

**CHAPTER VI**  
**CONTINUOUS CLOUD POINT EXTRACTION OF AROMATIC**  
**CONTAMINANTS FROM WASTEWATER: SURFACTANT**  
**ENTRAINMENT AND POTENTIAL SOLUTION**\*

**6.1 Abstract**

Cloud point extraction (CPE) has been successfully scaled up by our research group to continuously remove aromatic contaminants from wastewater in a multi-stage rotating disc contactor (RDC) using t-octylphenolpolyethoxylate, for which a high yield of extraction was achieved. However, the entrainment of coacervate droplets into the dilute phase (surfactant entrainment) problem was observed in our previous studies as it decreased the performance of the RDC compared to predictions from equilibrium stage operations. The entrainment of surfactant significantly increased as wastewater feed flow rate, surfactant feed flow rate, rotating disc speed, and concentration of solute in wastewater feed increased; on the other hand, the entrainment decreased as operating temperature and salt concentration increased. Effect of the lowering of surfactant feed location was proposed to resolve the problem; however, it was found that the surfactant feed location did not significantly reduce the amount of entrainment. Moreover, the toluene extraction efficiency significantly declined due to the shorter contact time between the coacervate and wastewater streams. The temperature-controllable settling unit designed in the shape of a separating funnel, was used to further treat the dilute phase after the extraction; and it showed high potential in the separation of the coacervate droplets from the dilute phase. From the study, the efficiency of the unit was mainly dependent on the settling temperature and residence time. The optimum conditions of the unit were also investigated.

*Keywords: cloud point extraction, coacervate, continuous, surfactant entrainment*

\* In preparation

## 6.2 Introduction

Cloud point extraction (CPE), a surfactant-based separation technique, which utilizes a nonionic surfactant as the separating agent, is recognized as an effective technique for removing dissolved aromatic contaminants from wastewater (1-16). For nonionic surfactant, at a temperature above its cloud point (CP), the homogeneous surfactant solution will separate into two immiscible phase, a micellar rich phase or coacervate phase and a micellar dilute phase, which have a large difference in the surfactant concentration. The phase volume of coacervate phase is usually relatively small compared to that of the dilute phase. The contaminants contained in the solution tend to incorporate or solubilize into the surfactant micelles and concentrate into the coacervate phase after the phases have separated. The dilute phase containing a lower concentration of solutes can be released to the environment if the concentration is below the regulated level as treated water.

The CPE technique was successfully scaled-up to remove aromatic contaminants from a wastewater stream continuously in a counter current multi-stage rotating disc contactor (RDC) by our research group (10-12). The utilization and practical significance of the CPE technique for large-scaled downstream processing in wastewater remediation was shown. Even, the CPE unit could provide up to 99% of extraction of hydrophobic aromatic contaminants (eg. toluene, ethylbenzene) (12); however, more attention should be paid on the efficiency of the surfactant extraction. It was found that the present of total surfactant in the coacervate stream and its partition ratio were comparable low to the equilibrium condition due to the entrainment of surfactant into the overhead effluent.

Surfactant entrainment or coacervate entrainment was defined as the entrainment or contamination of surfactant coacervate droplets into the upward dilute phase stream by the small density difference. This led to the high surfactant concentration in the dilute phase (treated water) after the extraction. The entrainment of coacervate into the dilute phase is the important factor observed which decreased performance of the RDC compared to predictions from equilibrium stage operations. As observed in our previous studies, the entrainment of surfactant significantly increased as wastewater feed flow rate, surfactant feed flow rate, rotating disc speed,

and concentration of solute in wastewater feed increased; on the other hand, decreased as operating temperature and salt concentration increased (9,10,12).

In this work, the potential solutions for the reduction of the surfactant entrainment are proposed in order to improve the efficiency of the continuous CPE. The effect of surfactant feed location on CPE of toluene from wastewater was investigated, as predicted that the lowering in the surfactant feed position might be possible to lengthen the coacervate droplet settling time inside the column. A study on an application of the coacervate settling unit for treating the surfactant or coacervate entrainment from the dilute phase after the extraction was also investigated.

### 6.3 Experimental

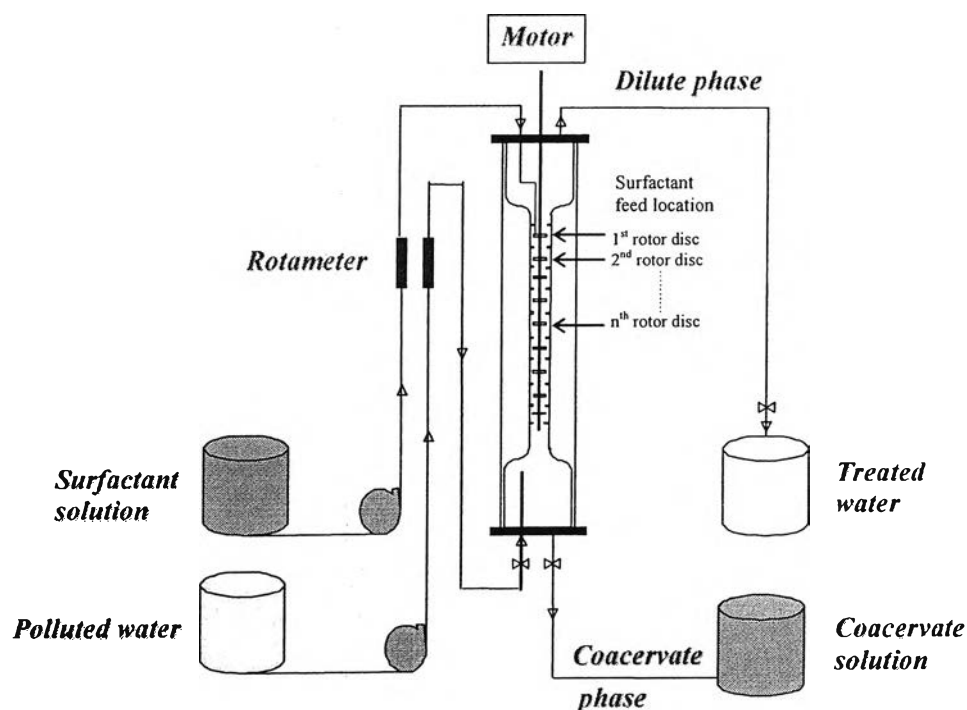
#### 6.3.1 Materials

A polydisperse commercial branched t-octylphenolpolyethoxylate, OP(OE)<sub>7</sub>, with an average of 7 moles of ethylene oxide per mole of octylphenol (trade name Triton X-114) contributed by Dow Chemical Inc. (South Charleston, USA) was used as the nonionic surfactant in this study. The CMC of the surfactant is reported by the supplier as 100 ppm or 0.186 mM at 25°C. Reagent grade 99.8% pure toluene were purchased from Carlo Erba Inc. (Italy). All chemicals were used as received. Distilled water was used for solution preparation.

#### 6.3.2 Apparatus

Figure 6.1 shows a schematic of the cloud point extraction pilot unit used in this work. The CPE apparatus consisted of a pyrex glass cylindrical column with a 1000 mm height, covered by an acrylic water jacket with a 149.2 mm inside diameter through which temperature controlled water was circulated. The extractor column had a mixing zone in the middle and settling or empty zones at either end of the column. The empty zones were implied in order to lengthen the residence time of the dilute and the coacervate stream before leaving at the top and bottom of the column. The heights of the mixing zone and empty zone were 700 mm and 150 mm, respectively. The rotor discs, stators, and shafts made of 316-stainless steel, were assembled and welded as a complete set before installation in the column, and fixed

by the teflon-supporter and screws at the either neck of the column. The agitator speed of the rotating discs was regulated by a speed adjustable motor. In the mixing zone, there were 32 horizontal rotor discs 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 were 33 annular stator rings with an outer and inner diameter of 29.2 mm and 20.44 mm, respectively, and 1 mm in thickness. The compartment spacing between stators was 22 mm. In order to study the effect of surfactant feed location on CPE, the surfactant solution was initially fed at different number of rotor discs along down the column as shown in the figure.



**Figure 6.1** Schematic of the cloud point extraction pilot unit.

### 6.3.3 Procedures

The contaminated feed water containing toluene as the trace pollutant and the surfactant solution were fed into the extractor countercurrently. The heavy surfactant solution was fed into the top, while the light wastewater was fed into the bottom of the extractor. The coacervate phase was beaten into tiny drops as rotating discs induced shear and these drops settled down to the bottom of the column; while

the dilute phase or the treated water was ejected at the top of the column. In all experimental runs, triplicates of the feed wastewater and the dilute phase samples were carefully collected, by transferred them to the vials and sealing them with aluminum caps and rubber septa coated with polytetrafluoroethylene (PTFE) to avoid a leakage of solutes. Due to the high volatility of toluene, the toluene concentration of the inlet wastewater was closely monitored every 45 minutes to ensure that it was unchanged for the entire operating time and that there was no significant loss of the solute by volatilization before feeding into the column. When the system reached steady state, as indicated by the absence of change in solute concentration in the dilute phase with time, triplicate samples were carefully collected from the effluent dilute phase and the coacervate phase. The concentrations of nonionic surfactant and solute were measured in order to evaluate the extraction performance of the column. Moreover, the flow rate of the dilute phase stream and the coacervate phase stream were determined by measuring the volume of the dilute phase and coacervate phase collected over a measured time interval.

The concentrations of OP(EO)<sub>7</sub> and toluene were measured by using a CE 2000 series UV-spectrophotometer (Cecil Instrument Limited, Cambridge, England) at 224 nm and a gas chromatograph with a flame ionization detector (Agilent Technology, USA), respectively. The conditions of the gas chromatograph for toluene determination were as follows: capillary column: HP-5; carrier gas: helium with a flow rate of 15 mL/min; make up gas: ultra pure nitrogen with a flow rate of 30 mL/min; oven temperature: 120 °C; injector temperature: 200 °C; and detector temperature: 300 °C. The external standard quantitative calibrations were obtained for the analysis of surfactant and toluene in both phases. Closure of the material balance was taken as evidence that leakage of toluene was negligible.

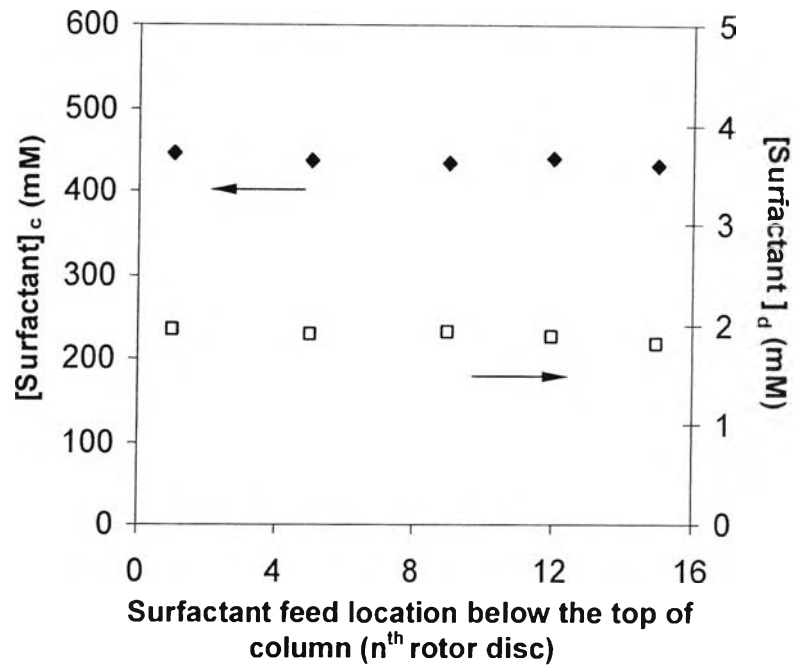
The external standard quantitative calibrations were measured for the analysis of surfactant and solute concentrations in both phases. Closure of the material balance (error of less than 10 percent) was taken as evidence that leakage of the volatile solute was negligible. For any particular studied conditions, the experiments were performed at least two times to ensure reproducibility, and the experimental data were averaged to yield the reported values. The standard deviation is less than ±5%.

## 6.4 Results and discussion

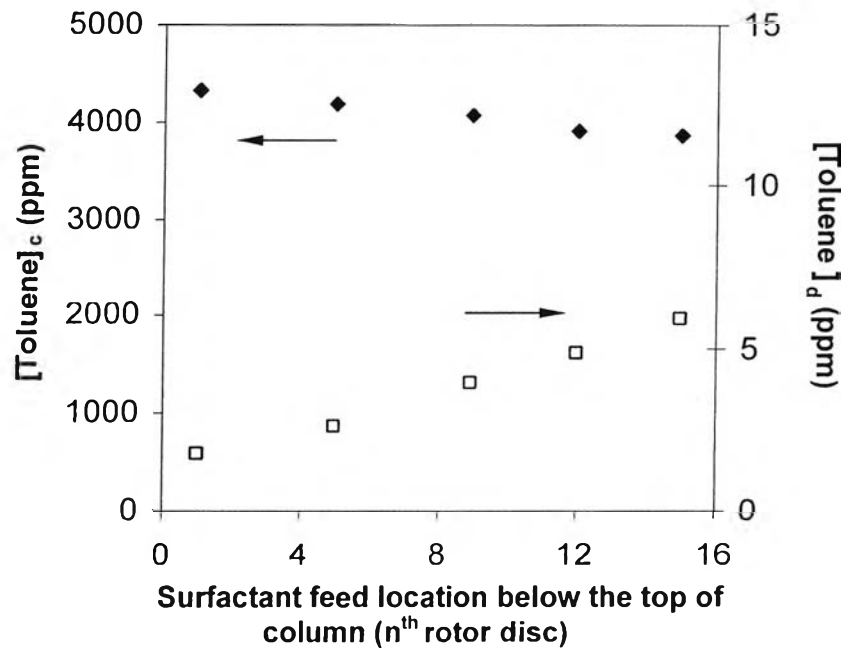
### 6.4.1 Effect of surfactant feed location on CPE and surfactant entrainment.

In order to investigate the effect of surfactant feed location on the efficiency of CPE, the surfactant and toluene concentration in dilute phase and coacervate phase, surfactant and toluene partition ratio, percentage of total surfactant in coacervate stream and percentage of toluene extracted were plotted as a function of surfactant feed location (at 1<sup>st</sup>, 5<sup>th</sup>, 9<sup>th</sup>, 12<sup>th</sup> and 15<sup>th</sup> rotor disc: from total of 32 discs) along down the column as shown in Figures 6.2, 6.3, 6.4 and 6.5. The operating conditions were as follows: operating temperature: 40 °C; concentration of surfactant in surfactant feed solution: 350 mM; concentration of solute in wastewater feed: 250 ppm; rotating disc speed: 100 rpm; wastewater/surfactant solution flowrate ratio: 12.65 where the flow rate of wastewater was fixed at 18.6 mL/min. The effect of surfactant feed location was firstly proposed to resolve an entrainment problem, since we predicted that the lower in the surfactant feed position could longer the residence time of the fine coacervate droplets leaving the column with the dilute phase stream. Hence, it might allow them to settle and retain inside the column.

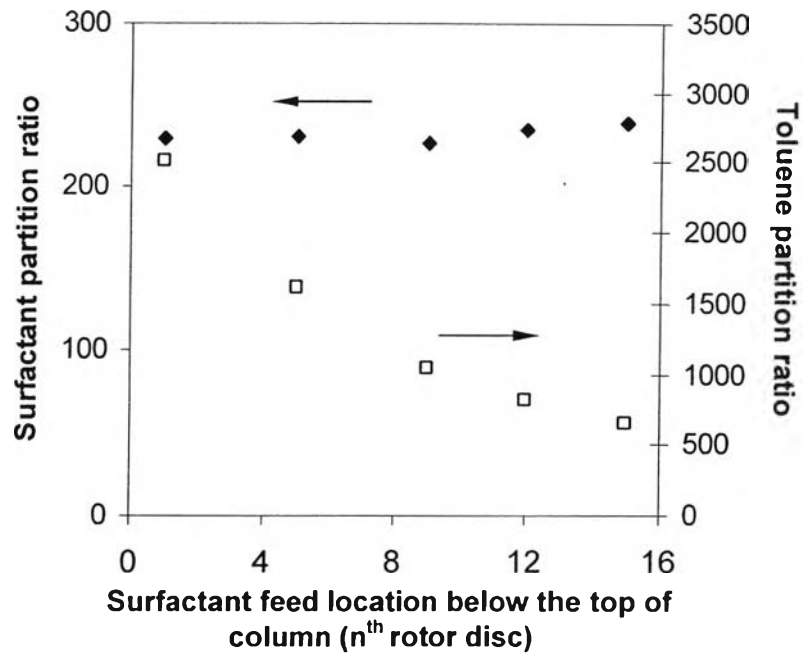
However, it was found from the study that the surfactant feed location did not have any significant effect on the reduction of the surfactant entrainment to the dilute phase; since, there was no significant change of the surfactant concentration in the coacervate and dilute phase in spite of the great difference of its surfactant feed location (1<sup>st</sup> to 15<sup>th</sup> rotor disc) (Figure 6.2). Conversely, we found that the lower the surfactant feed location resulted in the lower toluene concentration in dilute phase and the toluene extraction. The toluene concentration in the dilute phase increased from 1.73 ppm with the feed location at 1<sup>st</sup> rotor disc to 5.91 ppm with the feed location at 15<sup>th</sup> rotor disc (about half of the column). Since the lowering of the surfactant feed location leading to the lower in the residence or contact time of the coacervate droplets with the wastewater inside the column, toluene in the wastewater stream was less solubilized into the coacervate stream. This was evidenced by a decrease in the toluene concentration in the coacervate phase with the surfactant feed location (Figure 6.3). The partition ratio is the ratio of the solute or surfactant concentration in the coacervate phase to that of in the dilute phase. Figure 6.4, surfactant partition ratios showed insignificant change with the surfactant feed



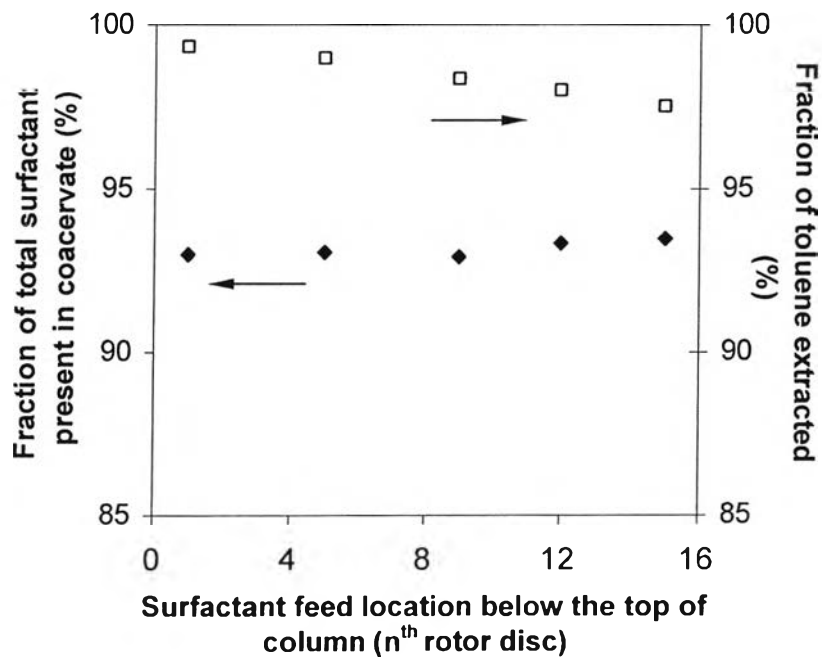
**Figure 6.2** Surfactant concentration in coacervate stream (c) (closed symbol) and dilute phase stream (d) (open symbol) as a function of surfactant feed location.



**Figure 6.3** Toluene concentration in coacervate stream (c) (closed symbol) and dilute phase stream (d) (open symbol) as a function of surfactant feed location.



**Figure 6.4** Surfactant partition ratios (closed symbol) and toluene partition ratios (open symbol) as a function of surfactant feed location.

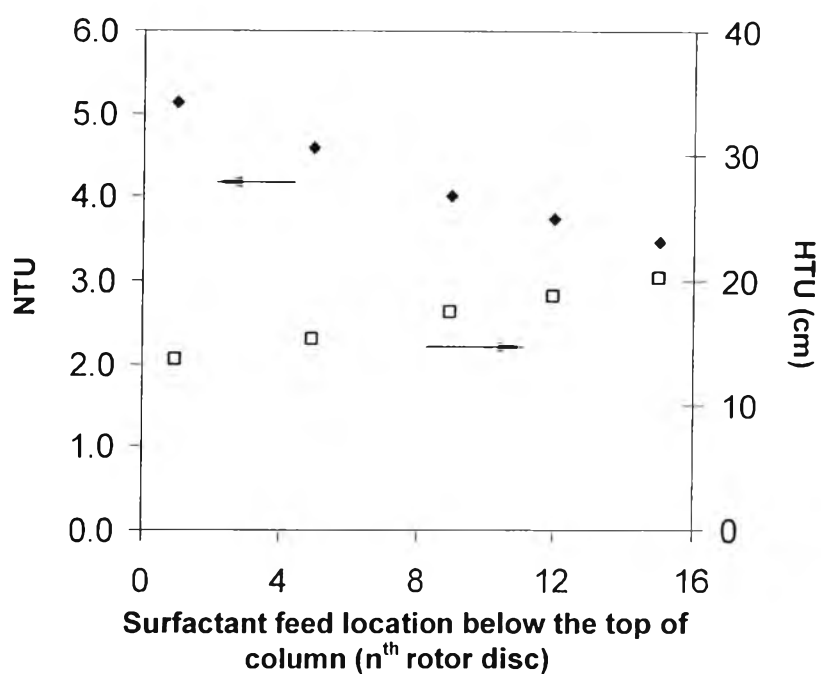


**Figure 6.5** Fraction of total surfactant present in coacervate stream (closed symbol) and fraction of toluene extracted in coacervate stream (open symbol) as a function of surfactant feed location.

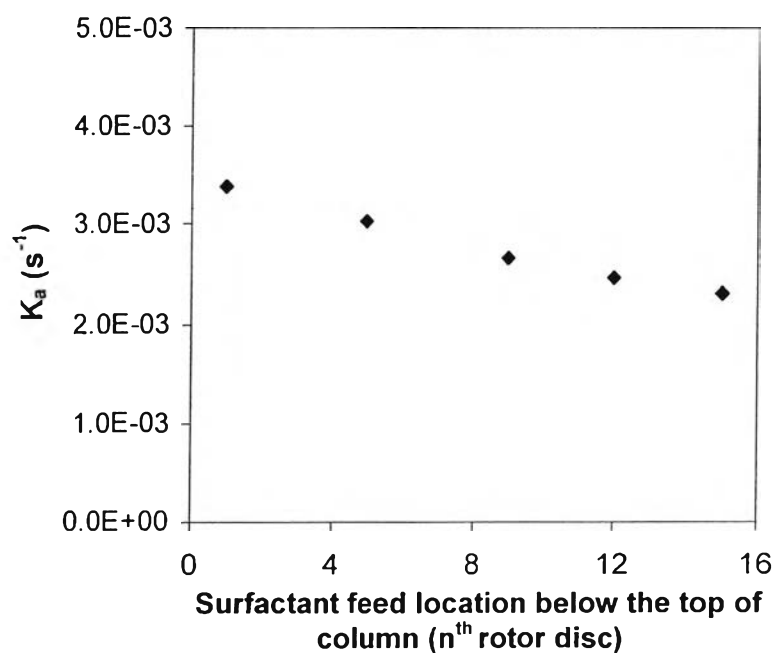


location, but toluene partition ratio drastically decreased with it. Also, the percentage of toluene extracted decreased from 99.3% at 1<sup>st</sup> rotor disc to 97.5% at 15<sup>th</sup> rotor disc, while the total surfactant in the coacervate stream remained almost constant as presented in Figure 6.5.

The height of transfer unit (HTU), the number of transfer unit (NTU), and the overall volumetric mass transfer coefficient ( $K_a$ ) were determined to evaluate the performance of the multi-stage RDC column. The HTU is the column height required to attain the separation which is equivalent to one equilibrium stage batch extraction and the NTU is the number of these single stage, batch extraction equivalents in the experimental column used. The HTU multiplied by the NTU equals the height of our experimental column (70 cm). Since, the lower the HTU or the shorter the column required when the higher the toluene partition ratio and the toluene extraction were obtained. The HTU was found to decrease by the feed location at higher position. As shown in Figure 6.6 the HTU of 20.2 cm per transfer unit from the feed location at 15<sup>th</sup> rotor disc was decreased to 13.7 cm per transfer unit from the feed location at 1<sup>st</sup> rotor disc. This result confirmed that the longer the contact time of coacervate effluent stream and wastewater, the better the solute extraction in a continuous unit. On the other hand, the NTU decreased with the lowering of the feed location, from 5.12 transfer unit from the feed location at 1<sup>th</sup> rotor disc to 3.46 transfer unit from the feed location at 15<sup>th</sup> rotor disc. The overall volumetric mass transfer coefficient or  $K_a$  also declined by the lowering of the surfactant feed location as shown in Figure 6.7. From all of the results, it could be investigated that even the time of the coacervate droplets in the column could be lengthen by the lowering of the surfactant feed location. This still did not enough to help the agglomeration of coacervate droplets to settle down, and could not help the reduction of surfactant entrainment, whenever the wastewater continuously flowed against upward.



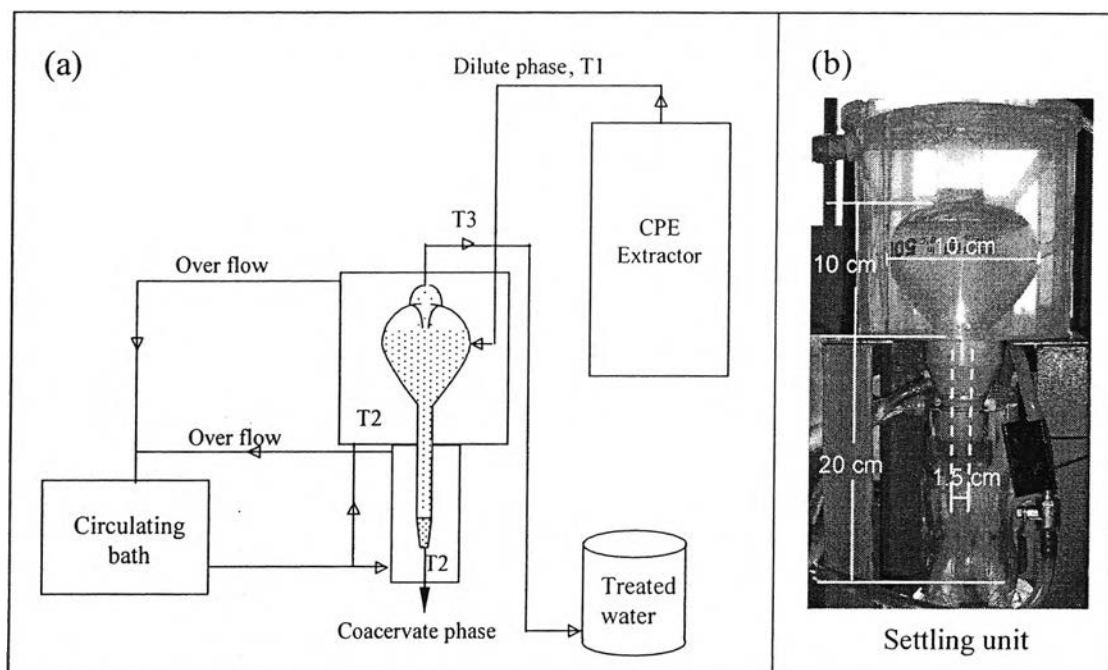
**Figure 6.6** Number of transfer unit (NTU) (closed symbol) and height of transfer unit (HTU) (open symbol) as a function of surfactant feed location.



**Figure 6.7** Overall volumetric mass transfer coefficient ( $K_a$ ) as a function of surfactant feed location.

#### 6.4.2 A study on an application of coacervate settling unit incorporation for treating the surfactant entrainment in the dilute phase after CPE.

The coacervate settling unit made of glass and designed in the shape of a separating funnel, was covered by a water jacket for controlling the temperature of the dilute phase stream after the extraction. Similar to a separating funnel, the unit has the shape of cone surmounted by a hemisphere, where the dilute phase after the extraction was fed and overflowed at the top of the unit. The cone shape area was used as an accumulation zone for accumulating the dispersed coacervate droplets in solution, helping the fine coacervate droplets to agglomerate and form the bigger and denser particles. The free settling velocity of the droplets, then, was enhanced due to the reduction of the frictional or buoyancy force; hence, the separation of the coacervate droplets from the dilute phase could be improved. The long rod joined between the lower part of cone with a stopper at its bottom was used as a settling zone. This was to retain the settled droplets and prevent the droplet disturbance or resettled. A process flow diagram and picture of the settling unit with its dimensions are shown in Figure 6.8. The total volume of the settling unit is about 450 ml. The dilute phase stream entering the settling unit (inlet dilute phase stream) and the dilute phase leaving the unit (outlet dilute phase stream) were taken every 45 minutes for 3 hours, and the concentration of surfactant in the inlet and outlet dilute phase stream was determined for evaluating the performance of the settling unit. The temperatures of system were monitored during the experiments. The temperature of the inlet dilute phase stream (T1) was constantly around 38 °C. The temperature of the controlled water in the water jacket (T2) was set as desired. When the system reached the steady state, T2 was considered as the settling temperature. The temperature of the outlet dilute phase stream was designated as T3. In this study, the operating conditions of the CPE column were maintained as follows: operating temperature: 40 °C; concentration of surfactant in feed solution: 350 mM; rotating disc speed: 100 rpm; wastewater/surfactant solution flow rate ratio: 10.88 where the flow rate of wastewater was fixed at 16.0 mL/min.

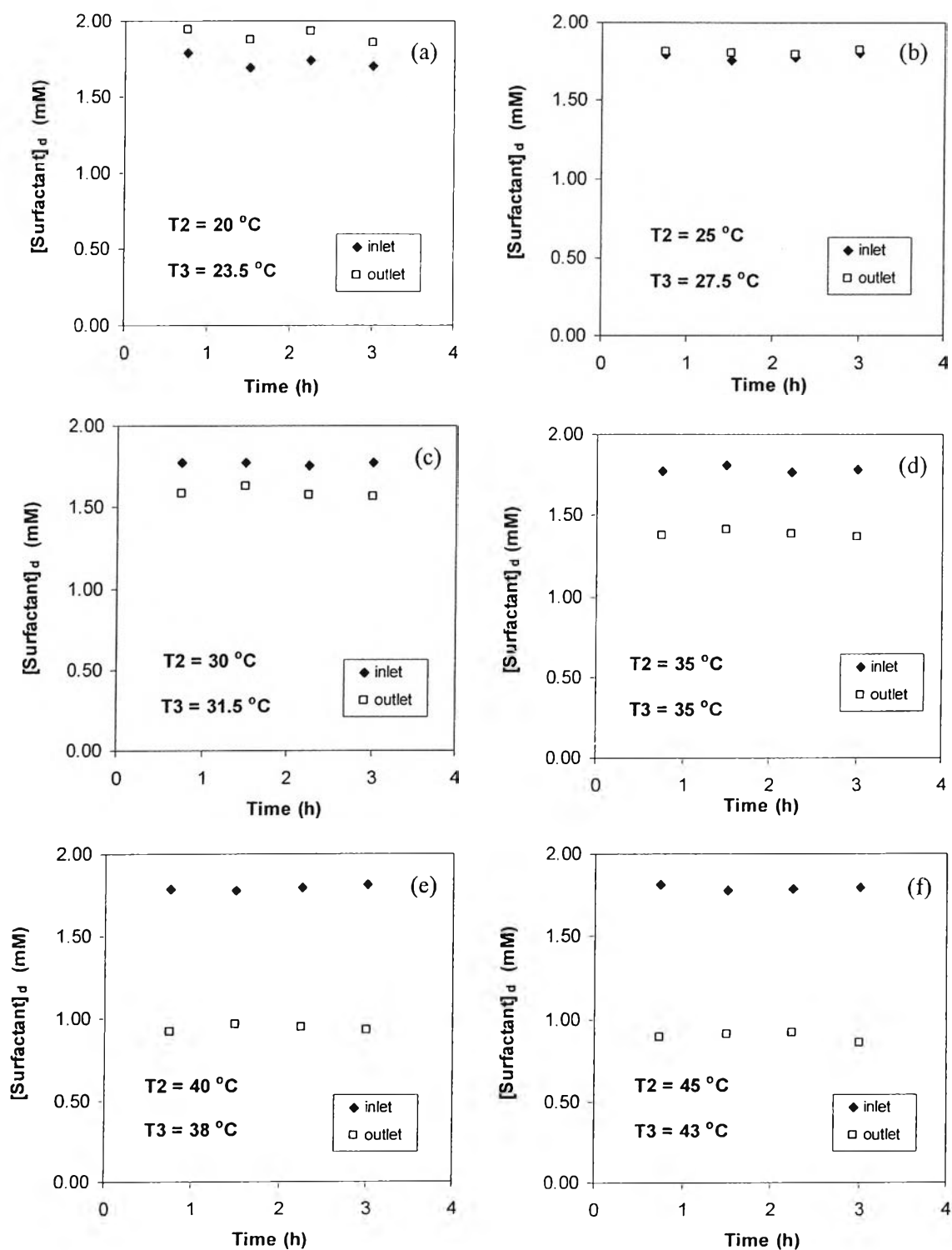


**Figure 6.8** Process flow diagram (a) and picture (b) of the coacervate settling unit.

#### 6.4.2.1 Effect of settling temperature.

In this study, the dilute phase after extraction or the inlet dilute phase stream was fed into the unit with a fixed flow rate of 16.0 ml/min, corresponding to the residence time of the dilute phase stream inside the unit of 28 minutes; whereas, the settling temperature of the settling unit was varied from 20 to 45 °C. The comparison of the concentrations of surfactant in both inlet (closed symbol) and outlet (open symbol) dilute phase streams as a function of operating time at different settling temperatures is illustrated in Figure 6.9.

Since the operating conditions of the CPE column were kept unchanged, the concentrations of surfactant in the inlet dilute phase stream from all experiments in the figure presented almost the same and constant for an entire operating time (average surfactant concentration = 1.80 mM). When the settling temperature was low at 20 and 25 °C, there was no separation of droplets investigated. Also, it was unexpectedly found that the surfactant concentrations in the outlet dilute phase stream were higher than in the inlet stream (Figure 6.9(a) and (b)). It seemed that even the coacervate droplets were much accumulated in the



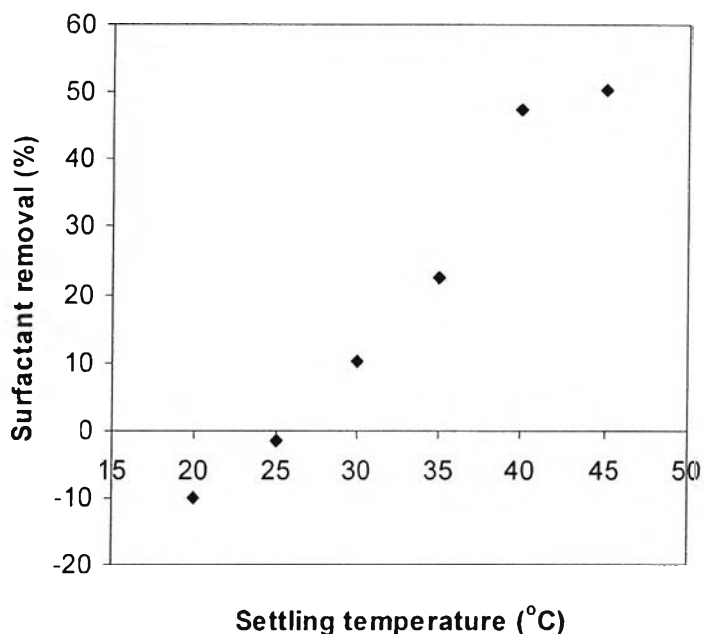
**Figure 6.9** Surfactant concentration in the inlet and outlet dilute phase as a function of operating time at different settling temperatures (T2).

accumulation zone; however, there was no settling of the droplets allowed at these temperatures. Hence, the droplets were entrained into the dilute phase stream overflowed. At the settling temperature of 30 °C and higher, the separation occurred and enhanced as the temperature increased. This could be seen from a consistently decrease of the surfactant concentration in the outlet dilute phase compared to the inlet dilute phase stream with higher settling temperature as shown in Figure 6.9(c), (d), (e) and (f), respectively. At 30 °C, the surfactant concentration of 1.80 mM (9.6 times of its CMC) in the inlet dilute phase was slightly decreased to 1.58 mM (8.5 times of its CMC) after the separation; whereas, at 40 °C, the surfactant concentration was greatly reduced to 0.92 mM (5.0 times of its CMC) (Figure 6.9(c) and (e)).

The effect of temperature on the droplets separation was also investigated by plotting the percentage of surfactant removal as a function of the settling temperature (Figure 6.10). The percentage of surfactant removal was calculated by a following equation:

$$\text{Surfactant Removal (\%)} = \frac{[\text{Surfactant}]_d \text{ inlet} - [\text{Surfactant}]_d \text{ outlet}}{[\text{Surfactant}]_d \text{ inlet}} \times 100$$

The percentage of surfactant removal consistently increased, when the settling temperature increased until 40 °C. At 45 °C, the percentage of surfactant removal was only slightly increased as compared with 40 °C. A substantial increase of surfactant removal was found when the settling temperature was from 35 to 40 °C. With only 5 °C increments, the percentage of surfactant removal increased from 22.4 to 47.5%. The minus value of the percentage of surfactant removal was generally obtained at 20 and 25 °C by the definition of the calculation, since the surfactant concentration of the outlet dilute phase was higher than that of the inlet dilute phase. As the cloud point of the studied t-octylphenolpolyethoxylate surfactant was approximately 25 °C, the poor separation obtained from the system operated at temperature lower or close to the cloud point was reasonable.



**Figure 6.10** Percentage of surfactant removal as a function of settling temperature.

At a high temperature of 40 °C, the temperature of the system which was much higher than the cloud point, could greatly enhance the settling rate of the coacervate. The coacervate of the high temperature system would be less water-soluble, more concentrated, and tend to separate more easily from the dilute phase. When the coacervate droplets were induced for an agglomeration, the denser coacervate droplets were allowed to settle at a faster rate. As the density of the aqueous dilute phase also decreases with increasing temperature, this might be another reason for an improvement of the separation. It should be noted that, the settling temperature of 40 °C might be considered a high enough temperature than the cloud point. Hence, for which the system of temperature higher than 40 °C, the only slightly increased in the separation was observed, as all of these explanations showed no more enhanced effect. Moreover, since the energy utilization at extensively high temperature should be concerned, the temperature of 40 °C was considered as an optimum temperature in this study.

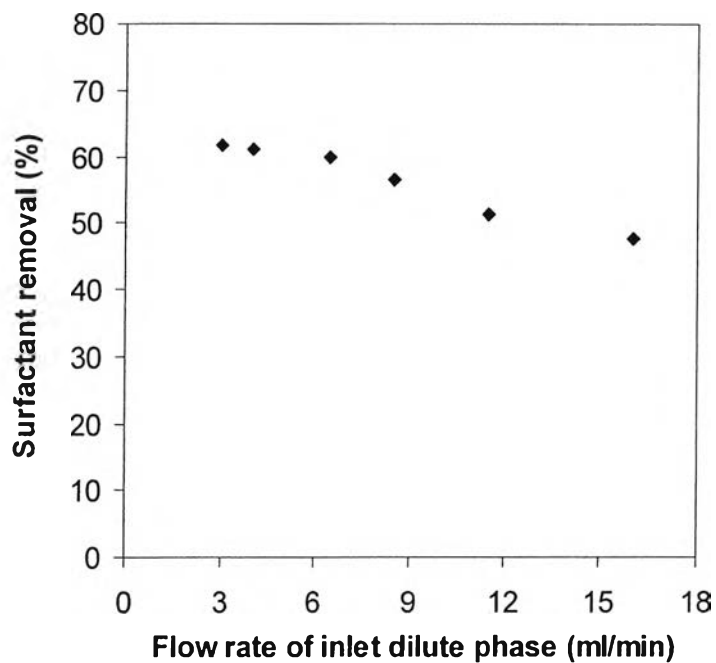
#### 6.4.2.2 Effect of residence time.

In order to design the settling unit, the flow rate and residence

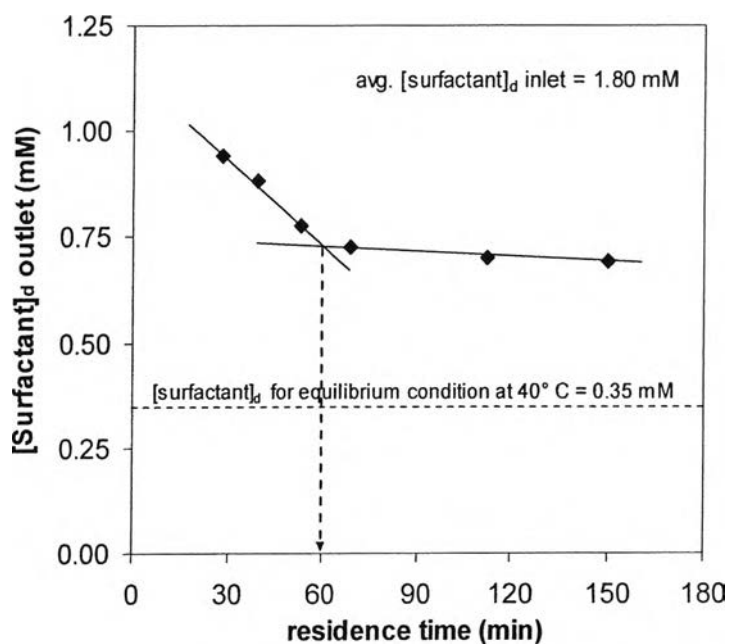
time of the stream through the settling system are the important factors needed to be concerned. For the investigated system, the proper flow rate of the dilute phase stream should be sufficiently slow to allow coacervate droplets to settle out. If the dilute phase flow is too high, the light or fine particles would not settle out and be discharged. A properly designed settling unit would reduce the stream flow and the turbulence, which will reduce the settling and disturb the settled droplets. In this study, the effect of residence time of the dilute phase stream in the settling unit was investigated, by varying the flow rate of the dilute phase stream, while the temperature of the unit was controlled at 40 °C. The lower stream flow rate or the longer residence time of dilute phase in the unit was examined in this study in order to enhance the efficiency of the separation. The flow rate of the inlet dilute phase stream before feeding into the unit was varied and regulated by the three-way valve for a desired flow rate.

The percentage of surfactant removal was plotted as a function of the flow rate of the inlet dilute phase, as shown in Figure 6.11. The result showed that the percentage of surfactant removal consistently increased from 47.5 to 59.9%, with decreasing the flow rate of the inlet dilute phase from 16.0 to 6.5 ml/min, or with increasing the residence time from 28 to 70 minutes. The slight increase in the separation efficiency was investigated when the flow rate was lower than 6.5 ml/min. The result of average surfactant concentrations in the outlet dilute phase after the separation plotted as a function of the residence time could clearly present the optimum residence time of the settling unit (Figure 6.12). From the figure, the effect of the residence time on the surfactant concentration in the outlet dilute phase stream could be divided into two ranges of residence time. At low residence time region, the surfactant concentration in the outlet dilute phase stream constantly decreased or the separation increased as the residence time increased. When the residence time further increased, the surfactant concentration in the outlet dilute phase stream appeared to insignificantly change (average concentration = 0.7 mM), which meant that the residence time no longer had effect on the separation. Some fine coacervate droplets still could not be settled out, even though the dilute phase stream was kept in the unit for the longer time. The separation efficiency of the unit was also limited so that the surfactant concentration in the treated dilute phase was impossible to be lower than





**Figure 6.11** Percentage of surfactant removal as a function of the flow rate of the inlet dilute phase.



**Figure 6.12** Surfactant concentration in the outlet dilute phase as a function of residence time.

the surfactant concentration in the dilute phase from the equilibrium batch experiment at 40 °C reported to be 0.35 mM. The optimum time was observed at the crossing point of the two straight lines of different residence time ranges and found to be approximately 60 minutes for which up to 60% of surfactant was removed.

## **6.5 Conclusions**

Surfactant feed location exhibited an insignificant result on the alleviation of the surfactant entrainment. The toluene extraction efficiency declined, however, due to the shorter contact time between the wastewater and coacervate stream. This study showed that the separation of coacervate droplets from dilute phase after extraction in the designed coacervate settling unit presented a high potential in solving a surfactant entrainment problem. An improvement in the efficiency of settling unit should be further studied. Multiple settling units should be applied in order to further treat and purify the dilute phase containing less concentrated coacervate droplets from the former unit. Effect of unit size and shape (volume, depth, width, length) should be adjusted and investigated. Other proper designs of settling unit could also be studied to minimize the settling disturbance.

## **6.6 Acknowledgements**

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