



## CHAPTER V

# RECOVERY OF NONIONIC SURFACTANT FROM VOC-CONTAMINATED COACERVATE PHASE SOLUTIONS BY CO-CURRENT VACUUM STRIPPING: EFFECT OF SURFACTANT CONCENTRATION, TEMPERATURE, AND SOLUTE TYPE\*

### 5.1 Abstract

To make a cloud point extraction (CPE) process economically feasible, the used surfactant in the concentrated or coacervate phase should be recycled and reused. We have demonstrated a co-current vacuum stripping using a 30.5-cm long column packed with glass Raschig rings effectively removes as much as 90% of the toluene from a surfactant coacervate phase solution without flooding or plugging. It was found that the apparent Henry's law constant of the volatile organic compounds (VOCs) was significantly reduced due to solubilization of VOCs in surfactant micelles, but increased substantially with increasing temperature. In addition, it was observed that the increase in hydrophobicity of the VOCs results in higher micellar solubilization in the coacervate solution, leading to lower VOC removal efficiency. However, over 87% of all studied VOCs (benzene, toluene, ethylbenzene, 1,2 dichloroethane, trichloroethylene, and tetrachloroethylene) were removed from a coacervate solution within a single stage operation. The removal of VOCs increases with decreasing surfactant concentration and VOC hydrophobicity, and also increases with increasing system temperature.

Key Words: VOCs; Coacervate; Co-current operation; Vacuum stripping; Cloud point extraction

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\* Accepted for publication in Separation and Purification Technology (Feb 11, 2009)

## 5.2 Introduction

The emission of volatile organic compounds (VOCs) from the petrochemical industry causes several environmental problems and health concerns [1, 2]. Since many of emitted VOCs are carcinogenic and mutagenic substances, even at very low concentration, a VOC treatment process is required to separate the VOC from the contaminated solution before discharging to a public water reservoir.

Surfactant-based separation processes are proposed for VOC recovery, since many surfactants are biodegradable and these processes generally consume less energy than conventional separation processes, e.g. distillation [3]. One attractive process is cloud point extraction (CPE), which utilizes nonionic surfactants as the separating agent. A number of CPE applications and reviews have been reported in the literature [4–8]. When a nonionic surfactant solution is heated above its cloud point temperature, the solution turns cloudy and then separates into two phases—a coacervate phase and a dilute phase. If there are organic solutes present in the system, the solutes tend to partition mainly to the coacervate phase or a micellar rich phase [9]. In general, this process is applied for the separation or purification of target organic compounds [10, 11], metal ions [12, 13], and biological products [14, 15], from an aqueous solution. Typically, the cloud point extraction efficiency increases with increasing temperature, surfactant concentration, electrolyte concentration, and solute hydrophobicity [10, 11, 16].

Most CPE studies have measured equilibrium extraction in small batch experiments (often test-tube scale). There are two aspects of CPE which are necessary to demonstrate to apply CPE to large flow rate waste streams economically. The first is to demonstrate that the CPE process is feasible in a continuous, multistage extraction column.

It has been shown that a continuous rotating disk contactor liquid-liquid extraction column is effective in removal of volatile aromatic contaminants from wastewater [17, 18]. A typical height of a transfer unit value is 30 cm for toluene. In a 70-cm high column, 85-95% of the volatile aromatic contaminants, toluene and ethylbenzene, were removed from wastewater.

From the mass balance, over 90% of the total surfactant ends up in the coacervate phase. These surfactants are the major cost of the CPE process; therefore, the second aspect of CPE to scale-up is to develop methods of surfactant recovery for reuse.

There are many processes that can be used to remove the VOCs from contaminated surfactant solutions: for example, air stripping, vacuum stripping, and pervaporation [19–27]. In a counter-current air stripping process, a direct contact between the air and liquid streams causes a significant amount of foam, leading to an inoperable process [20–22]. The use of the pervaporation process has been suggested to avoid foam formation; however, surfactants can form a gel layer on the pervaporating membrane surface and can consequently plug the membrane [25–27]. To overcome these problems, co-current vacuum stripping using a packed column is considered to be a promising process to remove the VOCs from a contaminated surfactant solution [23]. In the previous study, co-current vacuum stripping using a packed column was applied to remove toluene from the coacervate solution obtained from the CPE process [24]. Although the coacervate solution is very viscous, it successfully flows past the packing materials without plugging. The Henry's law constant of toluene is dramatically reduced by the presence of surfactants. However, as high as 90% of the toluene could be stripped from 300 mM OP(EO)<sub>7</sub>, for a single stage co-current operation. The process efficiency was enhanced by decreasing the feed flow rate and column pressure, and increasing the number of distributor holes. This study demonstrated the feasibility of recycling the solute-free viscous coacervate solution in the CPE process using vacuum stripping. In this subsequent study, further investigation of the effects of OP(EO)<sub>7</sub> concentration, operating temperature, and removal of VOCs with different properties on the vacuum stripping efficiency is studied.

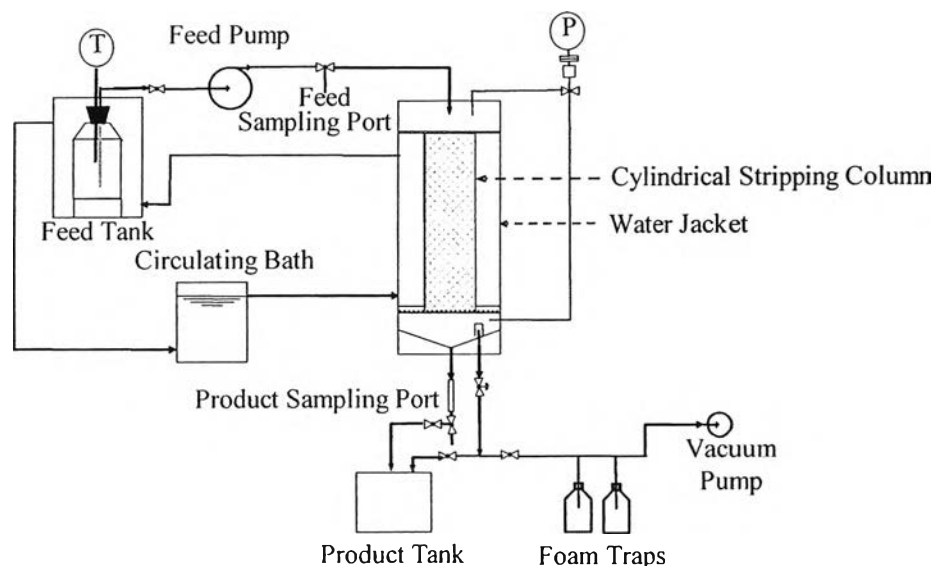
## 5.3 Experimental

### 5.3.1 Materials

In this study, the nonionic surfactant is a branched *t*-octylphenolpolyethoxylate, OP(EO)<sub>7</sub> having an average of seven repeating ethylene oxide units (Triton X-114) obtained from Dow Chemical Inc. (South Charleston, WV, USA). Reagent grade benzene with 99.8% purity, toluene, ethylbenzene, trichloroethylene, and tetrachloroethylene with 99.5% purity were purchased from J.T. Baker (Phillipsburg, NJ, USA). Reagent grade 1,2 dichloroethane with 99.8% purity was purchased from Labscan Co., Ltd. (Bangkok, Thailand). All chemicals were used as received. Deionized (DI) water was used for preparing all the solutions.

### 5.3.2 Apparatus: Vacuum Stripping Packed Column

A schematic of the vacuum stripping unit is given in Figure 5.1. The cylindrical stripping glass column had a 6.5-cm inside diameter and a 34-cm height, surrounded by a 16-cm diameter acrylic water jacket. The stripping column was connected with a 3-hole distributor at the top of the column, and was packed with 5 mm  $\times$  5 mm glass Raschig rings to a height of 30 cm. A 1-L feed tank was made of glass and was placed inside a 16-cm diameter acrylic water jacket. A product tank was made of stainless steel. The system was maintained under vacuum by a rotary vane pump. The feed solution was sent to the stripping column using a peristaltic pump. The operating temperature was controlled using a water circulating bath.



**Figure 5.1** Schematic of the vacuum stripping unit.

### 5.3.3 Methods

#### 5.3.3.1 Equilibrium Determination

The partitioning behavior of VOCs in water and in the surfactant solution was observed by modification of the Equilibrium Partitioning in Closed Systems (EPICS) method [26, 28]. For a typical experiment, 4 mL of the VOC with and without surfactant was prepared in several identical 22-mL glass vials with Teflon-coated septa and aluminum holed caps. To determine the equilibrium time, the vials were placed in an

isothermal chamber of the headspace autosampler (G1888, Agilent Technology, USA) at the studied temperatures (30, 40, and 50°C). The vapor sample in the headspace was automatically withdrawn and then analyzed by a gas chromatograph (6890N, Agilent Technology, USA) every 10 min. The time required to reach equilibrium of the VOC in the system was then determined.

Subsequently, the Henry's law correlation of the VOC-water system was determined. Several identical 22-mL glass vials containing 4 mL of an aqueous solution containing the VOC were prepared. The VOC was protected from leakage by capping the vials with Teflon-coated septa and aluminum holed caps. The VOC concentration was varied but was kept below its water solubility limit. After reaching equilibrium, the VOC concentration in the headspace and in the liquid phase was analyzed by the gas chromatograph connected with the headspace autosampler. Finally, the equilibrium correlation between VOC concentration in vapor and in liquid phases was obtained. For the surfactant-containing system, due to higher solubility in the surfactant solutions, the VOC concentrations were varied from 100 to 2 000 ppm. The experiments were conducted in a similar manner as those for the VOC-water system.

#### 5.3.3.2 *Continuous Operation*

The pressure inside the system was maintained under vacuum during the operation by using the rotary vane pump. The operating temperature was controlled using the water circulating bath. After the column pressure was stable, 1.1 mL/min of the feed solution was pumped onto the top of the column by the peristaltic pump. The viscous surfactant-containing liquid was able to flow down along the packed column and was stored in the product tank. A vapor suction line was attached at the bottom of the stripping column to operate in co-current mode. A cold trap was installed to prevent the rotary vane pump from liquid-induced damage. The vacuum stripper reached steady state within 90 min. The liquid sample was collected by a gas-tight syringe in the feed and product streams for VOC concentration analysis.

The operating conditions in the vacuum stripping packed column were varied as follows: operating temperature, 30–50°C; OP(EO)<sub>7</sub> concentration, 300–630 mM; VOC concentration, 300–2 000 ppm; and absolute column pressure, 38–94 Torr. According to our previous work [24], the feed flow rate was fixed at 1.1 mL/min and the number of distributor holes was 3. The aromatic hydrocarbon and chlorinated

hydrocarbon VOCs studied here are: benzene (BEN), toluene (TOL), ethylbenzene (ETB), trichloroethylene (TCE), tetrachloroethylene (PCE), and 1,2 dichloroethane (DCE).

The mathematical model and theory used to calculate parameters like the apparent Henry's law constant and the mass transfer coefficient from the experimental data reported here are described in our previous work [24].

## 5.4 Results And Discussion

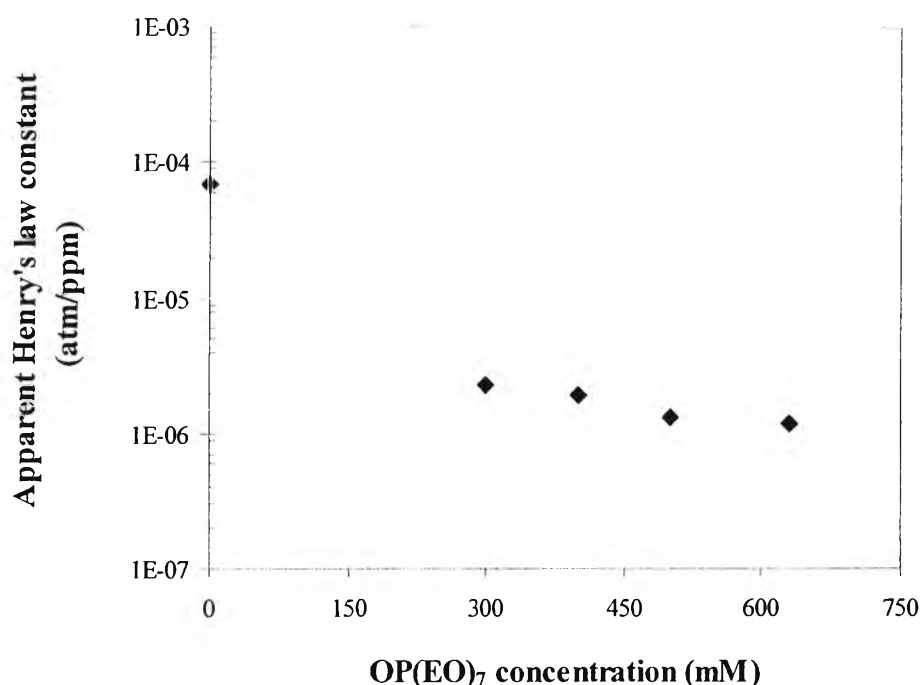
### 5.4.1 Effect of Surfactant Concentration on Toluene Vacuum Stripping

The previous work demonstrated that within a single stage operation, the co-current vacuum stripping successfully removed as much as 90% of the toluene from 300 mM OP(EO)<sub>7</sub>, indicating the feasibility of leaving the coacervate phase dilute in the VOC, permitting recycling for surfactant reuse [24]. The OP(EO)<sub>7</sub> concentration in the coacervate phase from the CPE process could possibly be higher than this 300 mM, depending on extraction conditions like temperature [18]. Therefore, this work studies the effect of OP(EO)<sub>7</sub> concentrations at 300, 400, 500, and 630 mM on the vacuum stripping of VOCs from the coacervate phase.

Following our previous study [24], toluene was first used as the model VOC. The volatilization of toluene in the OP(EO)<sub>7</sub> coacervate solution is quantified by determining the apparent Henry's law constant of toluene ( $H_{App,TOL}$ ). This value was observed by comparing the toluene concentration in both vapor and liquid phases between the surfactant-free system and the OP(EO)<sub>7</sub>-containing system. The initial toluene concentration was varied from 50 to 200 ppm for the surfactant-free system, and from 100 to 2 000 ppm for OP(EO)<sub>7</sub> system.

As illustrated in Figure 5.2, the  $H_{App,TOL}$  in the system containing the coacervate phase is almost two orders of magnitude less than the reported Henry's law constant of toluene in water at 30°C ( $6.8 \times 10^{-5}$  atm/ppm) [29]. A similar trend has also been observed in other studies with anionic, cationic, and nonionic surfactants [26, 28–30]. It can be explained that as the surfactant concentration increases above its CMC, more micelles are formed, which provide a greater hydrophobic volume to solubilize toluene. Therefore, toluene activity is reduced at the same total concentration, leading to a reduction in the Henry's law constant. Furthermore, the equilibrium data can be used to

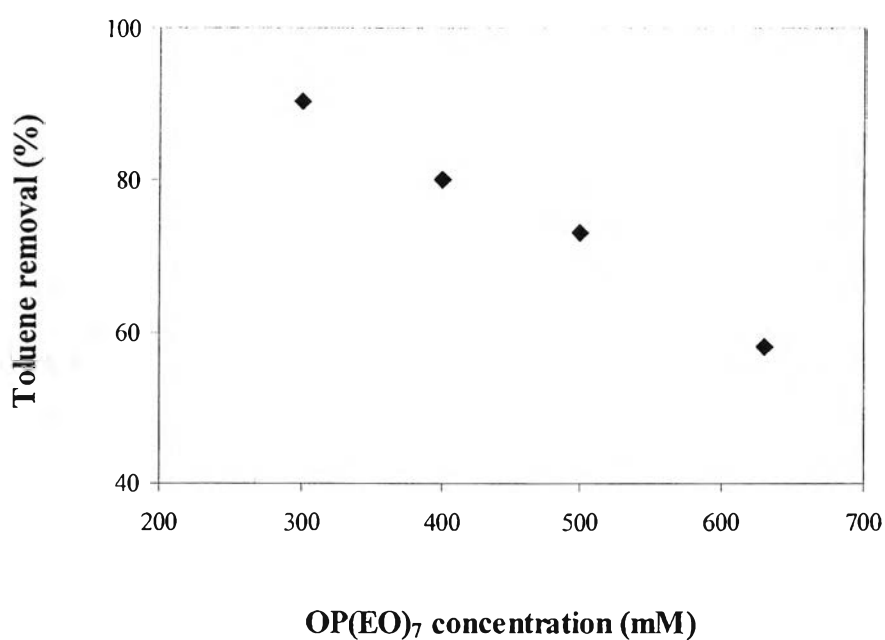
calculate the toluene solubilization constant ( $K_{s,TOL}$ ). From batch experiments, it was found that the  $K_{s,TOL}$  is relatively constant at  $90 \text{ M}^{-1}$  even though the OP(EO)<sub>7</sub> concentration was increased from 300 to 630 mM. According to the pseudo-phase separation model, the nearly unchanged  $K_{s,TOL}$  indicates that the equilibrium partitioning of toluene between the micellar and extramellar phases is not altered although more micelles are formed by the addition of OP(EO)<sub>7</sub>.



**Figure 5.2** Variation of apparent Henry's law constant of toluene with OP(EO)<sub>7</sub> concentration at 30°C.

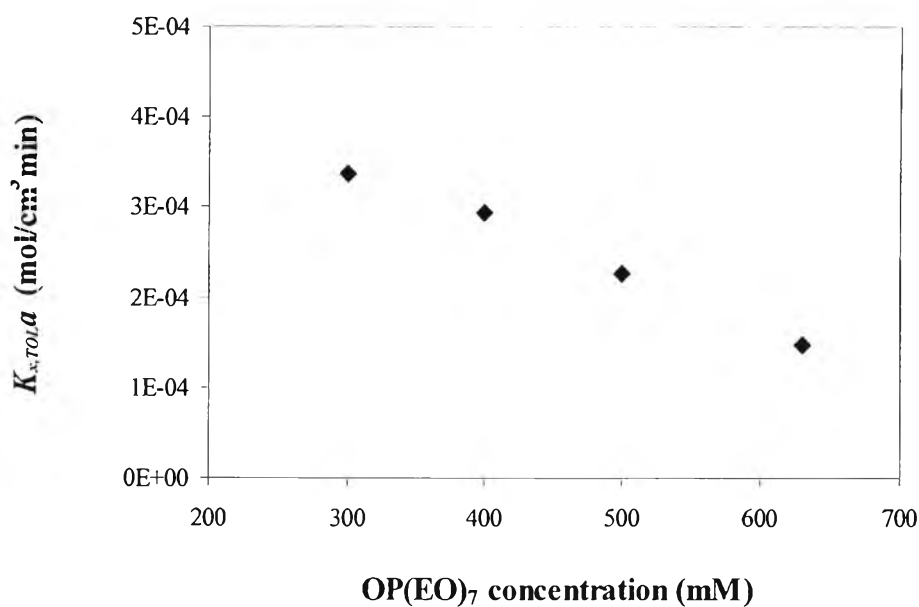
Figures 5.3 and 5.4 respectively show that the removal of toluene decreases from 90 to 58% and the overall liquid phase volumetric mass transfer coefficient of toluene ( $K_{x,TOL}a$ ) decreases from  $3.36 \times 10^{-4}$  to  $1.47 \times 10^{-4} \text{ mol}/(\text{cm}^3\text{-min})$  as the OP(EO)<sub>7</sub> concentration increases from 300 to 630 mM, respectively at the operating conditions: 1.1 mL/min feed flow rate, 38 Torr column pressure, 3-hole distributor, and 30°C. The decreases in toluene removal and the  $K_{x,TOL}a$  with increasing surfactant concentration generally are due to two possible causes [23, 26, 31, 32]. First, the

surfactant molecules are likely to adsorb at the interface and retard the liquid motion, leading to a higher solution viscosity and a lower toluene mass transfer. Second, the chemical potential of toluene is reduced when it solubilizes in the surfactant micelle and, as a result, the driving force for separation is decreased, which is in agreement with the lowering  $H_{App,TOI}$  value observed from the batch experiment. The increasing surfactant concentration significantly reduces the toluene removal efficiency and it is a crucial factor in the design of the stripping process [20]. In addition, foaming was observed under operation at high OP(EO)<sub>7</sub> concentrations (500–630 mM), resulting in a decrease in column performance.



**Figure 5.3** Toluene removal as a function of OP(EO)<sub>7</sub> concentration: 300 ppm toluene feed, 1.1 mL/min feed flow rate, 38 Torr column pressure, 3-hole distributor, and 30°C.

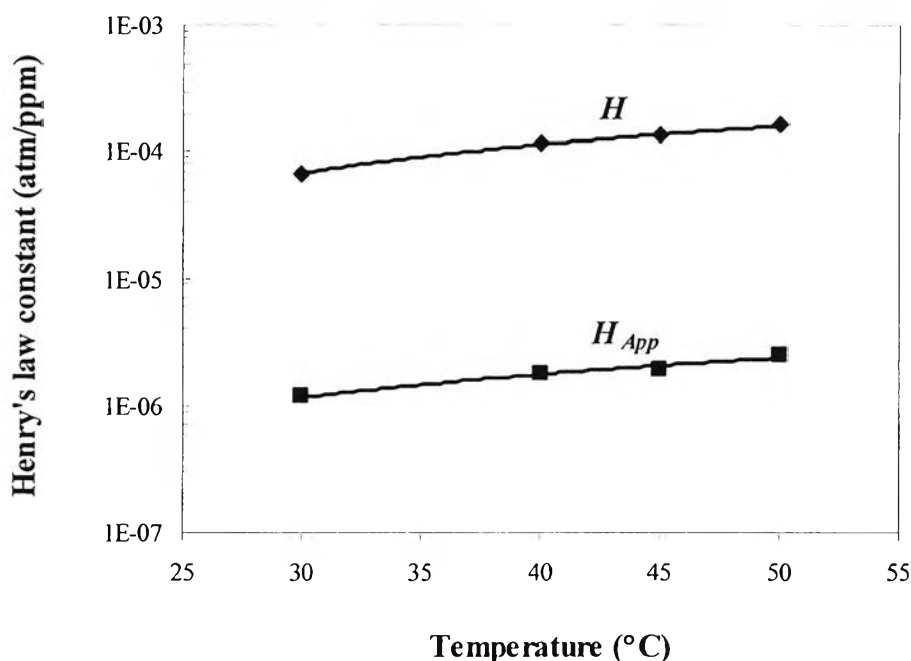




**Figure 5.4** Overall liquid phase volumetric mass transfer coefficient of toluene ( $K_{x,TOL}a$ ) as a function of OP(EO)<sub>7</sub> concentration: 300 ppm toluene feed, 1.1 mL/min feed flow rate, 38 Torr column pressure, 3-hole distributor, and 30°C.

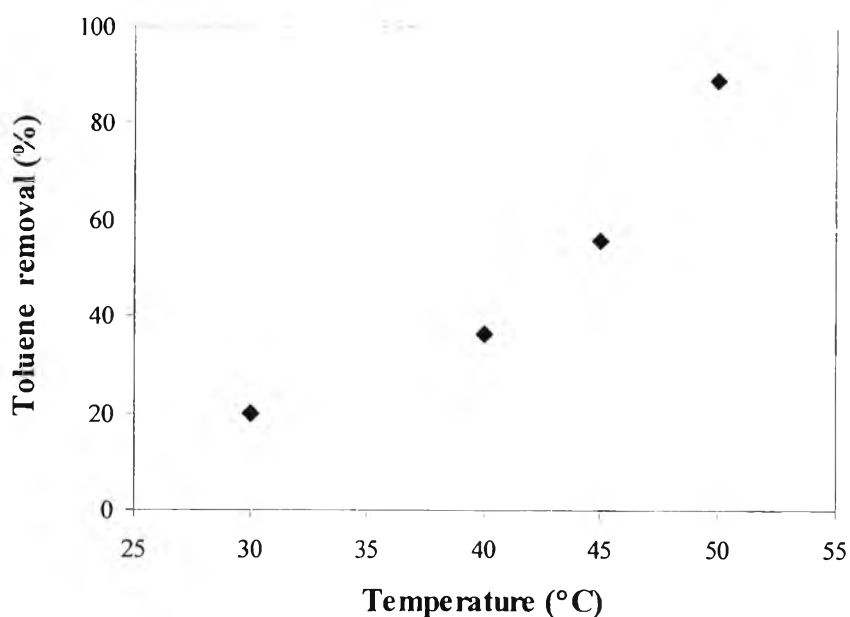
#### 5.4.2 Effect of Temperature on Toluene Vacuum Stripping

According to the literature report, the Henry's law constant of the VOC generally increases with increasing operating temperature, which indicates the higher volatilization of the VOCs in a system [29, 30, 33, 34]. The  $H_{App,TOL}$  was determined in this study at various temperatures in the range of 30–50°C. As shown in Figure 5.5, the Henry's law constant of toluene ( $H_{TOL}$ ) and  $H_{App,TOL}$  values continuously increase with increasing temperature due to the increasing toluene vapor pressure with temperature. This indicates the possibility of stripping more toluene from the coacervate solution in the packed column operating at higher temperature. However, Figure 5.5 also shows that the  $H_{App,TOL}$  is about two orders of magnitude lower than  $H_{TOL}$  over the entire studied temperature range due to the solubilization of toluene in surfactant micelles [29].

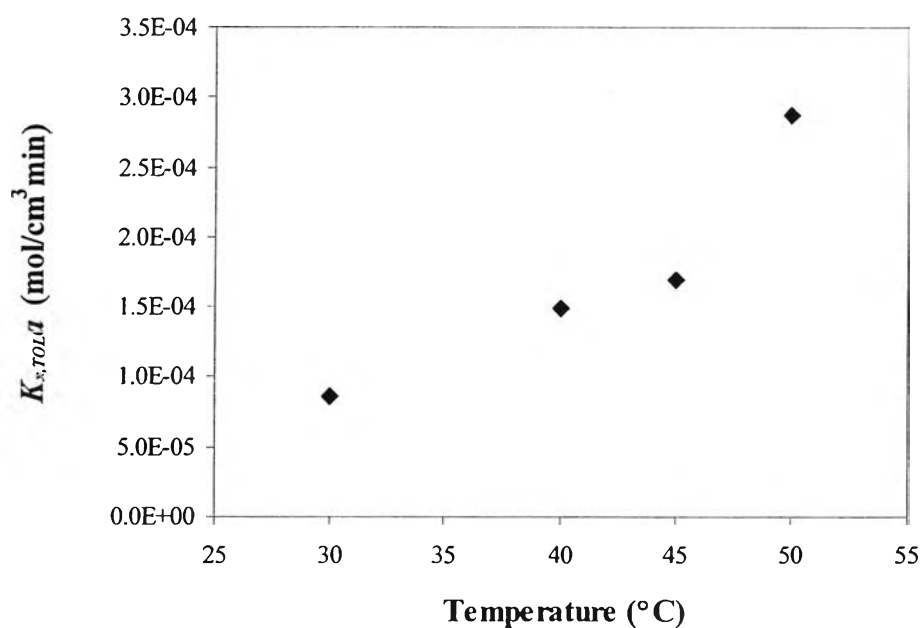


**Figure 5.5** Variation of Henry's law constants of toluene in water (◆) and 630 mM OP(EO)<sub>7</sub> (■) with temperature.

The stripping packed column was operated at 1.1 mL/min feed flow rate, 94 Torr column pressure, 3-hole distributor, and temperature ranging from 30 to 50°C. As illustrated in Figure 5.6 and 5.7, raising operating temperature from 30 to 50°C significantly increases the toluene removal from 20 to 90% and the  $K_{x,TOL}a$  from  $8.63 \times 10^{-5}$  to  $2.87 \times 10^{-4}$  mol/(cm<sup>3</sup>-min) due to the higher rate of toluene vaporization. Moreover, the improvement of the overall liquid phase volumetric mass transfer coefficient agrees well with those reported by Choori *et al.* [23]. In addition, the viscosity of the 630 mM OP(EO)<sub>7</sub> solution is greatly reduced when the temperature increased from 30 to 50°C [24]. The reduction of the viscosity also enhances the VOC mass transfer due to lower mass transfer resistance in the liquid solution [25]. Furthermore, no significant foaming was observed between 40°C and 50°C, probably due to the quick foam collapse caused by water drainage from the foam lamella of the lower viscosity coacervate solution [9]. The reduction of foam and coacervate viscosity at the high temperature allows the stripping in packed column to be done at the higher feed flow rate, which leads to higher unit capacity.



**Figure 5.6** Toluene removal as a function of temperature: 300 ppm toluene feed, 630 mM OP(EO)<sub>7</sub> feed, 1.1 mL/min feed flow rate, 94 Torr column pressure, and 3-hole distributor.




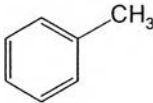
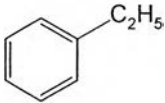
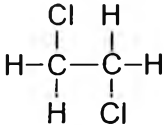
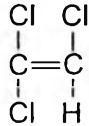
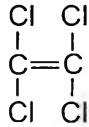
**Figure 5.7** Overall liquid phase volumetric mass transfer coefficient of toluene ( $K_{x,TOL}a$ ) as a function of temperature: 300 ppm toluene feed, 630 mM OP(EO)<sub>7</sub> feed, 1.1 mL/min feed flow rate, 94 Torr column pressure, and 3-hole distributor.

### 5.4.3 Effect of Solute Type on Vacuum Stripping

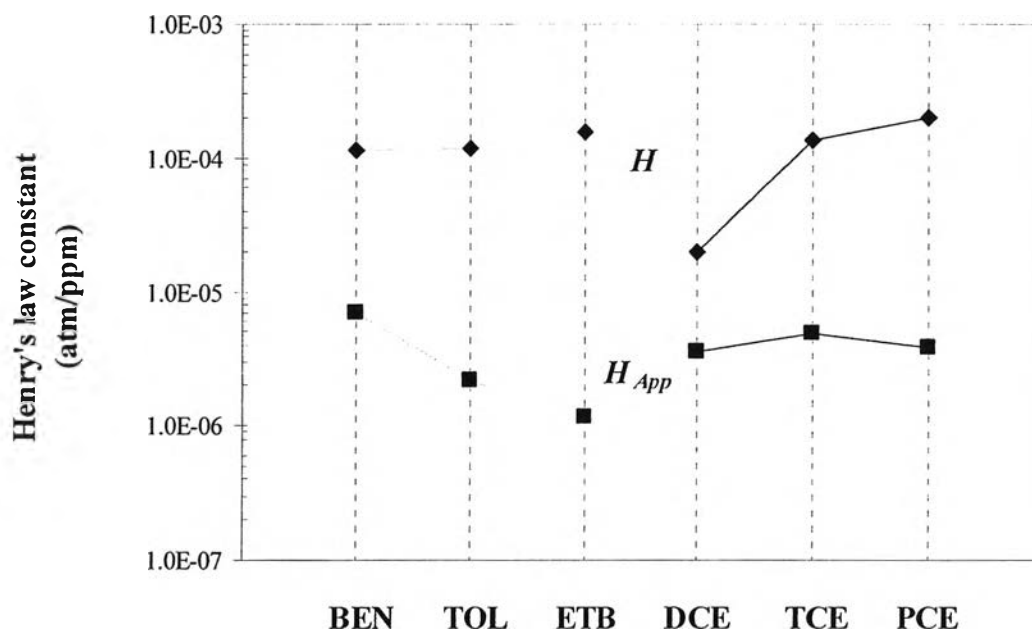
Generally, several types of VOCs can be present in the contaminated aqueous solution. A variety of VOCs have been effectively removed using the CPE process [10, 11, 16–18, 35]. After phase separation in CPE, most of VOCs are dissolved in the coacervate phase. The effect of hydrophobicity/volatility of two major types of VOC pollutants, aromatic hydrocarbons and chlorinated hydrocarbons, was investigated. The selected aromatic VOCs are benzene (BEN), toluene (TOL), and ethylbenzene (ETB); and the chlorinated VOCs are 1,2 dichloroethane (DCE), trichloroethylene (TCE), and tetrachloroethylene (PCE). Typically, as the degree of alkylation or chlorination of VOCs increases, the VOCs are more hydrophobic and tend to be more solubilized in the surfactant micelles. This might enhance the extraction efficiency but possibly cause a negative effect on VOC removal from surfactant solution.

It is interesting to put forward that the tendency of volatilization and solubilization of the VOCs in coacervate solution should be quantitatively represented by the  $H_{App}$  and  $K_s$ , respectively. To determine those of such VOCs, the Henry's law constants of each VOC are employed [29, 36, 37]. Table 5.1 shows the observed  $H$ ,  $H_{App}$ , and  $K_s$  values for all VOCs studied. In comparison, as shown in Figure 5.8, when the degree of alkylation or chlorination of the VOCs increases, the  $H_{App}$  significantly decreases at least one order of magnitude compared to the  $H$ ; however, the  $K_s$  increases dramatically (Table 5.1). The  $K_s$  values increase with increasing degree of VOC alkylation/chlorination (i.e. VOC hydrophobicity), due to the increase in hydrophobic attraction. As a result, the lower volatilization of the VOCs is observed. A similar observation was reported elsewhere [29, 30, 38].

**Table 5.1** The structure, Henry's law constant, apparent Henry's law constant, and solubilization constant of all VOCs in batch experiments. Condition: 20–800 ppm VOC in water, 100–2 000 ppm VOC in 450 mM OP(EO)<sub>7</sub>, and 40°C.

VOCs	Structure of the VOCs	Henry's law constant, $H$ (atm/ppm)	Apparent Henry's law constant, $H_{App}$ (atm/ppm)	Solubilization constant, $K_s$ (M <sup>-1</sup> )
BEN		$1.1 \times 10^{-4b}$	$6.8 \times 10^{-6}$	34
TOL		$1.2 \times 10^{-4a}$	$2.2 \times 10^{-6}$	108
ETB		$1.5 \times 10^{-4b}$	$1.2 \times 10^{-6}$	294
DCE		$2.0 \times 10^{-5c}$	$3.5 \times 10^{-6}$	10
TCE		$1.3 \times 10^{-4a}$	$4.9 \times 10^{-6}$	59
PCE		$2.0 \times 10^{-4a}$	$3.8 \times 10^{-6}$	113

Letters a,b,c express data cited from Refs. [29, 36, 37], respectively.



**Figure 5.8** Henry's law and apparent Henry's law constants of the VOCs in water (◆) and in 450 mM OP(EO)<sub>7</sub> (■) at 40°C.

For the vacuum stripping experiment performed at 1.1 mL/min feed flow rate, 59 Torr column pressure, 3-hole distributor, and 40°C, the resultant VOC removal and the  $K_x a$  values of the VOCs are tabulated in Table 5.2.


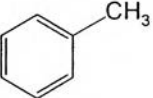
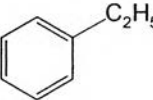
For the aromatic ones, the VOC removal is decreased with increasing VOC hydrophobicity, so does the  $K_x a$  values although the same order of magnitude is observed. This is because an increase in VOC hydrophobicity results in increasing solubilization of VOCs in the surfactant micelles.

As, for the chlorinated VOCs, the different trend can be observed for both the VOC removal and  $K_x a$  values. According to the Henry's law constants of three chlorinated VOCs, it is clear that the PCE is the easiest to be removed from water by a conventional air stripping process, followed by TCE, and then DCE. However, the solubilization effect plays a significant role in VOC volatilization in the presence of surfactant. The solubilization constant of the VOCs increases in the order of DCE < TCE < PCE, along with the order of their hydrophobicity. The increase in VOC

solubilization, in turn, retards the volatilization of the VOCs, as indicated by the higher differences between the  $H$  and  $H_{App}$  values (Figure 5.8).

It should be noted that more than 87% removal of all six VOCs can be attained from 450 mM OP(EO)<sub>7</sub> coacervate solution by the single stage vacuum stripping.

**Table 5.2** The structure, VOC removal, and the overall liquid phase volumetric mass transfer coefficient of all VOCs: 2 000 ppm VOC feed, 450 mM OP(EO)<sub>7</sub> feed, 1.1 mL/min feed flow rate, 59 Torr column pressure, 3-hole distributor, and 40°C.

VOCs	Structure of the VOCs	VOC removal (%)	The overall liquid phase volumetric mass transfer coefficient, $K_x a$ (mol/cm <sup>3</sup> min)
BEN		97.7	$4.4 \times 10^{-4}$
TOL		90.6	$3.3 \times 10^{-4}$
ETB		87.3	$3.1 \times 10^{-4}$
DCE	$\begin{array}{c} \text{Cl} \quad \text{H} \\   \quad   \\ \text{H}-\text{C}-\text{C}-\text{H} \\   \quad   \\ \text{H} \quad \text{Cl} \end{array}$	95.5	$3.9 \times 10^{-4}$
TCE	$\begin{array}{c} \text{Cl} \quad \text{Cl} \\   \quad   \\ \text{C}=\text{C} \\   \quad   \\ \text{Cl} \quad \text{H} \end{array}$	96.4	$3.8 \times 10^{-4}$
PCE	$\begin{array}{c} \text{Cl} \quad \text{Cl} \\   \quad   \\ \text{C}=\text{C} \\   \quad   \\ \text{Cl} \quad \text{Cl} \end{array}$	91.3	$3.1 \times 10^{-4}$

## 5.5 Conclusions

The co-current operation was effective to avoid flooding in the packed column although foaming was observed. From the batch experiment, the Henry's law constants of the VOCs in OP(EO)<sub>7</sub> solution were substantially reduced as compared to those in water, due to the solubilization of the VOCs in the surfactant



micelles. The Henry's law constant of a VOC was significantly decreased with increasing OP(EO)<sub>7</sub> concentration and VOC hydrophobicity, but was markedly increased with increasing temperature. From the continuous operation, if the OP(EO)<sub>7</sub> concentration was increased, the toluene removal and its  $K_{x,TOL}a$  value were drastically reduced. In addition, a significant amount of foam was also observed in the packed column, especially at high OP(EO)<sub>7</sub> concentration (500–630 mM). Moreover, if the operating temperature was increased from 30 to 50°C, the toluene removal and its  $K_{x,TOL}a$  were substantially increased. Foaming and viscosity of the coacervate solution were markedly reduced at operating temperatures of 40–50°C. Such an observation would allow the stripping column to operate at a higher feed flow rate, leading to a higher process capacity. Furthermore, based on the VOC types, the higher hydrophobic VOCs were more solubilized in the OP(EO)<sub>7</sub> solution, leading to a decline in stripping process efficiency.

## 5.6 Acknowledgements

The Thailand Research Fund (TRF) is acknowledged for providing a research grant and the Royal Golden Jubilee Ph.D. fellowships (S. Kungsanant). The Research Unit: Applied Surfactants for Separation and Pollution Control supported by the Center for Petroleum, Petrochemicals and Advanced Materials (PPAM) and the Ratchadapiseksompote Fund, Chulalongkorn University, are also acknowledged for providing research facilities and partial funding. In addition, support was received from the Industrial Sponsors of the Institute for Applied Surfactant Research, University of Oklahoma including Akzo Nobel, Clorox, Conoco/Philips, Church and Dwight, Dow, Ecolab, Halliburton, Huntsman, Oxiteno, Procter & Gamble, Sasol North America, Shell Chemical, and Unilever.

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