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Assessment of the micellar-enhanced ultrafiltration process with a tight UF membrane for the removal of aniline from water

Bahareh Tanhaei[a], Mahdi Pourafshari Chenar[a,*], Nasser Saghatoleslami[a], Mehrdad Hesampour[b], Mari Kallioinen[c], Mika Mänttäri[c]

[a]*Chemical Engineering Department, Faculty of Engineering, Ferdowsi University of Mashhad, Mashhad, Iran*
Tel. +98 511 8805020; Fax: +98 511 8816840; email: pourafshari@um.ac.ir
[b]*Kemira Oyj, R&D Center, Espoo, Finland*
[c]*Chemistry Department, Lappeenranta University of Technology, Lappeenranta 53850, Finland*

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ABSTRACT

Micellar-enhanced ultrafiltration (MEUF) is a promising method to remove low molecular weight (LMW) organic contaminants from water. In this work, a series of experiments were conducted in order to evaluate the efficiency of an MEUF process for the removal of aniline (as LMW organic contamination) using a 1 kDa molecular weight cut-off polyethersulfone membrane and sodium dodecyl sulfate (SDS) as an anionic surfactant. Furthermore, the effect of various parameters such as aniline and surfactant concentration, operating pressure, temperature, agitation velocity, the presence of a nonionic surfactant (Brij 35), and the pH of feed on the rejection of aniline and SDS, and relative permeation flux have been examined. In the presence of a nonionic surfactant, the maximum rejection of aniline (approximately 80%) was obtained. The results of this study also revealed that although the complete retention of aniline was not achievable, the MEUF process could, however, be utilized to facilitate aniline removal from aqueous phase.

Keywords: Micellar-enhanced ultrafiltration; Aniline; SDS; Rejection; Membrane

1. Introduction

Many industrial wastewaters are contaminated with organic solutes, which may be toxic for humans and the environment [1]. Aniline is one of these organic solutes which has been used extensively in agriculture, pharmaceutical products, resins, marking inks, perfumes, shoe polishes, dyes, conducting polymers, and many other chemicals of current domestic and industrial interest [2]. Aniline is known as a potentially toxic material for environment and humans. It is readily soluble in water (up to 3.6 wt.% at 20°C), therefore it can easily contaminate a large volume of water and cause serious environmental problems [3]. Aniline can also cause soil contamination by interacting with enzymes in soil or micro flora and forms carcinogenic azo-compounds and other oxidation products. Aniline is a blood toxin and long or repeated exposures may result in decreased appetite, anemia, weight loss, nervous system effects, and kidney, liver, and bone marrow damage [3]. Due to serious problems associated with aniline contamination, industrial wastewater containing significant

*Corresponding author.

levels of aniline should be purified before discharge to environment [4].

Several methods have been studied to remove aniline from wastewater, including biodegradation [5], adsorption [6], oxidation processes [7], and various membrane processes such as pervaporation [8], liquid membranes [1], nanofiltration [9], and especially reverse osmosis (RO) [4]. The efficiency of these methods varies but commonly they all use a great deal of energy and are relatively expensive.

Compared to other processes, membranes could be integrated with other process and chemicals, meaning can be added as a retrofit of existing plant. One example of chemical process integration is micellar-enhanced ultrafiltration (MEUF).

MEUF is a surfactant-membrane-based separation technique. Surfactants at higher concentration than critical micelle concentration (CMC) aggregate pollutants in aqueous phase and form micelles. These micelles are then filtrated through ultrafiltration membrane (UF) which has smaller pore size than micelles. This process combines the high efficiency of RO and the high relative flux (RF) of the UF. In the MEUF process, the solute rejection efficiency and RF depend on the characteristics of the solutes, surfactants,and membrane, as well as operating conditions [10].

MEUF is a viable alternative technique which might be economical and effective for the removal of dissolved organic contaminants, especially in the case of low molecular weight organic contamination [11] or multivalent ions from wastewater.

MEUF has been used to separate the organic pollutants, heavy metals using cationic surfactant, anionic surfactants, and mixed surfactants [10,12–15]. However, most of researches on MEUF have focused on the phenols' removal as organic matters from aqueous streams [11,16–18]. In the best of our knowledge, just a few works have been reported in the literature for the removal of aniline from wastewater using MEUF. Jadhav et al. [11] have studied the removal of phenol as an ionic compound and aniline as a nonionic compound using a counterionic surfactant (cetylpyridinium chloride (CPC)). The effect of operating parameters such as applied pressure, solutes, and surfactant bulk concentrations on the extent of organic solute separation was investigated.

In other work, ionic surfactants have been used for preconcentration of aniline derivatives [19]. Review these articles show that the removal of aniline was studied in combination with other contaminants and the effects of only few operating conditions was studied on the overall performance, e.g. the effect of temperature and pH was not studied. Therefore, the aims of this study are: first, to study MEUF performance on separation of aniline alone; second, to extend study and include the effect of more variables such as pH and temperature on MEUF performance.

This study was carried out with a tight UF membrane (PES NP010 membrane), ionic surfactant, sodium dodecyl sulfate (SDS), and nonionic surfactant (Brij 35). The effect of different operational parameters such as pressure, temperature, agitation velocity, aniline and surfactant concentrations in feed, the presence of a nonionic surfactant (Brij 35) and pH on the rejection of aniline, and SDS and relative permeation flux of membrane has been investigated. These results can be useful to achieve the practical application of this technique for the effective removal of organic matters.

2. Materials and methods

2.1. Chemicals and membrane

In this work, SDS as an anionic surfactant and polyethylene glycol lauryl ether (Brij 35) as a nonionic surfactant were utilized. The properties of the aniline are listed in Table 1. For the preparation of all solutions, deionized water with a conductivity of, roughly, 0.8 μS/cm was utilized. The characterizations of the membrane used in this work are presented in Table 2.

2.2. Determination of surface tension

The CMC of SDS was determined from the surface tension values. Pendant drop method [20] was used to measure the surface tension of the SDS aqueous solution.

Contact angle measurement (i.e. CAM) and interfacial surface tension value were measured by the KSV CAM 101 instrument (KSV Instruments Ltd., Finland).

2.3. Ultrafiltration procedure

Filtration was carried out using a dead-end Amicon stirred cell (model 8400) with a feed volume of 300 mL. A schematic diagram of the ultrafiltration set-up and principles of MEUF is shown in Fig. 1.

Table 1
Properties of aniline

Characteristic/property	Data
Molecular formula	C_6H_7N
Density	1.0217 gr/ml, Liquid
Molecular weight	93.13 gr/mol
Water solubility	35 g/L at 25˚C
Basicity(K_b)	9.3
Viscosity	3.71cP (3.71 mPa s) at 25˚C
Log(K_{ow})	0.9

Table 2
Characterizations of the used membrane

Membrane type	Manufacturer	Material	Membrane property	MWCO[a], kDa	M.O.P.[b], bar	M.O.T[c], ℃	P.W.F[d] (L m^2h)
NP010	Macrodyn® Nadir	Polyethersulfone	Hydrophilic	1	40	95	>200

[a]Molecular weight cut-off. [b]Maximum operation pressure. [c]Maximum operation temperature. [d]Pure water flux.Test conditions: 40 bar and 20˚C.

The membrane utilized in this work has an effective surface area of 40 cm^2. Before each experiment, the membrane was kept into deionized water for 2 h and then the pretreated membrane was placed in the cell and compacted at 540 kPa for approximately one hour using deionized water. The membrane permeability was determined from the pure water fluxes at various pressures. The pure water flux was measured at a pressure of 260 kPa before and after each experiment, and membrane fouling was estimated from the difference in pure water permeabilities. The membrane fouling was relatively low (below 10%) in test series.

The feed solution was prepared by weighing a definite amount of aniline and surfactant and dissolving them in the 250 mL of deionized water. Then, the solution was mixed for at least one hour to ensure that the solutes were evenly distributed in the feed solution. For the filtration, the cell was filled with the prepared feed solution. Next, the feed solution was stirred for 15 min under atmospheric pressure followed by setting the desired trans-membrane pressure with nitrogen gas. Filtration at a constant pressure of 260 kPa was sustained until 100 mL of permeate was collected (volume concentrated factor (VCF) = 1.7). To ensure the repeatability of the results, all the experiments were carried out three times and their average was used in calculations.

2.4. Measuring adsorption

Adsorption of aniline on membrane surface was measured in Amicon cell. During this, the test membrane was placed on impermeable support and the pressure was very low. The membrane was in contact with a solution containing aniline for about 8 h. The adsorption was calculated based on the difference between the initial and final concentrations of aniline in solution inside of Amicon cell.

2.5. Analysis

The concentration of aniline was measured by UV absorption at a wavelength of 280 nm with an UV-vis spectrophotometer (JASCO V-670, Japan), as was carried out by other researchers [1,11]. The SDS concentration was determined by titrating SDS solution with cationic solution (poly DADMAC, 0.001N) in a Mütek Particle Charge Detector (PCD 02, Germany).

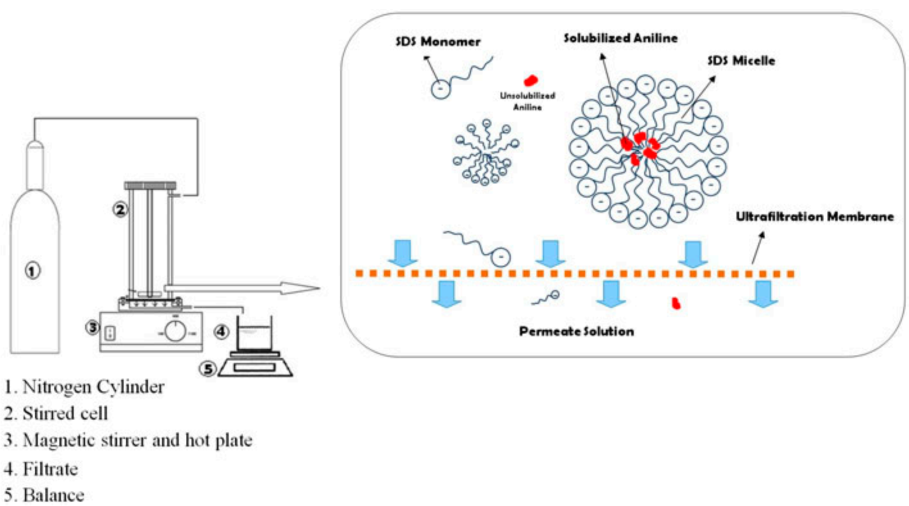

Fig. 1. Schematic diagram of the ultrafiltration unit and MEUF process of aniline.

2.6. Characterizations

In this work, the permeate flux of the UF was defined as:

$$J_i = \frac{Q_i}{A}$$

where J_i is the permeate flux (kg (m^{2}h)), Q_i is the mass rate (kg h), and A is the effective area of the membrane (m^2).

The RF here is defined as the ratio of permeate flux to pure water flux (J_W) at the same operating conditions:

$$\mathrm{RF} = \frac{J_i}{J_W}$$

VCF is defined as:

$$\mathrm{VCF} = \frac{V_{\mathrm{ini}}}{V_{\mathrm{fin}}}$$

Where V_{ini} and V_{fin} are the volumes of solution in the batch stirred cell initially and at the end of the test, respectively.

The rejection of aniline and the rejection of the surfactant ($R\%$) were defined as:

$$R\% = \left(1 - \frac{2C_p}{C_{F1} + C_{F2}}\right) \times 100$$

where C_p is the solute concentration in the permeate and C_{F1} and C_{F2} are the solute concentrations in the feed before (i.e. $t = 0$) and after the experiment, respectively.

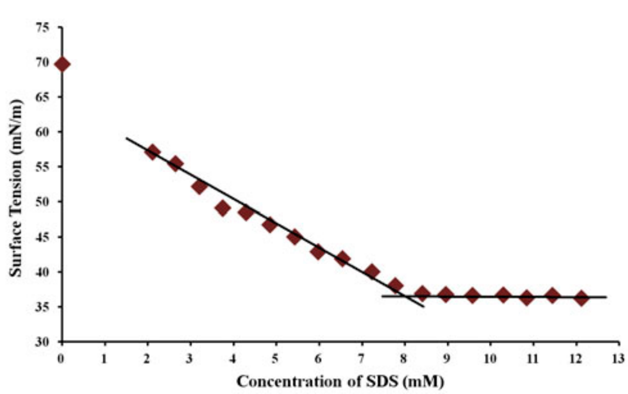

Fig. 2. Surface tension as a function of SDS concentration (mM) (pH = 7 and T = 25℃).

3. Results and discussion

3.1. Determination of CMC

The CMC is an important characteristic of a surfactant. The CMC values are estimated by plotting the surface tension as a function of the surfactant concentration (mM). Fig. 2 indicates that as the SDS concentration in pure water increases, the surface tension decreases accordingly. The SDS's Krafft temperature was 22℃ and experiments were carried out at 25℃ with a pH of 7. However, the surface tension remains constant after 8 mM of SDS where it demonstrates that the CMC value of SDS is approximately 8 mM, which is confirmed by values found in the literature [13].

3.2. Effect of surfactant concentration

To assess the optimum concentration of SDS required for the rejection of aniline, the concentration of SDS in the feed was varied from 0 to 240 mM, while keeping the aniline concentration constant at 5 ppm in the feed. The effects of the feed surfactant concentration on the rejections of aniline and SDS are shown in Fig. 3 revealing that the rejection of aniline was roughly 20% in the absence of the SDS surfactant. With the rise of the SDS concentration in feed, the micelle concentration in the solution increased accordingly. This results in increased solubilization of aniline into the SDS micelles. It is worth noting that an enhancement of the SDS surfactant amount raised the rejection of aniline until above approximately 120 mM concentration of the SDS, when the aniline rejection remained constant. This might be caused by changes in the shape and size of a micelle. At higher concen-

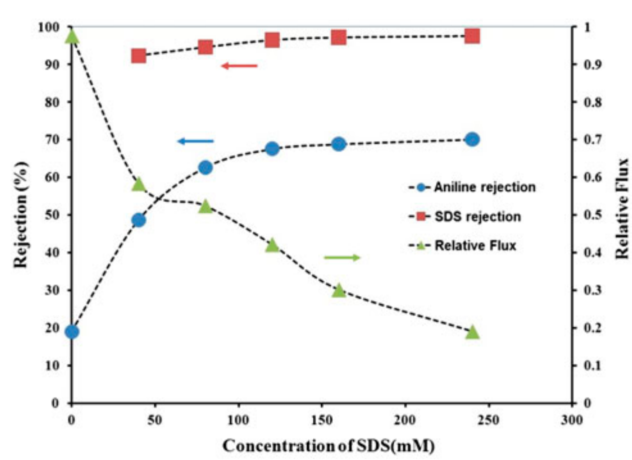

Fig. 3. Effect of the SDS concentration on the aniline rejection (%), SDS rejection (%), and RF (aniline concentration = 5 ppm, pH = 7, T = 25℃, p = 260 kPa, and VCF = 1.7).

tration, the surfactant head groups are more tightly packed and shape of micelle changes from spherical to rod-like micelles [18]. Normally, the conversion from spheres to rod-like micelles will cause a decrease in the tendency of aniline to solubilize into micelles, for a given total number of surfactant molecules [16]. Furthermore, it was observed that an addition of SDS in the feed solution stream caused the rejection of SDS to slightly increase from 93 to 97%. The pores of the UF are significantly smaller than the SDS micelles; consequently, at the SDS experimental concentration range, the SDS removal efficiency increased slightly.

The effect of the surfactant concentration on relative permeate flux is shown in Fig. 3. The figure shows that the RF decreases when SDS concentration increases from 0 to 240 mM. The presence of the micelles in the solution and their accumulation on the membrane surface would decrease RF.

3.3. Effect of aniline concentration

The effect of aniline concentration on the rejection of aniline and SDS is shown in Fig. 4. SDS concentration in feed was fixed at 120 mM and the aniline feed concentration was in the range of 5–20 ppm. All of the experiments were carried out at a pressure of 260 kPa and temperature of 25˚C. The results revealed that with increasing of aniline concentration, the aniline rejection decreased from 69% (at 5 ppm of aniline) to 58% (at 20 ppm of aniline) and were comparable with those obtained by Luo et al. in 2009. They reported that for low levels of phenol concentration, as the phenol concentration of feed increases, the phenol retention increases accordingly; however, at high level of phenol concentration, the phenol retention reduces [17]. Therefore, it could be concluded that solubilization of SDS micelle does not fluctuate with the enhancement of feed aniline concentration for the range of aniline concentrations utilized in this work. It elucidates the fact that as the concentration of micelles is almost constant, the solubilization capacity of the micelles would nearly be constant, as well (i.e. SDS micelles could dissolve a fixed amount of aniline). This means that the concentration of unsolubilized aniline increases by increasing aniline concentration and this additional aniline could pass through the membrane. The results of this study revealed that it was consistent with those obtained by other researchers [21,22]. Purkait et al. also investigated the MEUF of eosin (as an acid dye) and phenolic derivatives by Cetyl (hexadecyl) pyridinium chloride (CPC) as a cationic surfactant in two separated studies [21,22]. They demonstrated that further increase in dye and phenolic derivatives concentrations at constant CPC concentration causes the enhancement of unsolubilized dye and phenolic derivatives molecules in feed solution and the reduction in the retention of dyes and phenolic derivatives. Jadhav et al. showed that the solubilization equilibrium constants that were computed for the aniline molecules in CPC micelles did not vary over a wide range of aniline concentrations [11]. Due to higher rejection of aniline at 5 ppm, further test in this study was carried out at 5 ppm of aniline.

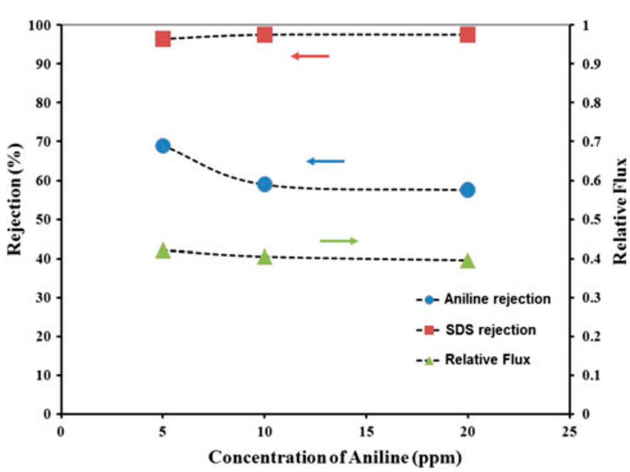

Fig. 4. Effect of the aniline concentration on the aniline rejection (%), SDS rejection (%), and RF (SDS concentration = 120 mM, pH = 7, T = 25˚C, p = 260 kPa, and VCF = 1.7).

As Fig. 4 demonstrates, the aniline concentration in the feed did not appreciably affect the SDS rejection, which perfectly agrees with the surfactant rejection rate obtained by other researchers [17,21,23]. Luo et al. demonstrated that phenol concentration has no significant effect on the surfactant separation efficiency of OTAB surfactant [17]. Purkait and Zeng also revealed that retention of CPC did not vary with the enhancement of dye and phenol concentrations, respectively [21,23].

The effect of aniline concentration on the RF is shown in Fig. 4. It is clear that the reduction of the flux was small (i.e. approximately 6%). The reduction of RF at the higher aniline concentration could be caused due to the increase of adsorption of free aniline molecules (approximately 14 ppm/m^3) on the membrane [24].

3.4. Effect of temperature

In the MEUF process, the temperature is an important criterion as the CMC of surfactant depends highly on the temperature. The CMC of the surfactant would

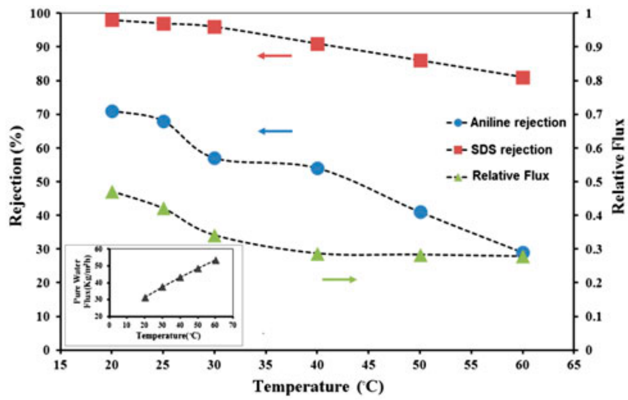

Fig. 5. Effect of feed temperature on the aniline rejection (%), SDS rejection (%), and RF (concentration of SDS = 120 mM, aniline concentration = 5 ppm, p = 260 kPa, pH = 7, and VCF = 1.7).

increase with the temperature due to the demicellarization process. Increasing of temperature can disorganize the palisade layer of the micelles [12]. So, the surfactant ions exit from the micellar bulks and the solvent molecules around the hydrophobic tails separate. This was verified as some researchers reported that the CMC values of SDS are 7.8, 8.5, and 9.4 mM at temperatures of 25, 40, and 45˚C, respectively [12].

In the present study, the effects of the feed temperature on the rejection of aniline, SDS, and RF were investigated at fixed aniline and SDS concentrations of 5 ppm and 120 mM. Fig. 5 shows the effect of the feed temperature on the rejection of aniline and SDS. It was observed that the rejection of aniline and SDS decreased considerably with the increase of temperature. In this temperature range, several researchers have shown that increasing temperature causes an increase of the CMC of SDS and also a decrease of the micelles' aggregation number [25]. Consequently, free aniline and SDS monomers would be increased in the solution. Furthermore, an increase of temperature could widen the membrane pores, and subsequently, more SDS and aniline molecules pass through the membrane.

The RF here is defined as the ratio of permeate flux to pure water flux at same temperature. As can be seen in Fig. 5, as temperature increases, RF decreases, while pure water flux increases even more than what can be assumed from the decrease of the water viscosity [26]. Increase in pure water flux could be attributed to enlargement of membrane pore size. At high temperature, a membrane has bigger pore size [27] and SDS is more in monomer form, so, small micelles or monomers of SDS can block the membrane pores. Therefore, at the temperature range of 20–40˚C, RF decreases with the temperature when surfactant solution was filtered. But above 40˚C, thermal expansion of the membrane material and lower viscosity of the solution hold decreasing of RF. At lower temperature, the rejection and flux are more stable.

3.5. Effect of pressure

The effects of the operating pressure on the rejection of aniline, SDS, and RF were investigated at fixed aniline and SDS concentrations of 5 ppm and 120 mM and temperature of 25˚C. The effects are shown in Fig. 6, which reveals that the aniline rejection decreases slightly with an increase of operating pressure, ranging from 68% at 200 kPa to 61% at 500 kPa. This could be due to the fact that at the higher operating pressure, micelles might become more compact, and therefore, the micelles can solubilize fewer aniline molecules [28]. Thus, a smaller amount of aniline would be solubilized in the micelles at a higher operating pressure. Enhancement of effective driving force would also increase the concentration polarization and, hence, raise the diffusive transport of solutes through the UF to the permeate solutions. This would also be the reason for the decline of aniline rejection with the enhancement of operating pressure.

Fig. 6 exhibits the rejection of SDS at different operating pressures. It also demonstrates, in particular, that the rejection of SDS was almost constant for the pressure range from 200 to 500 kPa. This could be caused owing to the fact that the surfactant concentration in the feed solution was much higher than the CMC; therefore, micelles had the tendency to settle on the membrane surface. Thus, a micellar aggregation

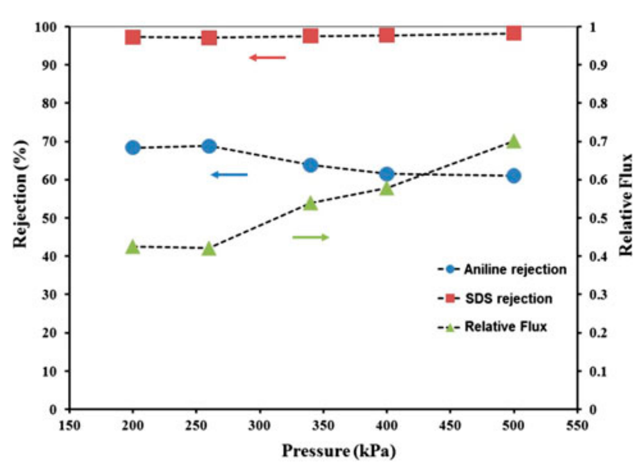

Fig. 6. Effect of the pressure on the aniline rejection (%), SDS rejection (%), and RF (concentration of SDS = 120 mM, aniline concentration = 5 ppm, T = 25˚C, pH = 7, and VCF = 1.7).

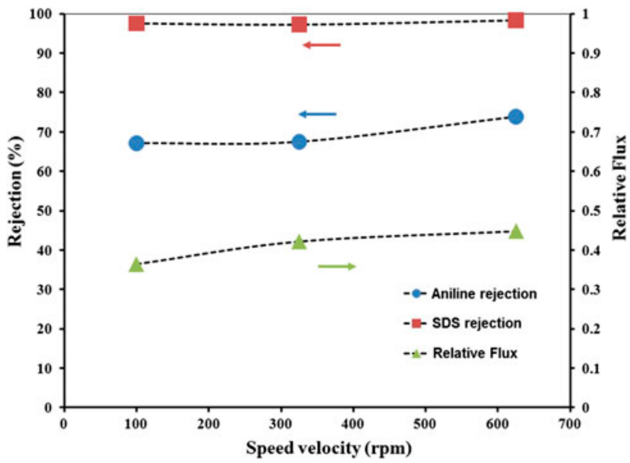

Fig. 7. Effect of the speed velocity on the aniline rejection (%), SDS rejection (%), and RF (concentration of SDS = 120 mM, aniline concentration = 5 ppm, pH = 7, T = 25˚C, p = 260 kPa, and VCF = 1.7).

layer was formed on the membrane surface [29]. This layer offers an extra resistance on the membrane surface; therefore, surfactant monomers could not penetrate through the membrane pores and hence higher pressure would be resulted as the layer was compacted [29].

Fig. 6 indicates the effect of operating pressure on RF. The operating pressure was found to have a positive effect on the RF. When increasing the pressure, the driving force also increases and, therefore, the RF is higher.

3.6. Effect of mixing speed

To assess the effect of the mixing speed, the aniline and SDS concentrations in the feed and the operating pressure and temperature were fixed at 5 ppm, 120 mM, 260 kPa, and 25˚C, respectively. In this work, the mixing speed ranged from 100 to 625 rpm. Fig. 7 shows that an increase in the mixing speed would improve only the aniline rejection.

Increasing of speed from 100 to 625 rpm increased RF by 16% and this indicates that concentration polarization decreased. But it does not have any significant effect on the rejection of SDS.

3.7. Effect of the presence of non-ionic surfactant

In this work, the effect of Brij 35 as a nonionic surfactant on the rejection of aniline and SDS and also the RF was assessed, as shown in Fig. 8. The amount of SDS that was utilized in the MEUF process was 120 mM. The results revealed that the aniline rejection efficiency increased up to 80% at a nonionic surfactant (Brij 35)/SDS molar ratio (α) of 0.3.

The mixed Brij35/SDS surfactants at various molar ratios had much lower CMC values than the pure SDS system [14,30]. The authors showed the effect of the presence of Brij 35 in the SDS solution on CMC of SDS in the previous published paper [30]. Huang et al. [14] suggested that mixed micelles could form at low surfactant concentrations. Therefore, it would cause more SDS molecules to take part in the micelle formation and, hence, result in higher aniline rejection. It was observed that nonionic surfactants decrease the electrostatic interactions between charges of ionic hydrophilic groups in the stern layer of the micelles by the inclusion into ionic surfactant micelles [18]. Therefore, in this condition, the formation of micelles in lower CMC is possible and more SDS monomer can form micelles. Therefore, this could be caused by the increase of the rejection of aniline with α.

The effect of α on the rejection of the SDS surfactant is shown in Fig. 8. It was observed that an increase in α could cause the rejection of SDS to increase accordingly. Therefore, as more surfactant micelles form, the quantity of the remaining SDS monomers in the solution would also drop. Hence, the enlarged micelles could be easily retained using UF with appropriate pore sizes.

Fig. 8 shows the dependency of the RF of MEUF on α, keeping other parameters constant. It was shown that an increase in the nonionic surfactant dosage decreased the RF accordingly (45%). This phenomenon could be explained in two ways. Firstly, the CMC of SDS would decrease with an increase in α, hence, enhancing the quantity of micelles in aqueous phase.

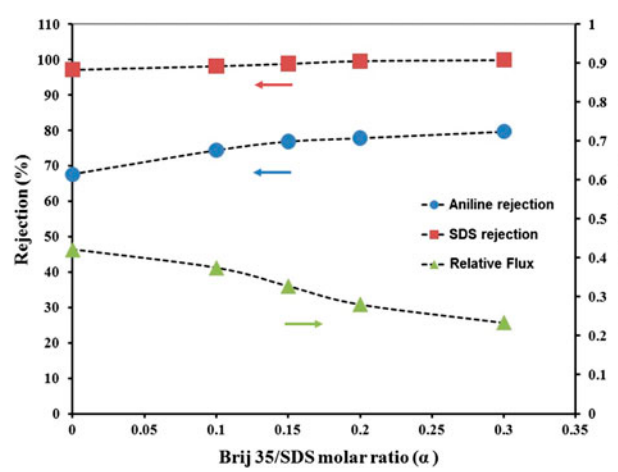

Fig. 8. Effect of the Brij 35/SDS molar ratio on the aniline rejection (%), SDS rejection (%), and RF (concentration of SDS = 120 mM, aniline concentration = 5 ppm, pH = 7, T = 25˚C, p = 260 kPa, and VCF = 1.7).

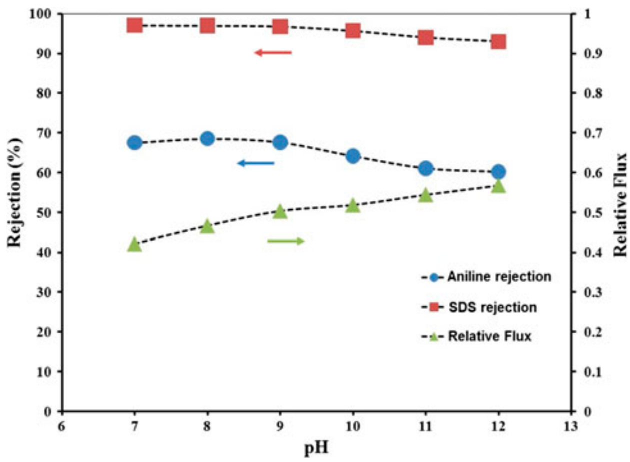

Fig. 9. Effect of the pH of feed on the rejection of aniline (%), SDS (%), RF (concentration of SDS = 120 mM, aniline concentration = 5 ppm, T = 25˚C, p = 260 kPa and VCF = 1.7).

This would also increase the retention of the solute and reduce the RF. Secondly, as the viscosity of the aqueous phase increases, the mass transfer coefficient decreases accordingly due to the addition of Brij 35, as was reported by other researchers [14].

3.8. Effect of pH

Knowing aniline is a weak base (pKa = 4.63) [31], the effect of pH on the aniline rejection was also assessed using MEUF, as shown in Fig. 9. As a result, its peak in UV-visible spectra was fully eliminated in an acidic medium. Thus, the effect of pH has been studied at pH values ranging from 7 to 12. The pH of the feed was adjusted using NaOH (0.1 N) to the desired value (pH = 7–12), and the initial surfactant concentration was kept constant throughout the experiment (120 mM). In this experiment, the rejection (R) was initially nearly constant until above pH = 9 and it only slowly decreased with the increase of the pH. At the high end of the pH range (i.e. 9–12), the drop of rejection might be due to the increase of the pore size of the membrane. The enhancement of the membrane pore size would lead to the reduction of the rejection of aniline and SDS, and rise of RF (Fig. 9).

4. Conclusion

In this work, MEUF has been employed to remove aniline from aqueous phase. The results of this study revealed that the initial surfactant concentration has a significant effect on aniline removal. Without surfactant, the aniline retention was 20%. The enhancement of the SDS amount in the feed up to 250 mM increased the rejection of aniline and SDS to 70 and 98%, respectively. Furthermore, the flux dropped due to the influence of concentration polarization. Fouling after water washing was always relatively low—below 10%. It was also observed that the aniline rejection and RF would reduce with the increase of aniline concentration in the feed, which has resulted from the constant solubilization capacity of the micelles. The temperature is also an important parameter in the MEUF process; the rise of temperature from 20 to 60˚C led to a decrease of aniline and SDS rejection. This could be caused by the expansion of the membrane pores and the enhancement of the CMC of SDS. The results showed that an increase of pressure would lead to a reduction of aniline rejection and an increase of RF. It was also concluded that as the mixing speed increased, the rejection of aniline remained rather constant. Furthermore, an increase of pH in the base media could slightly decrease the rejection of aniline; however, as a result of the enhancement of pore sizes of the membrane, the RF increases. Moreover, the addition of nonionic surfactants such as Brij 35 increased the rejection of aniline to 80%. It is approximately 10% higher than retention with the MEUF without a nonionic surfactant. This study proved that by combining ionic and nonionic surfactants, the retention of aniline can be improved in the MEUF process.

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