การแยกฟีนอลและโทลูอีนออกจากสารละลายผสมด้วยกระบวนการอัลตราฟิลเตรชันที่มี cetylpyridinium chloride เป็นตัวสร้างไมเซลล์

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บทคัดย่อ

งานวิจัยนี้ศึกษาผลการแยกสารอินทรีย์ระเหยได้ (VOC) 2 ชนิด คือฟีนอล และโทลูอีน โดยใช้สารลดแรงตึงผิวชนิดประจุบวกของซิทิลไพริดิเนียมคลอไรด์ (cetylpyridinium chloride; CPC) เพื่อสร้างไมเซลล์ และป้อนผ่านกระบวนการเยื่อแผ่นอัลตราฟิลเตรชันชนิดเซลลูโลสอะซีเทตแบบแผ่นเรียบ MWCO 10,000 การทดลองแบ่งออกเป็น 2 ส่วน คือ ส่วนที่ 1 สารละลายผสมระหว่างฟีนอลกับ CPC และโทลูอีนกับ CPC (ฟีนอล – CPC และโทลูอีน – CPC) และส่วนที่ 2 สารละลายผสมระหว่างฟีนอลและโทลูอีนกับ CPC (ฟีนอล – โทลูอีน – CPC)

ผลการแยกฟีนอลและโทลูอีนพบว่าฟลักซ์ของสารละลายผสมเพิ่มขึ้นเมื่อเพิ่มความดันป้อนและอัตราการไหลของระบบ แต่ฟลักซ์ของสารละลายผสมลดลงเมื่อเพิ่มความเข้มข้นของ CPC ลงในสารละลาย เมื่อพิจารณาค่าการกักกันพบว่าเมื่อเพิ่มความเข้มข้นของ CPC ลงในสารละลายผสม จะทำให้ค่าการกักกันของทั้งระบบฟีนอล – CPC และระบบโทลูอีน – CPC เพิ่มขึ้นจากร้อยละ 70 ถึง ร้อยละ 99 โดยประมาณ สำหรับระบบฟีนอล – โทลูอีน – CPC พบว่าฟลักซ์ของสารละลายผสมลดลงเมื่อเพิ่มความเข้มข้นของ CPC ค่าการกักกันของฟีนอลเพิ่มขึ้นเมื่อเทียบกับระบบที่มีเฉพาะฟีนอล (ฟีนอล – CPC) ในขณะที่ระบบมีสารละลายโทลูอีนผสมอยู่ แต่สำหรับค่าการกักกันโทลูอีนไม่เปลี่ยนแปลงเมื่อเทียบกับระบบที่มีเฉพาะโทลูอีน (โทลูอีน – CPC)

เมื่อพิจารณาผลของความสามารถในการละลายของฟีนอลและโทลูอีนในไมเซลล์พบว่าเมื่อเพิ่มความเข้มข้นของทั้งฟีนอลและโทลูอีน ทำให้ความสามารถในการละลายในไมเซลล์ลดลง และเมื่อเปรียบเทียบความสามารถในการละลายระหว่างฟีนอลและโทลูอีนในไมเซลล์พบว่าโทลูอีนสามารถละลายเข้าไปในไมเซลล์ได้มากกว่าฟีนอล ซึ่งสอดคล้องกับค่าการกักกันที่พบว่าโทลูอีนมีค่าการกักกันสูงกว่าฟีนอล

คำสำคัญ : อัลตราฟิลเตรชัน / ฟีนอล / โทลูอีน / ซิทิลไพริดิเนียมคลอไรด์ / การละลาย

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Separation of Phenol and Toluene from Aqueous Solutions by Micellar-enhanced Ultrafiltration using Cetylpyridinium Chloride

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Abstract

The feasibility of micellar-enhanced ultrafiltration (MEUF) for separation of volatile organic compound (VOC) was investigated. Cationic surfactant, cetylpyridinium chloride (CPC) was used for the formation of micelles. The VOCs selected were an ionic compound (phenol) and a non-ionic compound (toluene). The experiments were carried out by a flat sheet UF module using cellulose acetate membrane with MWCO 10,000. MEUF experiments were divided into two systems: single organic-CPC and binary organics-CPC systems.

The results showed that flux increased with increasing applied pressure and flow rate for single organic-CPC system, but the presence of CPC reduced flux in both phenol-CPC and toluene-CPC system. The rejections of phenol and toluene (70 to 99%) in phenol-CPC as well as toluene-CPC system were observed to increase with increasing CPC concentration. In phenol-toluene-CPC system, it appeared that flux decreased with increasing CPC concentration, but the rejection of phenol was increased by the presence of toluene while the rejection of toluene was not affected by phenol.

The extent of solubilization decreased with an increasing concentration of both phenol and toluene. Solubilization of toluene in CPC micelles was more than that of phenol, therefore, toluene rejection was higher than phenol rejection.

Keywords : Micellar-Enhanced Ultrafiltration / Phenol / Toluene / Cetylpyridinium Chloride / Solubilization

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1. Introduction

Micellar-enhanced ultrafiltration (MEUF) is the extended separation by ultrafiltration membranes which makes use of the micellar properties of surfactant solutions for removal of organics and/or dissolved ions from aqueous streams [1]. Potential applications of MEUF for separations of toxic metal ions and organics have been studied by a number of researchers. Among these works phenol and phenol compounds removal by MEUF has been the subject of interest due to its toxicity [2-4]. Toluene is another toxic organic that may be present in aqueous waste streams and proper treatment is required. There has been no report on MEUF of toluene solutions. The followings summarize previous works which have demonstrated efficient separation of phenols by MEUF.

Dunn and Scamehorn [2] studied the removal of 4-tert-butylphenol (TBP) using n-hexadecylpyridinium chloride (CPC) as the surfactant. Overall rejection of TBP was greater than 99% and did not increase with increasing pressure drop. Micelles were also completely rejected. Flux and retention analysis during MEUF of phenol and aniline using CPC as a surfactant was reported [3] for a single solution of CPC and for solubilization studies but separately for phenol and aniline. Solubilization equilibrium constant for phenol in CPC micelles was approximately four times that of aniline. While CPC, the cationic surfactant, has been shown to be effective for separation of phenols the use of other surfactants such as sodiumdodecyl sulfate (SDS-anionic), oxyethylated methyl dodecanoate (OMD-5 and OMD-9, non-ionic), and hexadecyltrimethyl ammonium bromide (CTAB-cationic) was also studied [4]. The use of mixed surfactants can also improve the separation efficiency provided that the proper ratios of the surfactant concentration were employed.

Although MEUF has been used to separate organic pollutants, heavy metal using various surfactants, however, most of research on MEUF has focused on the single-pollutant system, and few researchers reported a multi-pollutant system [5]. In the aqueous solutions in which both polar and non-polar organics are present the solubilization of each solute in the surfactant micelles can be different and may affect separation. The focus of this work is to investigate flux and rejection characteristics of MEUF of single solutions of phenol and toluene and their mixture. The parameters influencing the solubilization and separation were taken into consideration.

2. Materials and methods

CPC, toluene, and phenol were all analytical grade supplied by Sigma Chemical (USA), Labscan (Ireland), and Merck (Germany), respectively. In the preparation of all samples the predetermined amount of solute was solubilized in reverse osmosis and deionized water to obtain the required concentration. The mixture was continuously stirred for 15 min before being fed into the membrane module.

The experiments were carried out at room temperature (25°C) by a flat sheet crossflow minitan module of Millipore previously described [6]. The experimental set-up is shown in Fig. 1. The membrane was cellulose acetate, PLGC with MWCO (molecular weight cut off) 10,000 and a surface area of 60 cm^2. For each experimental condition, the permeate (10 ml) was collected at intervals to monitor flux and was quickly returned to the feed reservoir (2 l) after the sample was drawn for analysis. The parameters varied were phenol, toluene, and CPC concentrations, feed flow rate, and the applied pressure. After each run which lasted 90 to 120 min the membrane was rinsed with RO water and was later cleaned by 0.1 M NaOH to restore the initial water flux. The typical water flux at room temperature and at 98.07 kPa was 31.76 $l/m^2.h$.

Phenol concentration was determined by UV-VIS spectrophotometer (Hitachi, model U-3000) at a wavelength of 700 nm [7]. A gas chromatograph (Shimadzu, model GC-14B+C-R7A) using a BX-10 column (3m×33mm.) was used for determination of toluene concentrations. CPC concentrations were measured by a conductivity meter (Satorious, model PP-50) [8].

The surfactant concentration was chosen as 1.40, 2.79, 13.97, and 27.93 mM; the feed pressure was varied to be at 98.07, 147.10, and 196.14 kPa; and the flow rates were at 4, 9, and 14 l/min. During experiments, two variables are held constant while the third was varied.

The solubilization studies were carried out by varying concentrations (separately for phenol and toluene) from 0.6 to 1.0 mM at a surfactant concentration of 2.79 mM and at a constant applied pressure of 98.07 kPa and flow rate of 9 l/min.

The flux data reported herein were the averages obtained from 2 membrane pieces being used under the same experimental conditions which showed negligible differences. The analysis of samples were carried out in triplicates.

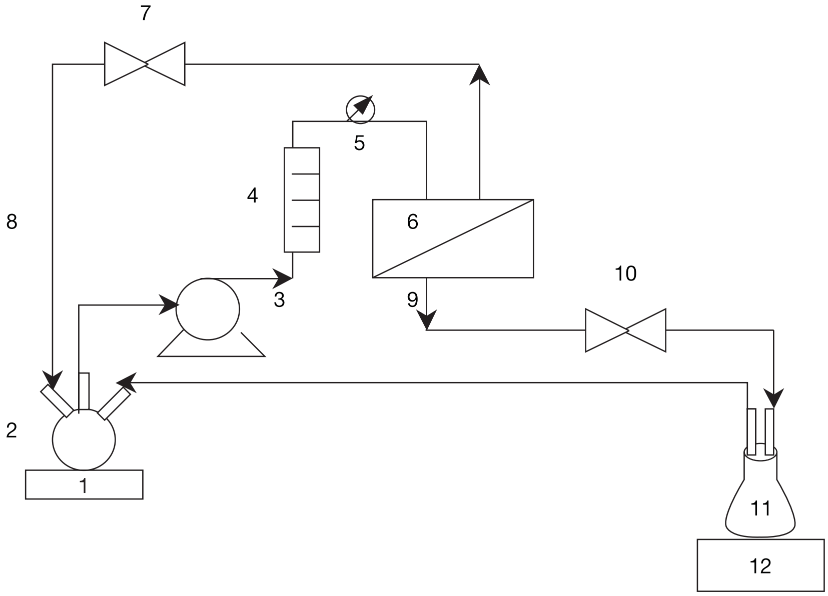

Fig. 1 The schematic diagram of the experimental set-up: (1) magnetic stirrer, (2) feed tank, (3) circulating pump, (4) flow meter, (5) pressure guage, (6) membrane module, (7) pressure control valve, (8) retentate, (9) permeate outlet, (10) pressure control valve, (11) permeate collector, (12) analytical balance.

3. Analysis

A conceptual schematic diagram of MEUF for the removal of organic solutes is shown in Fig. 2.

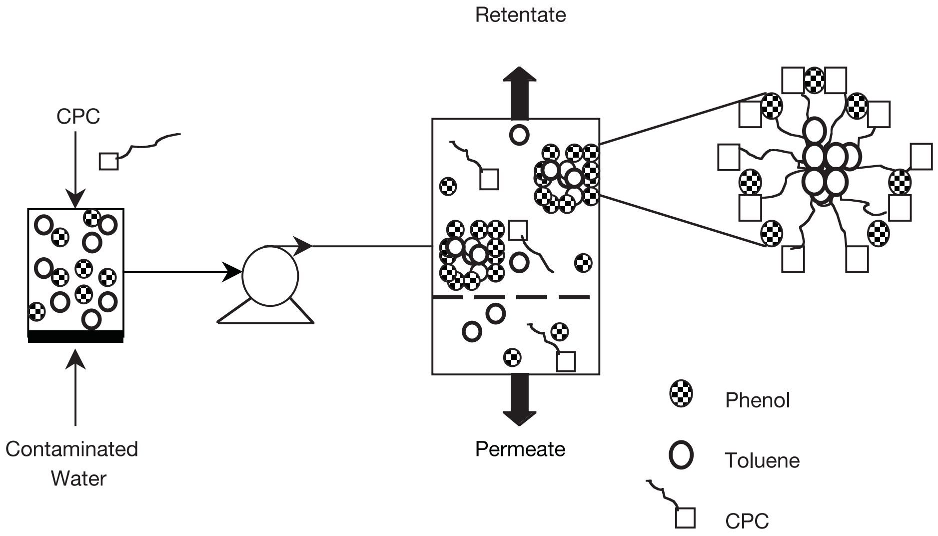

Fig. 2 A schematic diagram of micellar-enhanced ultrafiltration for removal of phenol and toluene.

The hydraulic membrane resistance (R_m) is obtained from the linear relationship of pressure versus water flux data which can be obtained by the following equation:

$$J_w = \frac{\Delta P}{\mu_w R_m} \quad \textbf{(1)}$$

where : J_w = water flux ($m^3/m^2.s$)

ΔP = applied pressure (Pa)

μ_w = viscosity of water (Pa.s)

R_m = membrane resistance (m^{-1})

The permeation flux of the solution can be calculated from the following equation;

$$J_v = \frac{\Delta P - \Delta\pi}{\mu_v R_t} \quad \textbf{(2)}$$

where : J_v = volumetric flux ($m^3/m^2.s$)

ΔP = applied pressure difference (Pa)

$\Delta\pi$ = osmotic pressure difference (Pa)

μ_v = viscosity of solution (Pa.s)

R_t = total resistance (m^{-1})

The permeation, therefore, is depended upon the applied driving force (ΔP - $\Delta\pi$) and other operating parameter, such as flow rate and concentration.

The rejection of each solute was determined from the following equation;

$$R = \left[1 - \left(\frac{C_P}{C_R}\right)\right] \times 100 \quad \textbf{(3)}$$

where : R = rejection (%)

C_P = solute concentration in the permeate

C_R = solute concentration in the retentate

The amount of solute solubilized in the surfactant aggregates was obtained from the mass balance

$$C_O V_O = C_R V_R + C_P V_P + M_{micelle} \quad \textbf{(4)}$$

where : C_o = initial (feed) solute concentration

V_o, V_R, and V_P = initial volume, retentate volume, and permeate volume, respectively.

C_oV_o, C_RV_R, C_PV_P, and $M_{micelle}$ are the amounts of solutes present initially, in the retentate, in the permeate, and solubilized in the micelles, respectively.

4. Results and discussion

The membrane was initially tested for pure water flux at 25°C and its resistance was determined to be 1.2×10^{13} m^{-1}. Fluxes and rejections of single solute solutions were also determined at the concentrations 0.65 mM and 1.09 mM for phenol and toluene and at 1.4 mM to 27.93 mM for CPC. For phenol and toluene solutions, fluxes and rejections were of the same order of magnitude, i.e., they were 20-30 l/m^2.h and 3-5%, respectively. The membrane cannot retain both solutes because their molecular weights (94 and 92 g/mol for phenol and toluene, respectively) are much lower than MWCO of the membrane.

The range of CPC concentrations studied (1.4-27.93 mM) was much higher than the CMC value (0.88 mM) [9]. It was, therefore, expected that most CPC molecules were in the form of micelles. The flux variation with the surfactant concentration in the feed (Fig. 3) revealed that flux decreased from 20 l/m^2.h to 5 l/m^2.h. This is because at higher CPC concentration the effect of concentration polarization becomes more significant. This polarization possibly created a CPC concentration greater than CMC (critical micelle concentration) level. Individual molecules tended to aggregate as micelles. Furthermore, an increase in the feed concentration may also enhance both the number and size of micelles at the membrane surface [9]. The high concentration of CPC in the retentate can increase the osmotic pressure ($\Delta\pi$). When CPC concentration increased from 1.4 to 27.93 mM, the osmotic pressure significantly increased from 6,800 to 69,000 Pa. Hence the flux was decreased due to the reduced driving force.

Because the rejection of the surfactant depends on the presence of the micelles in the feed solutions (Fig. 3). It was found that the permeate concentration of CPC decreased from 0.67 to 0.34 mM with increasing CPC concentration. This result means that CPC micelles were large enough not to pass the membrane pore, but on the other hand free monomers of CPC can pass the membrane because its molecular weight is lower than MWCO of the membrane. Theoretically, the concentration of CPC in permeate can be increased to near CMC, 0.88 mM. Baek et al. [10] reported analogous results on the rejection of CPC and permeate concentrations at below the CMC (0.7 to 0.75 mM).

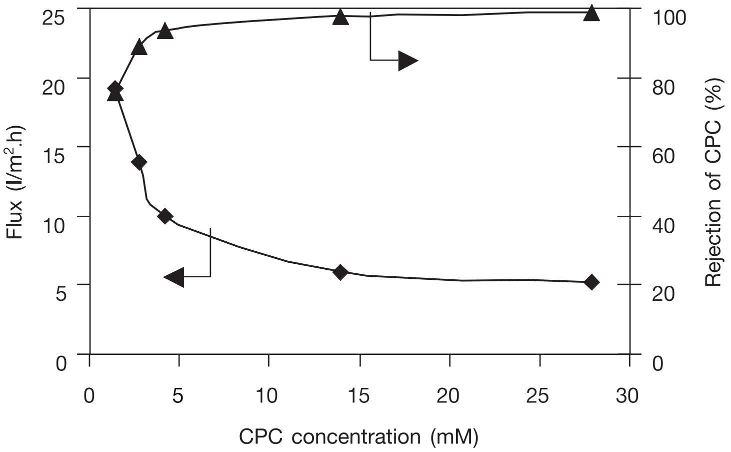

Fig. 3 Variation in flux and rejection with CPC concentration.
ΔP = 98.07 kPa and flow rate = 9 l/min.

4.1 The effect of operating parameters on flux and rejection of phenol-CPC system

The flux of phenol-CPC solutions for different applied pressures and flow rates is shown in Fig. 4. From this figure, it can be observed that flux decreased in the beginning due to concentration polarization but the trend was to approach constant flux. The same trend was also presented by Syamal et al. [11]. From the results, it can be explained that flux depended upon the combined effects of applied pressure and osmotic pressure. With the increase of pressure, the effective driving force for the transport of the solvent is high, hence, flux was increased, but the rejection decreased (Fig. 5). It was likely that micelles may be distorted and changed to smaller sizes and part of phenol molecules penetrated the membrane.

The effect of flow rate on flux is presented in Fig. 4. Higher flow rate resulted in minimizing the concentration polarization at the membrane surface by increasing the diffusion of micelles away from the membrane surface towards the bulk. When flow rate was increased, CPC rejections tended to decrease (Fig. 5). It was possible that under high shear near the membrane surface the micelles were deformed. Therefore, both phenol and CPC molecules were free and can pass the membrane.

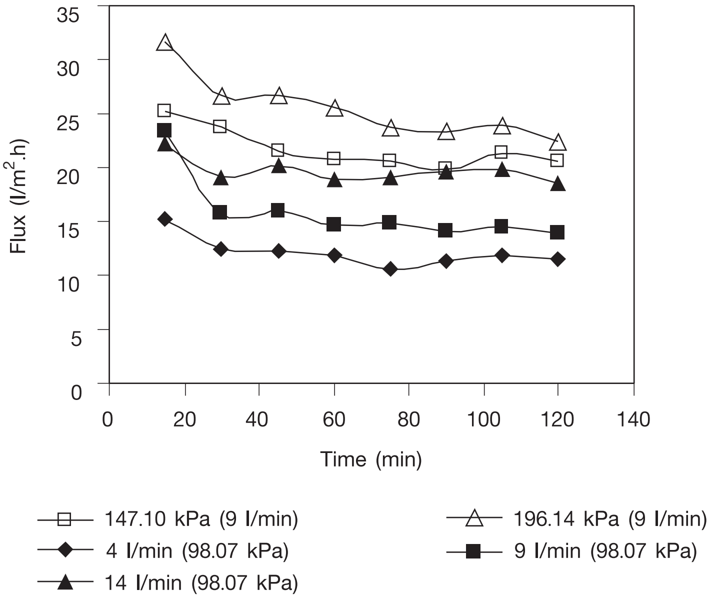

Fig. 4 Variation in flux with time at different pressures and flow rates. CPC and phenol concentration = 2.79 and 0.64 mM, respectively. Pressures (kPa): white symbols and flow rates (l/min): black symbols.

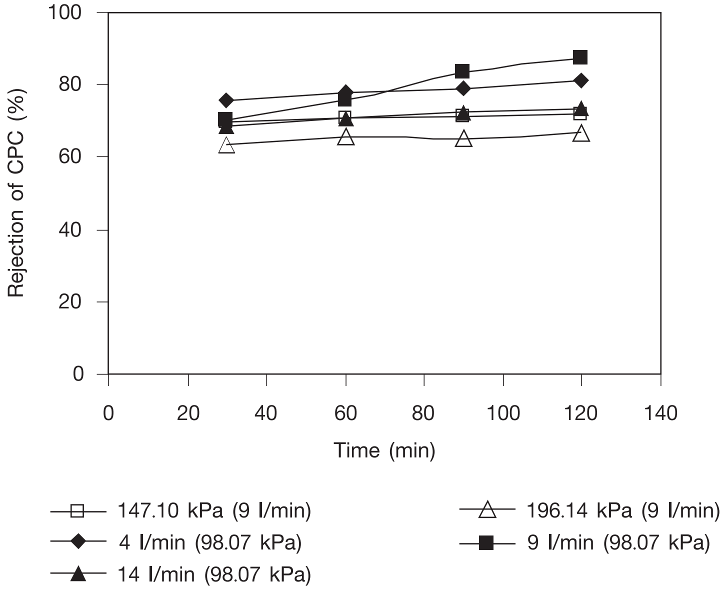

Fig. 5 Variation in rejection with time at different pressures and flow rates. CPC and phenol concentration = 2.79 and 0.64 mM, respectively. Pressures (kPa): white symbols and flow rates (l/min): black symbols.

4.2 Solubilization of organic solutes within the micelles

The solubilization study of phenol (counter-ion) and toluene (non-ionic) in CPC micelles was for observing the influence of organic solutes and their concentrations on the permeate organic concentrations and for comparing the extent of solubilization of phenol and toluene within CPC micelles.

The results (Fig. 6) are plotted for permeate concentrations of organic solutes as a function of time. The concentration of CPC was kept constant at 2.79 mM for both solutes. It was observed from the figure that the permeate phenol and toluene concentrations decreased with increasing operation time. This was because of increasing of extent of solubilization of organic solutes in micelles. It can be stated that the extent of solubilization increased with time.

At 2 hrs. of operation, the extent of solubilization in both organic solutes (Fig. 7) was at 1.11 and 0.81 mmole for phenol 0.64 mM and 1.06 mM, respectively and 1.29 and 1.25 mmole for toluene 0.65 mM and 1.09 mM, respectively.

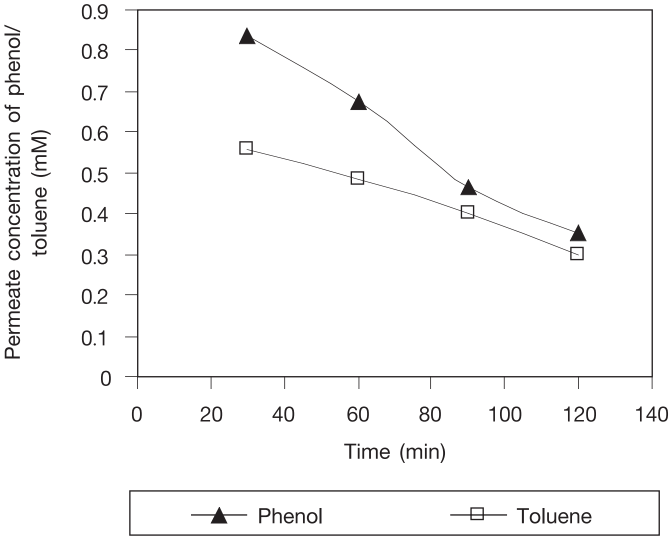

Fig. 6 Variation in permeate solute concentrations during MEUF. CPC concentration = 2.79 mM. Phenol and toluene concentration = 0.64 and 0.65 mM, respectively. ΔP = 98.07 kPa and flow rate = 9 l/min.

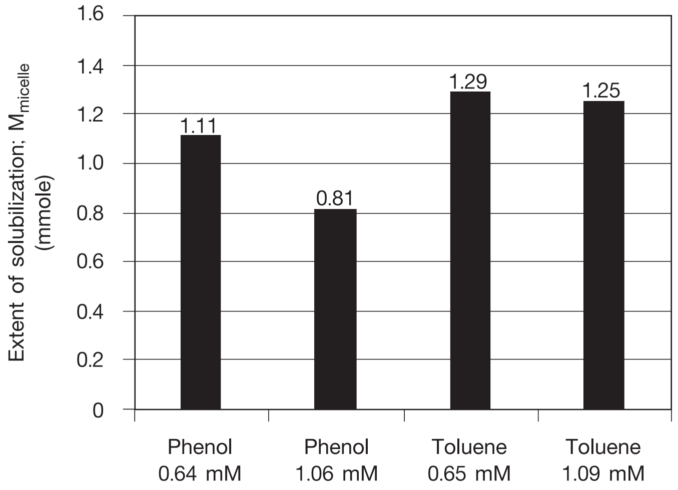

Fig. 7 Variation of the extent of phenol and toluene solubilization. CPC concentration = 2.79 mM. ΔP = 98.07 kPa and flow rate = 9 l/min.

From the above results, it revealed that the extent of solubilization decreased with increase in initial concentration in both solutes. It was obvious from the result that solubilization capacity generally decreases with increase in the molar volume (high concentration) of phenol and toluene, which corresponded with the study of Gadelle et al. [12]. The molecular weight of phenol was higher than that of toluene, therefore, the extent of phenol solubilization was lower than toluene.

The difference of solubilization values of both solutes results from the nature of the organic solubilized and the type of interaction occurring between the surfactant and the organic solutes. CPC micelles (cationic surfactant) can interact electrostatically with highly polar solutes (phenol) because the large surface charge densities of these aggregates lead to strong ion-dipole interactions. In addition, CPC micelles ordinarily have an extensive hydrophobic-core region which can interact strongly with hydrocarbon groups of solutes like toluene. Hydrophobic effects have often been considered to be dominant in determining the locus of solubilization, but the effects of electrostatic interactions should also be considered in relation to the solubilization of organic solutes in CPC micelles. Jadhav et al. [3] had proposed the theory of the exact location in CPC micelles. In their work, anionic solute, phenol, was adsorbed on the surface of cationic CPC micelles. On the other hand, non-ionic solute, aniline, was solubilized into the interior of micelles (for normal micelles in aqueous medium), which corresponded with the data in this study. Phenol should be solubilized to a greater extent in the head-group region of CPC micelles than toluene (non-polar compound) that is located within the hydrophobic-core region. This is in agreement with Singh [8], who stated that the more hydrophobic the organic solute, the higher the degree of solubilization.

Fig. 8 presents the rejection of phenol as well as toluene. It can be stated that rejection of toluene (99.77% and 84.53%) was higher than phenol (84.55% and 69.88%). The permeate concentration was lower for toluene than phenol, which compared well with the results in Fig. 6. Singh [8] also reported that the rejection of phenol with CA membrane was lower than toluene. One possible reason for his low rejection was that non-solubilized phenol was preferentially sorbed at the CA membrane–solution interface and, therefore, passed through the membrane with water. Therefore, from the above results, it may be concluded that highly hydrophobic compounds will be almost quantitatively removed from wastewater streams, whereas, compounds that are more soluble will ordinarily be less effectively removed by MEUF processes.

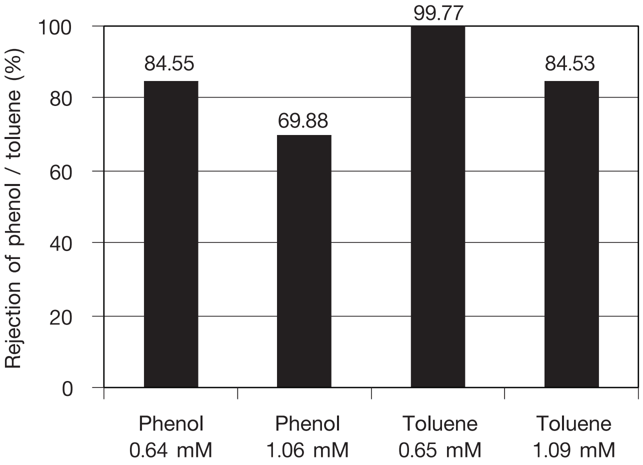

Fig. 8 Rejection of phenol and toluene. CPC concentration = 2.79 mM. ΔP = 98.07 kPa and flow rate = 9 l/min.

4.3 Flux and rejection of phenol-toluene-CPC system

Flux decreased from 3.79 l/m^2.h to 1.59 l/m^2.h as feed CPC concentration increased from 13.97 mM to 27.93 mM, respectively (Fig. 9). This was due to concentration polarization on the membrane surface. The rejection of phenol (Fig. 10) was higher than the rejection in phenol-CPC system (Fig. 8) and increased from 98.89% to 99.74% as the CPC concentration increased from 13.97 mM to 27.93 mM. The presence of toluene increased the phenol rejection significantly compared to phenol-CPC system. It can be explained that the presence of solubilized hydrocarbon (toluene) in the surfactant micelles increased the solubility of polar compounds (phenol) in these micelles. The solubilized hydrocarbon causes the micelle to swell or expand, and this may make it possible for the micelle to incorporate more polar compound within the micelles [9]. The rejection of toluene in this system is shown in Fig. 11. The rejection of toluene increased in proportional to feed concentration of CPC added in the system as shown in the toluene-CPC system. The rejection was similar to that in the toluene-CPC system. This means that the presence of phenol in the system did not affect the rejection of toluene. However, the rejection of toluene was still higher than phenol in phenol-toluene-CPC system (permeate toluene concentration less than in phenol case). Fig. 12 describes the CPC rejection as a function of concentrations. The tendency in CPC rejection was similar to that in phenol-CPC system, where the rejection was 93.69% at the CPC concentration of 13.97 mM. At CPC concentration of 27.93 mM, the rejection increased to 99.60%.

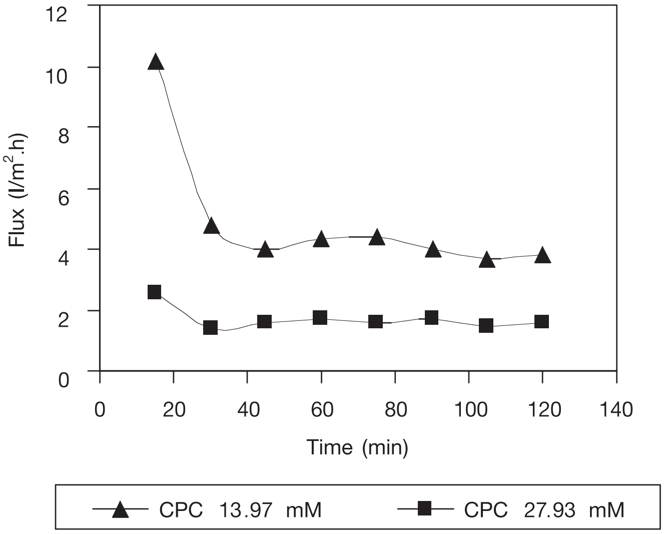

Fig. 9 Variation in flux of phenol-toluene-CPC system with time. Phenol and toluene concentration = 0.64 and 0.65 mM, respectively. ΔP = 98.07 kPa and flow rate = 9 l/min.

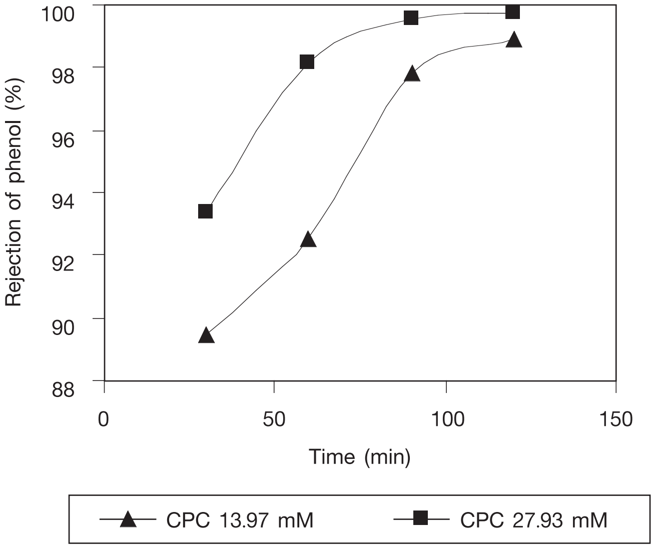

Fig. 10 Variation in rejection of phenol of phenol-toluene-CPC system with time. Phenol and toluene concentration = 0.64 and 0.65 mM, respectively. ΔP = 98.07 kPa and flow rate = 9 l / min.

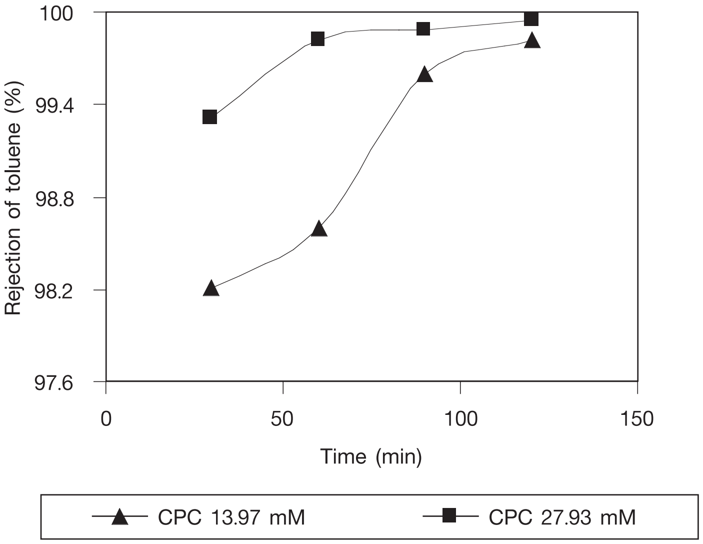

Fig. 11 Variation in rejection of phenol of phenol-toluene-CPC system with time. Phenol and toluene concentration = 0.64 and 0.65 mM, respectively. ΔP = 98.07 kPa and flow rate = 9 l/min.

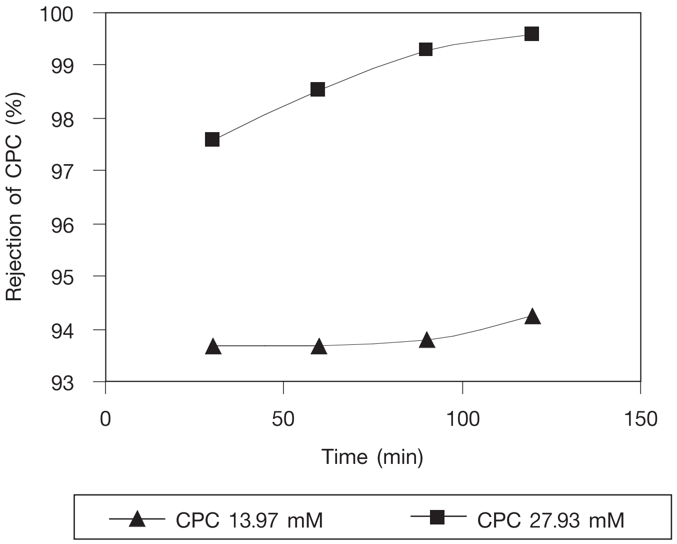

Fig. 12 Variation in rejection of CPC of phenol-toluene-CPC system with time. Phenol and toluene concentration = 0.64 and 0.65 mM, respectively. ΔP = 98.07 kPa and flow rate = 9 l/min.

5. Conclusions

Separations of volatile organic compounds (VOCs), phenol and toluene, by MEUF using cetylpyridinium chloride (CPC) were investigated. The following conclusions can be drawn.

For the single solutions, flux of phenol and toluene in the absence of CPC decreased with increasing bulk concentration. The rejections of both phenol and toluene were very low (3-5%) because the size of these molecules were much lower than MWCO of the membrane. The rejections of single CPC were higher than those of both phenol and toluene due to the formation of the micelles at the concentrations of CPC higher than the CMC.

For the solutions of single organic with the surfactant (CPC), flux increased with pressure and flow rate, but the rejections decreased. Flux also decreased with increasing CPC concentrations, but the rejections increased due to the increase in osmotic pressure. The extent of solubilization decreased with an increasing molecular volume of both phenol and toluene. Phenol was solubilized at the hydrophilic heads of cationic CPC micelles, whereas, toluene was preferentially solubilized in the hydrocarbon-core region of the CPC micelles. Therefore, toluene rejection was higher than phenol rejection.

The systems with the presence of both organics with CPC showed that the rejection of phenol was increased by the presence of toluene while the rejection of toluene was not affected by phenol.

In MEUF of single organic solutes (phenol-CPC and toluene-CPC), with the increase of CPC concentrations (five to thirty times of CMC value), the permeate solute concentrations were estimated to be around 0.001-0.3 mM (0.094-28.2 ppm) which were lower than the initial concentration, 0.65 mM (60 ppm). In case of the phenol-toluene-CPC system, the permeate solute concentrations were also low enough (0.001-0.008 mM of phenol and toluene in the permeate) compared with the initial concentration. Moreover, the addition of toluene substantially improved rejection of phenol, whereas toluene rejection remained higher than 98%. The rejection of CPC was high and the concentration of CPC in the permeate was low, hence, the MEUF process could be an alternative for the treatment of aqueous solutions contaminated with phenol and toluene.

6. References

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