figure 5. Fraction of total surfactant present in coacervate and fraction of toluene extracted in coacervate stream as a function of temperature (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio and 150 rpm agitation speed).

Effect of Added Electrolyte on the Cloud Point Extraction of Toluene

The addition of NaCl to the micellar solution of polyethoxylated nonionic surfactants in water can reduce the cloud point due to the salting-out effect.^[9,11,12,24] The cloud point depression is greater as the NaCl concentration increases.^[23] Hence, an increase in electrolyte concentration is analogous to increasing operating temperature at constant solution composition, in that an increase in the difference between operating temperature and cloud point can be attained either way. As shown in Figs. 6 and 7, the concentration of surfactant and of toluene in the coacervate phase increases with increasing electrolyte concentration, while these concentrations in the dilute phase decrease, which correspond qualitatively to the effect of increasing operating temperature. The addition of NaCl does not only depress the cloud point of the system but also reduces the solubility of the nonionic surfactant in the dilute phase. A decrease in surfactant concentration in the dilute phase causes a substantial increase in surfactant partition ratio from 156 without added electrolyte to 704 with the addition of 0.6 M NaCl, at 40°C as shown in Fig. 8. Likewise, the toluene partition ratio increases several fold from 82 without added electrolyte to 282 with the addition of 0.6 M NaCl. This salinity is approximately equivalent to more than 20°C temperature



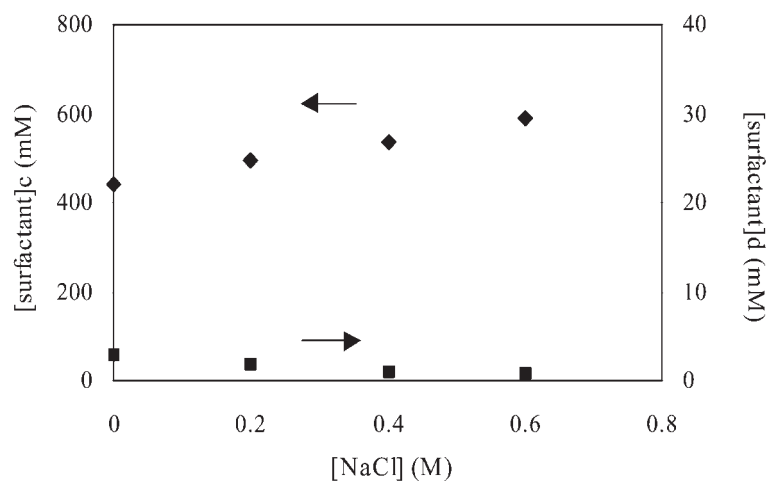


Figure 6. Surfactant concentration in coacervate stream (c) and dilute phase stream (d) as a function of NaCl concentration (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio, 150 rpm agitator speed and 40°C).

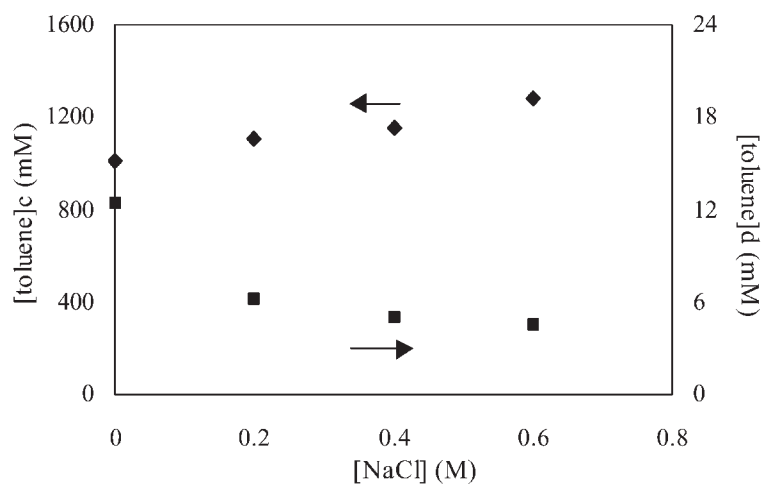


Figure 7. Toluene concentration in coacervate stream (c) and dilute phase stream (d) as a function of NaCl concentration (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio, 150 rpm agitator speed and 40°C).



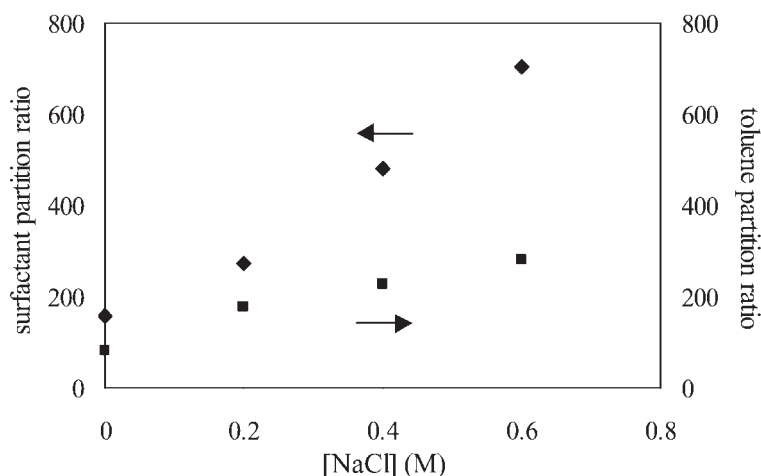


Figure 8. Surfactant and toluene partition ratio as a function of NaCl concentration (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flowrate ratio, 150 rpm agitator speed and 40°C).

increase in the effect on the toluene partition ratio. The fraction of surfactant and toluene present in the coacervate stream are enhanced by an increase in NaCl concentration, as shown in Fig. 9. As much as 95% of the toluene is extracted in the coacervate at 40°C with the addition of 0.6 M NaCl. Moreover, the addition of NaCl and an increase in temperature decrease the coacervate entrainment, as shown by a lower surfactant concentration in the dilute phase in Figs. 6 and 2. These results correspond qualitatively to those obtained from batch experiments. Since an increase in operating temperature can be an energy intensive procedure, the addition of electrolyte is an alternative to achieve a greater extraction efficiency. The other method is to adjust the nonionic surfactant structure to depress the cloud point to substantially below the operating temperature.

Determination of Number of Transfer Unit, Height of Transfer Unit, and the Overall Volumetric Mass-Transfer Coefficient

Use of the data from the RDC to calculate the height of transfer unit (HTU), number of transfer units (NTU) in our experimental column, and the mass-transfer coefficient (K_a) was discussed in Part 1 of this series.^[15]



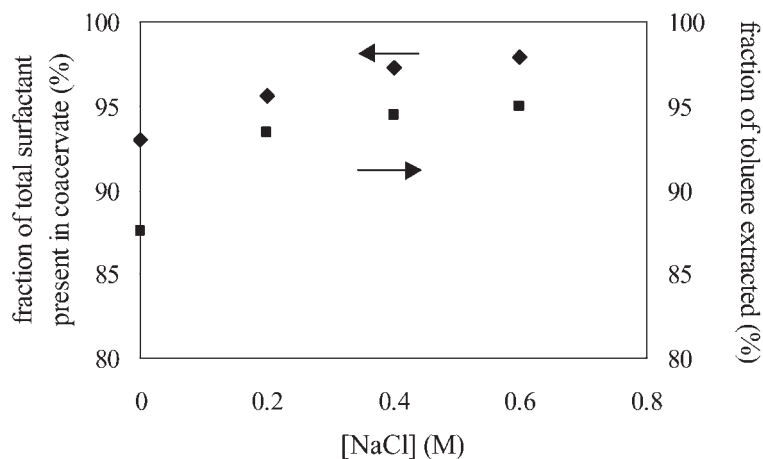


Figure 9. Fraction of total surfactant present in coacervate and fraction of toluene extracted in coacervate stream as a function of NaCl concentration (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio, 150 rpm agitation speed and 40°C).

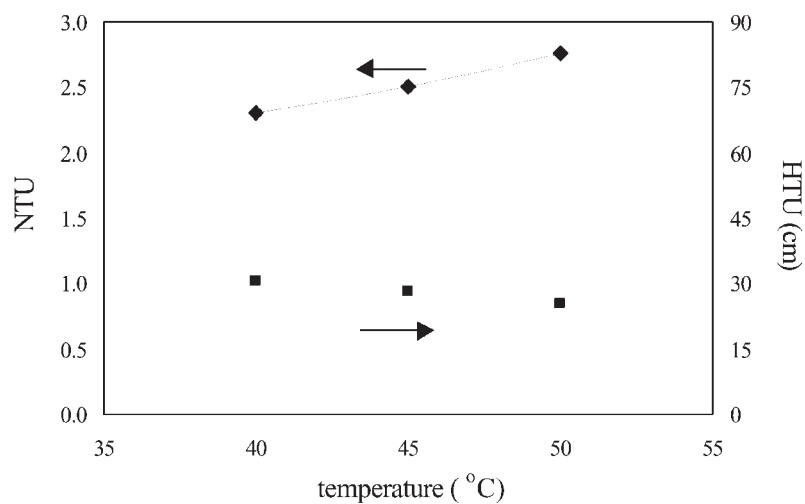


Figure 10. The NTU and HTU as a function of temperature (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio, 150 rpm agitator speed and 40°C).



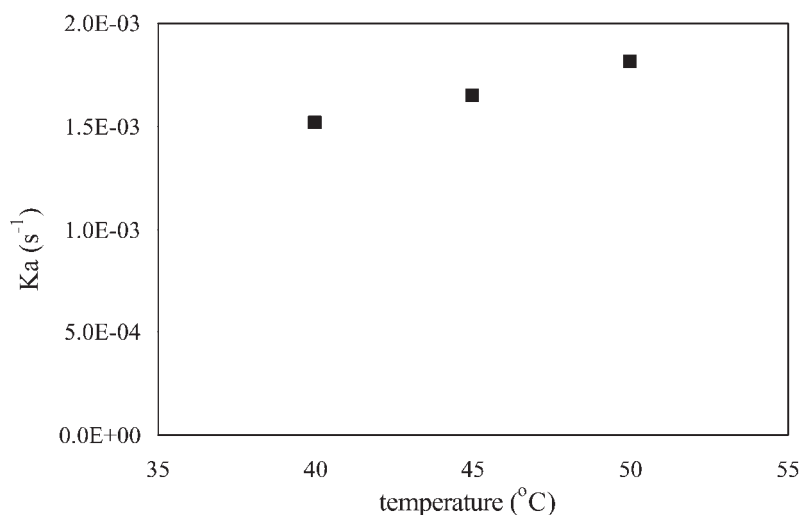


Figure 11. Overall volumetric mass-transfer coefficient (K_a) as a function of temperature (system: 100 ppm toluene, 300 mM surfactant solution, 6.9/1 wastewater to surfactant solution flow rate ratio, 150 rpm agitator speed and 40°C).

When temperature increases, a transfer of toluene to the coacervate droplets is enhanced, as indicated by a higher toluene partition ratio and toluene concentration in the coacervate phase. Therefore, at a certain column height, the NTU increases from 2.3 to 2.75 transfer units, indicating a better extraction efficiency, as operating temperature increases 10°C from 40°C to 50°C as shown in Fig. 10. In turn, the HTU decreases 5 cm from 30.4 to 25.4 cm. The relationship between K_a and operating temperature is shown in Fig. 11. The K_a increases by a factor of 1.2 or 20%, when temperature increases by 10°C.

The effect of NaCl addition on NTU, HTU, and K_a is not discussed here because of the unavailability of the equilibrium data from batch experiments needed for the calculation. However, the effect of added NaCl is expected to be similar to the effect of increasing temperature.

ACKNOWLEDGMENTS

Financial support for this work was provided by The Thailand Research Fund under The Royal Golden Jubilee Ph.D. Program and The Basic Research



Grant. In addition, support was received from the industrial sponsors of the Institute for Applied Surfactant Research including Akzo Nobel Chemicals Inc., Albemarle Corporation, Amway Corporation, Clorox Company, Colgate-Palmolive, Dial Corporation, Dow Chemical Company, DowElanco, E. I. DuPont de Nemours & Co., Halliburton Services Corp., Henkel Corporation, Huntsman Corporation, ICI Americas Inc., Kerr-McGee Corporation, Lever Brothers, Lubrizol Corporation, Nikko Chemicals, Phillips Petroleum Company, Pilot Chemical Company, Procter & Gamble Company, Reckitt Benckiser North America, Schlumberger Technology Corp., Shell Chemical Company, Sun Chemical Corporation, Unilever Inc., and Witco Corporation. John Scamehorn holds the Asahi Glass Chair in Chemical Engineering at the University of Oklahoma.

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Received March 2003

Revised August 2003



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