



## Removal of Nickel(II) and Chlorobenzene from Water by Micellar-Enhanced Ultrafiltration with Anionic/Non-Ionic Surfactant

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(Received: 16 April 2010;

Accepted: 30 November 2010)

AJC-9346

The effect of a non-ionic surfactant, Tween 80 (TW80), on nickel ion removal in the ultrafiltration enhanced by an anionic surfactant, sodium dodecyl sulfate (SDS), was investigated. The simultaneous removal of nickel ions and chlorobenzene using ultrafiltration in the presence of the mixed SDS-TW80s was then studied. The mass ratios of SDS to TW80 were 3:1, 1:1 and 1:3. In the ultrafiltration of nickel alone, the removal efficiencies of nickel by SDS or SDS-TW80s increased with surfactant concentrations. Given the total surfactant concentration, increasing the TW80 fraction in SDS-TW80s brought about reduction in nickel rejection. The rejection efficiencies of nickel by surfactants are in the sequence of SDS > 3:1 SDS-TW80 > 1:1 SDS-TW80 > 1:3 SDS-TW80. In the rejection of both chlorobenzene and nickel, the removal efficiencies of nickel by SDS-TW80s are in order of 3:1 SDS-TW80 > 1:1 SDS-TW80 > 1:3 SDS-TW80. The maximum removal percentage of nickel is up to 84.7%. The removal efficiencies of chlorobenzene by SDS-TW80s are in order of 1:3 SDS-TW80 > 1:1 SDS-TW80 > 3:1 SDS-TW80. The maximum removal percentage of chlorobenzene is 86.4%. The flux reduction by surfactants and the effects of pH values and sodium chloride concentrations in feed solutions were also investigated. The results indicate that anionic-non-ionic surfactants could be potential in micellar-enhanced ultrafiltration for the removal of both organics and metal ions.

**Key Words:** Micellar-enhanced ultrafiltration, Nickel, Chlorobenzene, Mixed surfactant.

### INTRODUCTION

Micellar-enhanced ultrafiltration (MEUF) is an effective separation technique to remove metal ions and/or soluble organic solutes from water<sup>1</sup>. The advantages of this method are low energy needed for the treatment process and high removal efficiency due to the effective interaction between solutes and micelles. Metal ions bind to the charged surface of micelles by electrostatic interaction while organic solutes partition into the core of micelles. The micelles (5-10 nm) with contaminants are enough to be rejected effectively by ultrafiltration membrane.

The removal of anionic or cationic metal ions by MEUF, such as Zn(II), Cu(II), Pb(II), Cd(II), Ni(II)<sup>2-6</sup>, Cr(VI)<sup>7</sup>, As(V)<sup>8</sup>, Au(III)<sup>9</sup> and so on was reported. In general, anionic surfactants (for example, sodium dodecyl sulfate, SDS) are used to remove cationic ions while cationic surfactants (for example, cetyltrimethylammonium bromide, CTMAB and cetylpyridinium chloride, CPC) to remove anionic metal ions such as CrO<sub>4</sub><sup>2-</sup> and AsO<sub>4</sub><sup>3-</sup><sup>7,8</sup>. However, one concern with the use of ionic surfactants for ultrafiltration is the high surfactant concentration in the permeate. Non-ionic surfactants can not be utilized to remove metal ions due to no attraction of their micelles to charged metal ions<sup>9</sup>. The studies on MEUF for organic

pollutants mainly focus on phenols<sup>10</sup>, aromatics<sup>11</sup>, dyes<sup>12</sup>, chlorinated compounds<sup>13,14</sup> and so on. Because the organic solutes are removed through partitioning into the core of micelle, the rejection efficiencies of organics depend on the solubilization capacity of the used surfactant. In general, non-ionic surfactant has low critical micelle concentration (CMC) and large solubilization capacity for organic compounds<sup>15-17</sup>. Non-ionic surfactant is potential to removal of organic solutes from wastewater using MEUF<sup>18</sup>.

Anionic and non-ionic surfactant solutions usually form mixed micelle aggregates that frequently exhibit properties remarkably different from those of the individual components<sup>19,20</sup>. Mixed surfactants can exhibit synergistic solubilization for organic compounds. Addition of small amount of non-ionic surfactant to an anionic surfactant usually results in a decrease in the CMC of the anionic-non-ionic systems, relative to the CMC of the pure anionic one. In addition, the capacity of anionic surfactant to bear hard water will be enhanced after addition of non-ionic surfactant. Those properties indicate the potential of anionic-non-ionic surfactant in MEUF application. In previous study<sup>18</sup>, the mixed SDS-TW80s were used to enhance the removal of chlorobenzene from water by MEUF. Given surfactant concentration of 5 g/L, the rejection efficiencies of

chlorobenzene by surfactants were in the order of 1:3 SDS-TW80 > TW80 > 1:1 SDS-TW80 > 3:1 SDS-TW80 > SDS.

Organic contaminants and heavy metal ions often occur together in wastewater. Although a single ionic surfactant can be used to remove organic and inorganic contaminants simultaneously<sup>21</sup>, the CMCs of ionic surfactants are generally larger than those of non-ionic surfactants. More monomers go through the membrane due to a large CMC of ionic surfactant. Thus, the mixed surfactant might be an alternative. A few studies have been conducted on the removal of cationic metal ions with mixed anionic-non-ionic surfactants<sup>22-25</sup>. The results show that anionic-non-ionic surfactant at an optimal ratio enhances the rejection efficiency of metal ion. The anionic surfactant dosage used and its concentration in permeate decrease. However, few studies have been done on simultaneous removal of metal ions and organic compounds using ultrafiltration enhanced by anionic-non-ionic surfactant<sup>26</sup>.

In the present study, SDS and TW80 are chosen as the representatives of anionic and non-ionic surfactants. The first objective is to study the effects of addition of TW80 on nickel removal and flux in SDS-enhanced ultrafiltration. The second objective is to optimize SDS-TW80 mixture in removing both nickel ions and chlorobenzene simultaneously from water. Finally, the effects of parameters such as pH values and sodium chloride concentration in feed solution on the rejection efficiency of nickel and chlorobenzene will be investigated. The results could provide an implication for simultaneous removal of metallic ions and organics from water through anionic-non-ionic micelle-enhanced ultrafiltration.

## EXPERIMENTAL

Nickel sulfate hexahydrate with analytical grade was obtained from Tianjin Kaitong Chemical Company, China and chlorobenzene with analytical grade from Tianjin Suzhuang Chemical Company, China. Sodium dodecyl sulfate (SDS) (> 99 % purity, 288.38 g/mol molar weight, 1586 mg/L CMC) was purchased from Tokyo Chemical Industry Kabuskiki Kaisha, Japan and Tween-80 (TW80) (>99 % purity, 1309 g/mol molar weight, 73.76 mg/L CMC) from Acros Organics, USA. All chemicals were used as received without further purification. The membrane used is a hollow fiber membrane of type UEOS-503, manufactured by Tianjin Motian Membrane Engineering Technology Company, China. The membrane dimensions ( $\Phi \times L$ ) are 50 mm  $\times$  366 mm. The membrane MWCO is 6000 and the effective filtration area is 1.5 m<sup>2</sup>. Deionized water was used in all experiments.

**Procedure:** The ultrafiltration runs were carried out at room temperature (around 25 °C) with the experimental set up in Fig. 1. Synthetic wastewater-containing nickel ions or both nickel ions and chlorobenzene was prepared and surfactants were added into the synthetic wastewater. The mixed SDS-TW80s were prepared at the mass ratios 3:1, 1:1 and 1:3 of SDS to TW80. The feed nickel concentration was 50 mg/L in all experiments and the chlorobenzene concentration was 100 mg/L. The feed solutions were agitated to provide efficient mixing and stood for 3 h (pre-experiment indicated that 3 h was needed for nickel ion adsorption equilibrium). 3 L of feed solution was filtrated at the transmembrane pressure (TMP)

of 0.07MPa in all experiments. The permeate and feed solution were sampled in the specified time. After each experiment the membrane was washed successively with deionized water, 0.01 mol/L HNO<sub>3</sub>, 0.1 mol/L NaOH, 1 % NaClO and deionized water under the pressure of 0.03 MPa. The deionized water was filtered to determine the permeate flux and to check the permeability of the membrane.

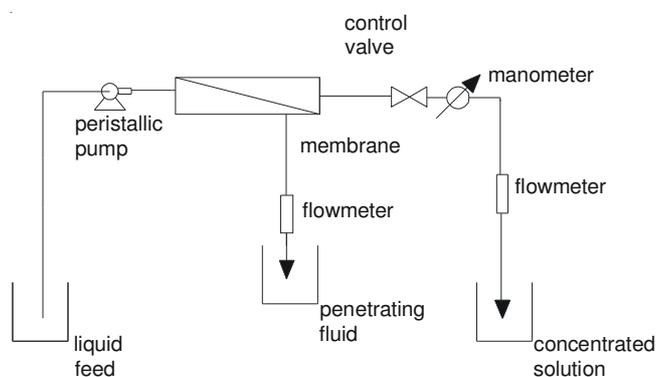


Fig. 1. Schematic of micellar-enhanced ultrafiltration process

The rejection efficiency of nickel or chlorobenzene was calculated as:

$$R (\%) = \left( 1 - \frac{C_p}{C_i} \right) \times 100 \quad (1)$$

where R is the rejection efficiency; C<sub>i</sub> (mg/L) and C<sub>p</sub> (mg/L) are the concentrations of nickel or chlorobenzene in the feed and in the permeate, respectively. The total rejection efficiencies of both nickel and chlorobenzene (R<sub>T</sub>) were calculated as:

$$R_T (\%) = \left( 1 - \frac{C_{p,CB} + C_{p,Ni}}{C_{i,CB} + C_{i,Ni}} \right) \times 100 \quad (2)$$

where C<sub>i,CB</sub> and C<sub>i,Ni</sub> are the concentrations of chlorobenzene and nickel in feed solution; C<sub>p,CB</sub> and C<sub>p,Ni</sub> (mg/L) the concentrations of chlorobenzene and nickel in permeate solution.

**Sample measurements:** For the samples containing nickel alone, the nickel ions were tested by dimethyl glyoxime spectrophotometric method at 530 nm with 1.0 cm quartz cell on spectrophotometer (Model 752, Shanghai Spectrum Instrumental Company, China). For the samples containing both nickel and chlorobenzene, two parallel samples were prepared. One of them was diluted to 25 mL with deionized and then chlorobenzene was tested at 210 nm with 1 cm quartz cell on spectrophotometer. Another one was used to analyze the nickel concentration by the method mentioned above. The concentrations of components were quantified from the calibrations.

## RESULTS AND DISCUSSION

**Rejection of nickel alone:** The relationship between the removal efficiencies of nickel ion and the concentrations of single SDS or mixed SDS-TW80s are shown in Fig. 2. When no surfactant is used, the removal efficiency of nickel is 19.6 %, due to the adsorption of membrane. By single SDS, the removal efficiencies of nickel increase with SDS concentrations. When the concentrations of SDS are changed from 0.5 to 5.0 g/L, the

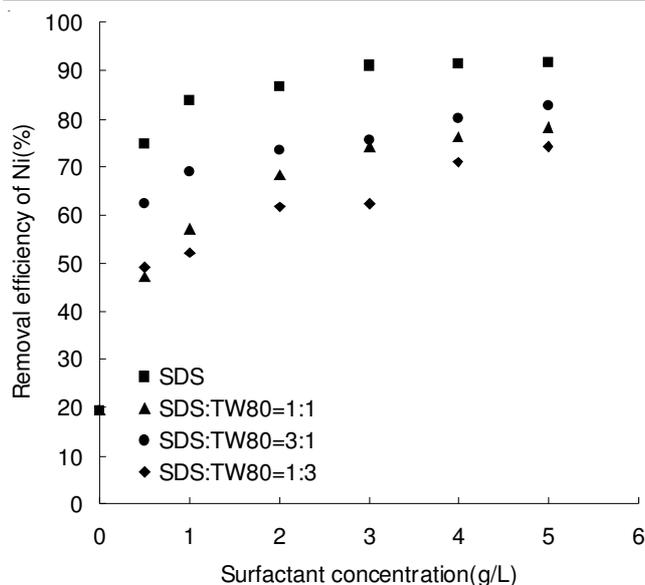


Fig. 2. Relationship between the removal efficiencies of nickel and surfactant concentrations in the ultrafiltration of nickel alone

removal of nickel gradually increases from 74.9 to 91.7 %. When SDS concentration is below its CMC obvious rejection is also found. It is believed that there may be an accumulation of SDS molecules on the membrane surface. The sodium dodecyl sulfate concentration in the gel layer can exceed the CMC and micelles can exist in the region. The region is called as concentration polarization layer<sup>27</sup>, which can hold metal ions by electrostatic interaction.

As for the mixed SDS-TW80s, the nickel rejection is also enhanced by surfactant addition (Fig. 2). However, significant reduction is found in rejection efficiencies by mixed SDS-TW80s compared with single SDS. When the concentrations of surfactants are larger than 1 g/L, the removal efficiencies by surfactants are in order of SDS > 3:1 SDS-TW80 > 1:1 SDS-TW80 > 1:3 SDS-TW80. Given 5 g/L of surfactant concentration, the per cent rejections by SDS, 3:1 SDS-TW80, 1:1 SDS-TW80 and 1:3 SDS-TW80 are 91.7, 82.7, 78.2 and 74.2 %, respectively. Therefore, the addition of non-ionic surfactant results in reduction of rejection. Obviously, the charges on micelles decrease as long as the mass fraction of non-ionic surfactant increases. It has been accepted that in a MEUF operation, cationic metal ions are rejected by the electrostatic attraction between metal ions and the anionic micelles. As the fraction of SDS in mixed surfactant decrease, SDS molecules in the mixed micelles decrease, which bring about that negative charges on the mixed micellar surface and nickel ion rejection decrease<sup>25</sup>.

The above results show that single SDS exhibits a significant enhancement for nickel removal. However, it is a concern that the rejection efficiency by single SDS for organics<sup>18</sup> is not effective due to its low solubilization capacity and the large CMC. Based on the results of nickel removal by SDS-TW80s above and chlorobenzene removal by SDS-TW80s in previous study<sup>18</sup>, the mixed SDS-TW80 could be expected to enhance organic and inorganic pollutants ultrafiltration simultaneously.

Fig. 3 shows the effects of single SDS and mixed SDS-TW80s on the flux in the procedure of nickel ultrafiltration.

The flux reduces with increasing the concentrations of surfactants. Polarization may be the reason for flux reduction<sup>10</sup>. When the concentration of surfactant is below its CMC, no micelle exists in the bulk solution. The surfactant concentration in layer adjacent to the membrane surface is higher than that in the bulk solution. When the concentration of surfactant is over its CMC, micelles are present in the bulk solution. Meanwhile, many micelles accumulate at the membrane surface and the membrane resistance increases. As shown in Fig. 3, the flux in the presence of surfactants is in order of 1:3 TW80-SDS > 3:1 TW80-SDS > 1:1 TW80-SDS ~ SDS. To some extent, the mixed SDS-TW80s are of advantage to flux.

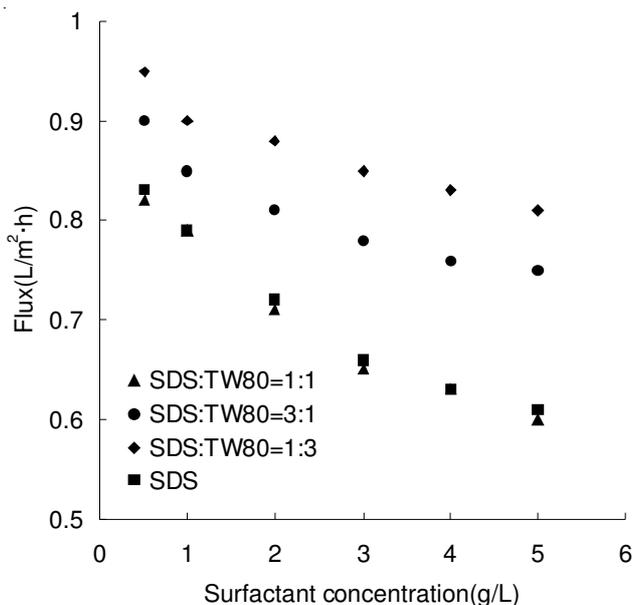


Fig. 3. Effects of surfactant concentrations on the flux in the ultrafiltration of nickel alone

**Rejection of both nickel and chlorobenzene:** The relationship between the removal efficiencies of chlorobenzene and the concentrations of the mixed SDS-TW80s is shown in Fig. 4. The rejection of chlorobenzene by SDS-TW80s increases with increasing SDS-TW80 concentrations. Given surfactant concentration, the rejection efficiencies decrease with the mass fraction of TW80 in mixed SDS-TW80s. When the surfactant concentrations are 5 g/L, the removal efficiencies of chlorobenzene by 1:3 SDS-TW80, 1:1 SDS-TW80 and 3:1 SDS-TW80 are 86.4, 85.2 and 83.5 %, respectively. These results are similar to those reported previously<sup>18</sup>, in which the single SDS, TW80 and the mixed SDS-TW80s at the mass ratios were used to enhance removal of chlorobenzene from water by the same ultrafiltration device. Given 5 g/L of surfactant concentration, the rejection efficiencies of chlorobenzene by surfactants were in order of 1:3 SDS-TW80 > TW80 > 1:1 SDS-TW80 > 3:1 SDS-TW80 > SDS. The mixed SDS-TW80s exhibit a relative high rejection for chlorobenzene due to their low CMCs and synergistical solubilization for hydrophobic organic compounds<sup>17,28</sup>. The synergistical solubilization is the condition when solubilization capacity of the mixture is better than those attainable with individual components by themselves. On the other hand, the intrinsic non-ionic surfactant has a large solubilization capacity. Thus, the more fraction of

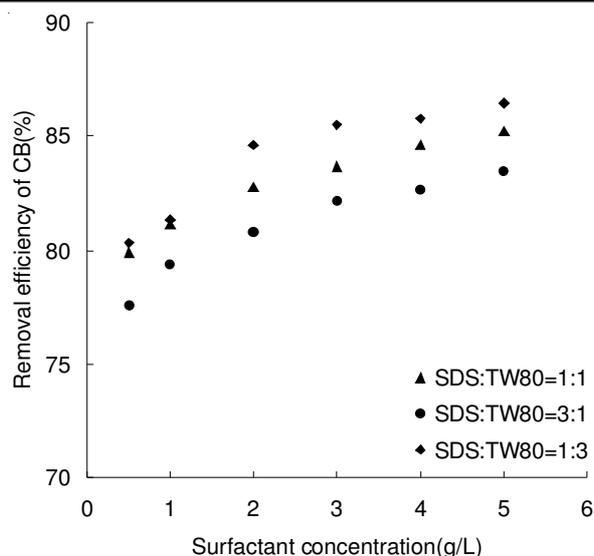


Fig. 4. Relationship between the removal efficiencies of chlorobenzene and the mixed surfactant concentrations in the ultrafiltration of both nickel and chlorobenzene

non-ionic surfactant in mixed surfactant, the higher rejection efficiencies for organics.

Fig. 5 shows the relationship between the removal efficiencies of nickel and the concentrations of mixed SDS-TW80s in simultaneous rejection processes. The tendency of removal efficiencies with mixed surfactant concentrations is similar to that in the ultrafiltration of nickel alone at large. However, the nickel rejection seemed to be enhanced slightly in the presence of chlorobenzene, which may be caused by a slight reduction in the CMC of surfactant by the addition of organic solute<sup>29</sup>. As shown in Fig. 5, it is also found that the removal efficiencies of nickel increase with the concentrations of mixed SDS-TW80s. The maximal removal of nickel is 84.7 % by 5 g/L 3:1 SDS-TW80. Given the surfactant concentration, the extent of nickel removal decreased in order of 3:1 SDS-TW80, 1:1 SDS-TW80 and 1:3 SDS-TW80.

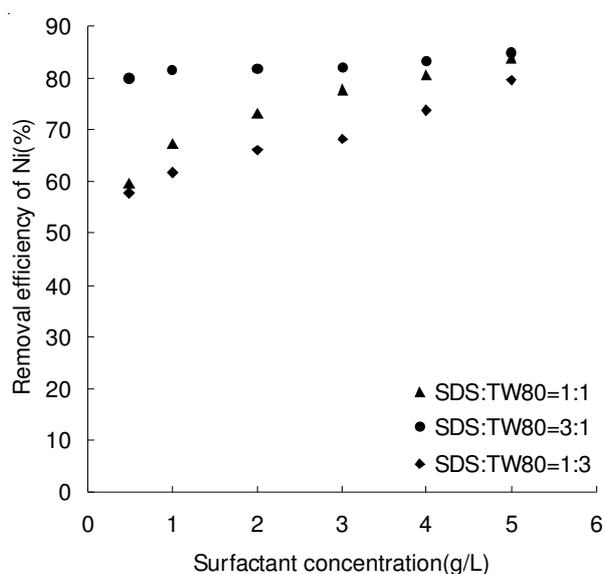


Fig. 5. Relationship between the removal efficiencies of nickel and the mixed surfactant concentrations in the ultrafiltration of both nickel and chlorobenzene

Table-1 lists the results of total removal efficiencies of contaminants by mixed SDS-TW80s. When SDS-TW80s are at low concentrations (0.5 and 1.0 g/L), the total rejection of nickel and chlorobenzene by 3:1 SDS-TW80 are larger than those by 1:1 and 1:3 SDS-TW80. Little difference is found in the total rejection by mixed SDS-TW80s at high concentration (4 and 5 g/L). To compare the effects of mixed SDS-TW80s on ultrafiltration of both nickel and chlorobenzene, 5 g/L of single SDS and single TW80 were also used to remove both nickel and chlorobenzene in ultrafiltration. The removal percentages for nickel, chlorobenzene and both nickel and chlorobenzene were 24.8, 80.0 and 61.6 % by single TW80 while 91.4, 68.1 and 75.9 % by single SDS. However, the CMC of SDS is quite higher than that of TW80 and those of mixed SDS-TW80s (see the following calculation). The CMC of the binary surfactant on the basis of ideality<sup>19</sup>,  $C_{12}^*$ , is expressed as

$$\frac{1}{C_{12}^*} = \frac{\alpha}{C_1} + \frac{1-\alpha}{C_2} \quad (3)$$

where  $\alpha$  is the mole fraction of surfactant 1 in the mixed system in the solution, *i.e.*, the mole fraction of surfactant 2 equals  $1-\alpha$ .  $C_1$  and  $C_2$  are the critical micelle concentrations of pure surfactant 1 and 2.  $C_{12}^*$  for 3:1, 1:1 and 1:3 SDS-TW80 are calculated as 0.72, 0.30 and 0.14 mmol/L (272.6, 155.9 and 112.2 mg/L), respectively, which are substantially less than that of pure SDS. As mentioned above, the large CMC of single SDS may lead to more monomers going through the membrane. Therefore, anionic-non-ionic surfactant system might be an alternative to remove cationic metal ions and organic solutes simultaneously from water by ultrafiltration.

TABLE-1  
TOTAL REMOVAL EFFICIENCIES ( $R_T$ ) OF BOTH NICKEL AND CHLOROBENZENE BY THE MIXED SDS-TW80s

Surfactant concentration (g/L)	$R_T$ (%)		
	3:1 SDS-TW80	1:1 SDS-TW80	1:3 SDS-TW80
0.5	78.4	71.8	71.6
1.0	80.2	75.6	73.5
2.0	81.2	79.2	77.8
3.0	82.0	81.5	79.1
4.0	82.8	83.1	81.6
5.0	83.9	84.7	83.9

The effects of SDS-TW80 concentrations on the flux are shown in Fig. 6. The flux reduces with increasing the concentrations of mixed SDS-TW80s and the extent of flux reduction by mixed SDS-TW80s is in order of 1:3 > 3:1 > 1:1.

**Effects of pH and NaCl on rejection of both nickel and chlorobenzene:** The effects of pH values of feed solution and sodium chloride (NaCl) concentrations in feed solution on ultrafiltration of nickel and chlorobenzene in the presence of 1:1 SDS-TW80 at 3 g/L concentration are shown in Figs. 7 and 8. The removal efficiencies of nickel increase with increasing pH values. With pH values changing from 2 to 9, the per cent removals of nickel increase from 51.2 to 85.2 %. When pH is less than 3, the competitive adsorption of  $H^+$  on micelle surface will significantly affect the removal of metal ions<sup>27</sup>. The removal efficiencies of chlorobenzene change slightly,

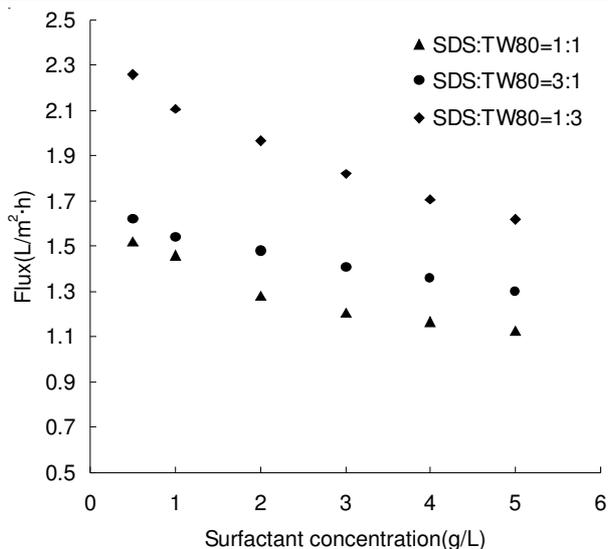


Fig. 6. Effect of the mixed surfactant concentrations on the flux in the ultrafiltration of both nickel and chlorobenzene

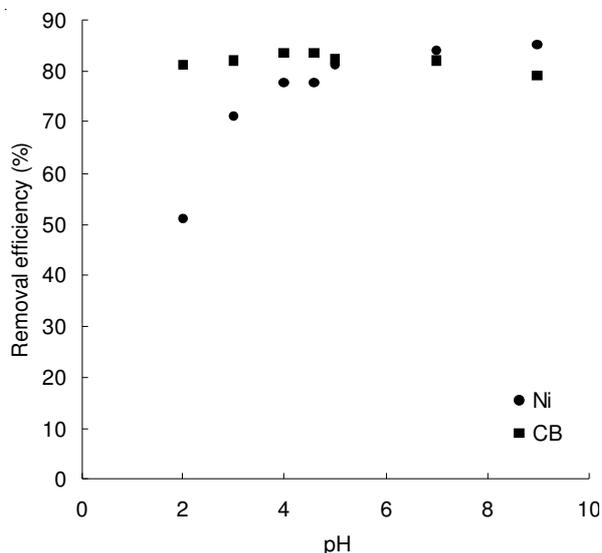


Fig. 7. Effects of pH values on the removal efficiencies of nickel and chlorobenzene enhanced by 3 g/L of 1:1 SDS-TW80

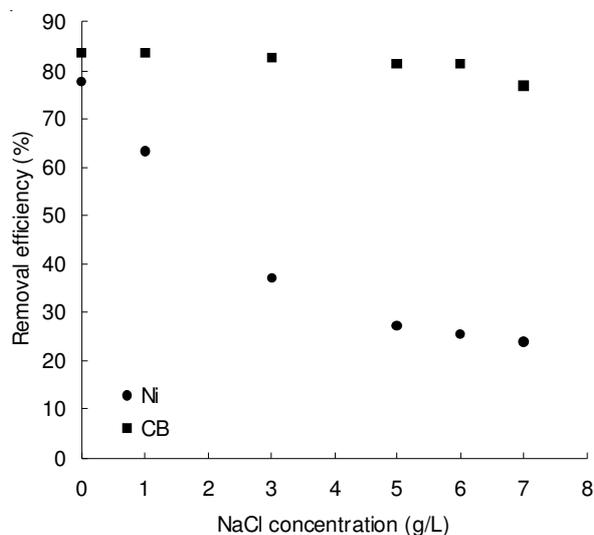


Fig. 8. Effects of NaCl concentrations on the removal efficiencies of nickel and chlorobenzene enhanced by 3 g/L of 1:1 SDS-TW80

due to the little effects of pH on solubilization extent of chlorobenzene by mixed surfactants<sup>28</sup>.

As show in Fig. 8, the removal efficiencies of nickel can be greatly reduced by the addition of NaCl. When the concentrations of NaCl change from 0 to 7 g/L, the per cent removals of nickel reduce from 77.8 % to 23.9 % while that of chlorobenzene decreases gradually from 83.7 % to 76.8 %. On the one hand, the CMCs of surfactants can be decreased with addition of electrolyte (*i.e.*, NaCl), which reduce the surfactant concentration in permeate. On the other hand, the interaction between cationic metal ions of electrolyte and charged micelles brings about decreasing adsorption of target ions onto micelles. In addition, a complex compound may be formed by the target metal ions with the anionic ions of electrolyte, which also restrains metal ions onto micelle surface<sup>23</sup>. Thus, the pH values and electrolyte concentration in feed solutions should be taken into consider if the anionic-non-ionic surfactant would be used for simultaneous removal of metal ions and organic contaminants from water.

### Conclusion

The work presents that addition of TW80 leads to reduction of nickel rejection in ultrafiltration enhanced by anionic surfactant SDS. The rejection efficiencies of nickel by surfactants are in order of SDS > 3:1 SDS-TW80 > 1:1 SDS-TW80 > 1:3 SDS-TW80. The reduction in flux by surfactants is in order of SDS ~ 1:1 SDS-TW80 > 3:1 SDS-TW80 > 1:3 SDS-TW80. In the procedure of ultrafiltration of both nickel and chlorobenzene enhanced by mixed SDS-TW80s, the rejection extents of nickel by SDS-TW80s are in order of 3:1 SDS-TW80 > 1:1 SDS-TW80 > 1:3 SDS-TW80 while those of chlorobenzene in order of 1:3 SDS-TW80 > 1:1 SDS-TW80 > 3:1 SDS-TW80. The results indicate that mixed anionic-non-ionic surfactants with an appropriate ratio of anionic to non-ionic one could be an alternative to enhance ultrafiltration of metal ions and organic compounds simultaneously. In addition, significant effects of pH values and NaCl concentration on the rejection of nickel were found in simultaneous removal process.

### ACKNOWLEDGEMENTS

This work was financially supported by the Program for Changjiang Scholars and Innovative Research Team in University (IRT0966), the Master Supervisor Project of Gansu Education Committee (0704-04) and the Qinglan Talent Project (2004) of Lanzhou Jiaotong University.

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