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Advanced bamboo industry wastewater treatment through nanofiltration membrane technology

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ABSTRACT

The present study reports the results of nanofiltration (NF) for treating COD, ammonium, color, and conductivity of bamboo industry wastewater (BIWW). The influence of operational parameters such as trans-membrane pressure (TMP), influent concentration, pH, permeate flux and operating temperature on the membrane rejection efficiencies were investigated. Molecular weight distribution (MWD) and gas chromatography–mass spectrometer (GC–MS) analyses were also performed in the study. Results demonstrated that the color obtained during rejection was higher than 99% regardless of any operating parameter. However, permeate flux, COD, ammonium, and conductivity rejections were affected by operational parameters' discrepancies. The operational changes along with the polarization concentration and accumulative mass had mainly influenced the effluent water quality. The permeate flux was recorded higher than 40 L/m^2 h, while the TMP was around 7 bar. Moreover, during the experiment, 90, 84, and 83% rejection of COD, ammonium, and conductivity, respectively, were observed. MWD data indicated that the NF module effectively removed most of the macromolecular organics and GC–MS analysis revealed the majority of organic compounds in BIWW were rejected by NF membrane.

Keywords: BIWW; Nanofiltration; Permeate flux; Rejection; MWD; GC–MS

1. Introduction

The bambooware-based industry has displayed rapid growth and development in many countries during recent years. However, the accumulation of by-products generated by the industrial process has also been a critical problem for the ambient environment. To prevent corrosion, boiling the bamboo material is an essential step in bamboo industrial process. Many pollutants, such as saccharides, organic acids, amino acids, flavonoid, tannins, and phytochromes, are generated in the stewing process resulting in higher COD concentration (10,000–30,000 mg/L). Hence, with subsequent employment of H_2O_2 and dyes, the effluent is characterized by a low pH value (3.0–3.5) and high chromaticity (2,000 times). For the characteristics mentioned above, there has not been any effective treatment method for bamboo industry wastewater (BIWW). Recently, many researchers have reported the growing concerns on BIWW problems and consequences. Several

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methods, such as supercritical water oxidation [1] and air flotation + anoxic/oxic (A/O) [2] have been presented for treatment, but the effluent cannot fulfill the discharge standards.

As newly developed techniques, membrane-based separation processes, including ultra filtration (UF), nanofiltration (NF), and reverse osmosis (RO) [3–6] have been widely used in a variety of industrial effluents in recent years. NF spans the porosity-size gap between UF and RO. The retention effect of NF is caused not only by its sieving mechanism caused by pressure difference and concentration gradient across the membrane [7,8], but also governed by the charging effect on the membrane surface [9–11]. According to the retention mechanisms, NF is able to reject organic micro-molecules with molecular weight less than 1,000 Da in solution. Additionally, NF also has a significant effect on decreasing the chromaticity, water hardness, and peculiar smell of the sewage. With its advantages of relatively lower trans-membrane pressure (TMP) and higher permeate flux, NF is widely applied for the treatment of textile, dyeing wastewater, and landfill leachate [12–16].

However, the membrane fouling, caused by polarization concentration and particle accumulation on the membrane surface along with the increasing operational time, considerably restricts the retention capability of NF [17]. In practical applications, with a certain membrane material and structure, the membrane fouling of NF is mainly caused by the TMP [18,19], concentration of pollutants [20], initial pH [21], and operational temperature [21,22]. To obtain the optimum operational conditions, these factors have to be taken into consideration.

In this research, the NF technology was applied as a post-treatment option for the A/O effluent of BIWW. The TMP, initial COD concentration, pH, and operational temperature were investigated as the influential parameters. The retention rates of COD, ammonium, chromaticity, and conductivity were determined, and coupled with molecular weight distribution (MWD) and gas chromatography–mass spectrometer (GC–MS) analyses to evaluate the effect of NF membrane for treating BIWW.

2. Material and methods

2.1. Origin of feed water

Initially, the raw BIWW was diluted to a 20,000 mg/L COD concentration and treated by the expanded granular sludge bed (EGSB). The effluent COD concentration of the anaerobic stage was reduced to about 2,000 mg/L. Subsequently, an A/O biochemical treatment was applied and the effluent (COD ≈ 1,000 mg/L) was diluted and used as the feed water in the experiment. The characteristics of A/O effluent are listed in Table 1.

Table 1
Characteristics of A/O effluent

Parameter	Mean value ± standard deviation
COD (mg/L)	985 ± 15
Color[a]	1,000 ± 150
NH_4^+–N (mg/L)	103 ± 4
Conductivity (μs/cm)	1,389 ± 26
pH	6.5 ± 0.2

[a] The color is measured by diluted multiples method.

2.2. NF unit and membrane modules

Fig. 1 presents the schematic diagram of the continuous cross flow NF unit. The feed solution was pumped to the membrane cell from the feed tank through a channel which ended with the metal support of the membrane. The rejected solution was re-cycled to the feed tank, and two rotameters were placed in the retentate and permeate lines of the cell in order to measure the flow rates. The pressure inside the cell was maintained by manually operating the valve. Permeate samples were collected from the permeate line.

The bench-scale NF module applied in our experiment was supplied by Dow-Filmtec. The surface area of the polyamide composite membrane was 7.6 m^2. At the operation conditions of 25℃ and 4.8 bar, the theoretic rejection rate and permeate flux of solution containing 2,000 ppm NaCl could be 85% and 29.05 L/m^2 h, respectively; for solution containing 2,000 $MgCl_2$, the data could be 97% and 38 L/m^2 h, respectively.

2.3. Experiments

The effects of operation time, TMP, initial pH, COD feed concentration, and temperature on the

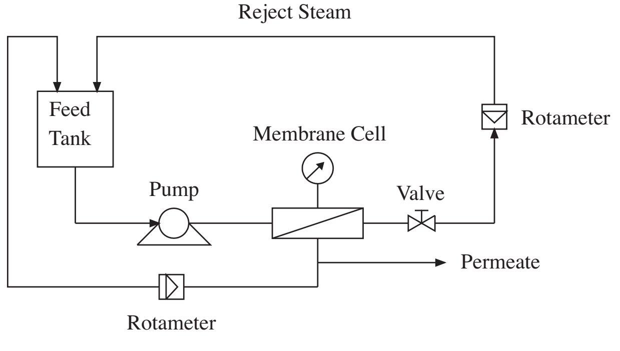

Fig. 1. Schematic of continuous cross flow NF cell.

performance of the NF process were studied in the subsequent experiment to obtain the best operating parameters of the NF unit. The COD, ammonium, color, conductivity, and permeate flux were analyzed by changing the operating parameters.

2.4. Analytical methods

The COD concentration was determined by titrating ferrous ammonium sulfate after heating the sample in a COD digestion meter (Hach, USA). Ammonium concentration was determined by the Nessler's Reagent spectrophotometry method using a UV–visible Spectrophotometer (Unico Co. Ltd., China); sample color was determined by diluted multiples method and the conductivity was determined by a conductivity meter (Keyi Science & Technology Co., Ltd., China).

The permeate flux was calculated as follows:

$$J = \frac{V}{At} \tag{1}$$

where J (L/m^2 h) was the permeate flux; V (L) was the volume; A (m^2) was the surface area of a NF membrane; and t (h) was the retention time.

Rejection of COD, ammonium, color, and conductivity were calculated as follows:

$$R = [1 - (C_p/C_f)] \times 100\% \tag{2}$$

where R was the rejection factor; C_p and C_f represented the values of a particular component in the permeate and feed water.

The MWD of samples were determined by using a gel permeation chromatograph (Waters, America) equipped with an Ultrahydrogel^{TM}500 and an Ultrahydrogel^{TM}120 column in series. The sample size was 50 μL with a flow rate of 0.8 mL/min and the column temperature was 40℃.

GC–MS was carried out to identify organic fractions in BIWW. Samples were extracted thrice with n-hexane and dehydrated with anhydrous sodium sulfate. The organic fraction was evaporated, dissolved in equal volume of n-hexane, and then analyzed by a GC–MS.

The pH values were adjusted by 0.1 M HCl and NaOH solutions with a digital pH meter (Mettler-Toledo Instruments (Shanghai) Co. Ltd., China).

3. Results and discussion

3.1. NF performance on color rejection of BIWW

The raw wastewater appeared to be brownish yellow, with a color content of 400–800 times. As a result of NF treatment, permeates of different operation parameters were almost transparent and the color content was lower than 5 times, with color reductions all higher than 99%, indicated that most of the chromogenic groups in BIWW were effectively rejected by the NF membrane. Subsequent experiments indicated that the color reduction process was not much influenced by the operating parameters.

3.2. Effect of TMP on permeate flux and rejection of permeate characteristics

Fig. 2 shows the effect of TMP on the steady-state permeate flux and rejection of COD, ammonium, and conductivity, respectively. The permeate linearly increased with the TMP from 27.63 to 59.21 L/m^2 h which was mainly attributed to the raising impetus along with the TMP. The permeate flux was com-

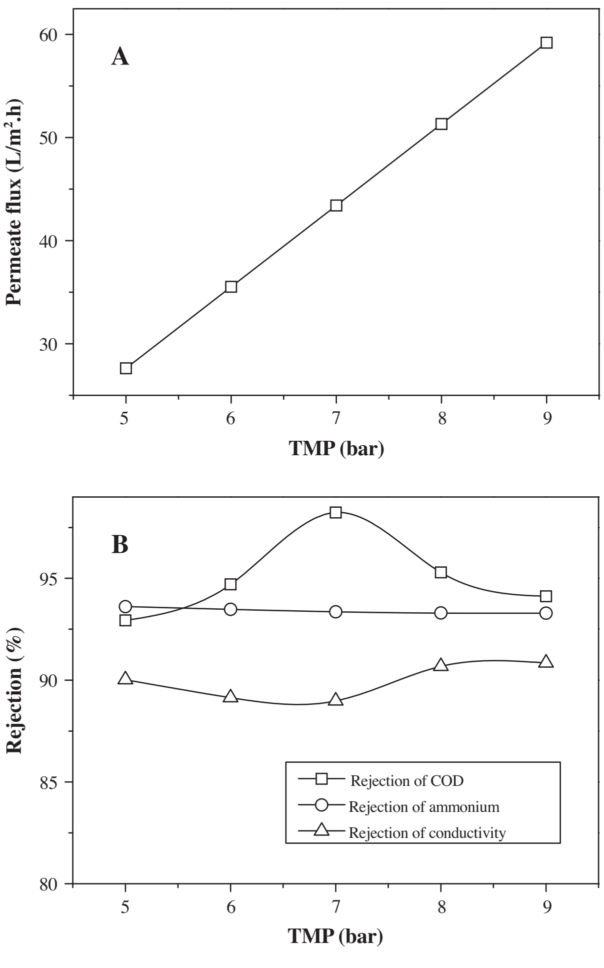

Fig. 2. Effect of TMP on: (A) permeate flux; (B) rejection rates (feed COD concentration = 680 mg/L; ammonium concentration = 44 mg/L; conductivity = 755 μs/cm; pH = 8.54; and temperature = 25℃).

monly described by using a resistance model of the following form [23]:

$$J = \frac{\Delta P - \sigma \Delta \pi}{\mu R_{\text{tot}}} \tag{3}$$

where J was the flux, ΔP was the TMP, σ was the reflection coefficient, and $\Delta \pi$ was the osmotic pressure difference. Under ideal conditions (while feed is deionized water), the value of $\sigma \Delta \pi$ is 0, indicating the permeate flux linearly changing with the TMP, which was consistent with the present experiment.

As shown in Fig. 2, the conductivity rejection rate was slightly decreased from 93.61 to 93.29% with the increasing TMP. Similar trends were observed by Hakimhashemi et al. [24], which was considered to be not statistically significant. However, the unconspicuous change of reduction may be attributed to the two effects canceling each other out. Firstly, as the increasing impetus along with the TMP, the ions in solution could easily pass through the membrane surface and that contributed to a decrease in the conductivity reduction. Secondly, an increased TMP led to higher surface tightness of membrane, which enhanced the reduction potential reversely. The permeate flux was affected by both the factors mentioned above, and as the former one had a greater influence, a decreasing tendency was also observed.

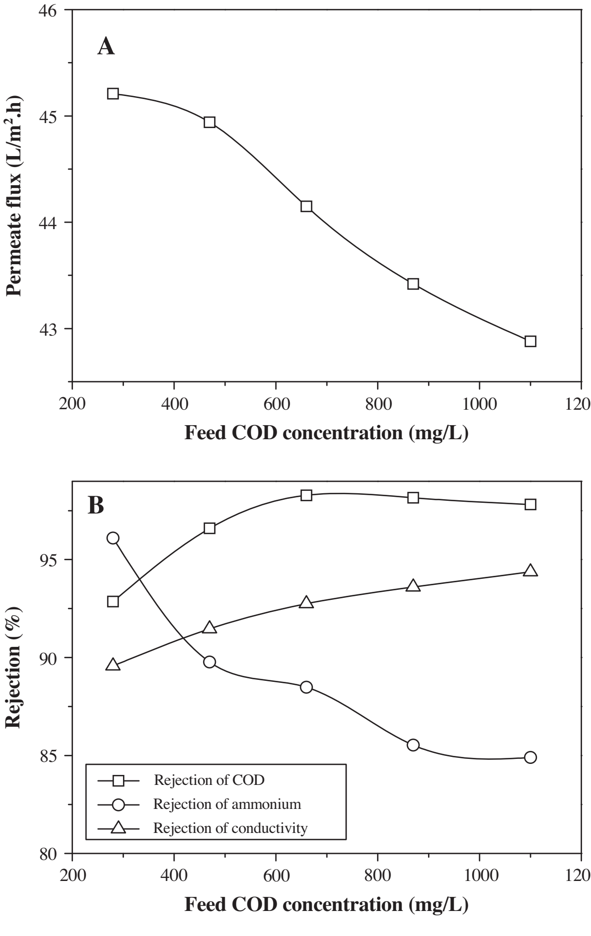

Fig. 3. Effect of feed COD concentration on: (A) permeate flux; (B) rejection rates (TMP = 7 bar; pH = 8.46; and temperature = 25℃).

The rejection of COD initially increased but ultimately decreased with a rise in TMP. The highest rejection obtained at 7 bar was higher than 98%. When the TMP was lower than 7 bar, the macromolecular organic pollutants (saccharides, amino acid, and cellulose) accumulated at the membrane surface, which resulted in a gradual increase in the rejection of COD during the filtration time. This phenomenon was given the title of a "secondary membrane" in a previously reported study [25]. However, as the TMP continually increased, the effect of concentration polarization aggravated, making the permeate diffuse reversely to the feed side and finally caused a lower rejection.

The rejection of ammonium displayed a pattern opposite to that of COD removal. It decreased initially as TMP raised and later increased when the TMP was higher than 7 bar. That could be explained by assuming a smaller molecule size for ammonium than organic pollutants contained in COD. While the TMP was lower than 7 bar, as it increased, the higher impetus facilitated ammonium passage through the membrane, leading to a lower rejection. Once the pressure was higher than 7 bar, the ammonium accumulation at the surface increased, resulting in smaller space for ammonium to pass. Additionally, as the ammonium concentration of 44 mg/L was lower than COD (680 mg/L), polarization concentration could hardly affect the ammonium removal. As a result, the rejection increased when the TMP was higher than 7 bar.

3.3. Effect of feed COD concentration on permeate flux and rejection of permeate characteristics

Fig. 3 shows the influences of feed COD concentration on the state-steady permeate fluxes and rejections. The permeate flux slightly decreased with COD concentration, which was similar to the results obtained by another study [26]. It was mainly because the osmotic pressure difference was raised with an increased feed concentration according to the resistance model mentioned in Section 3.1, the permeate flux decreased as expected. Additionally, the effects of membrane fouling and polarization concentration should not be ignored. Fig. 4 shows the effect of TMP

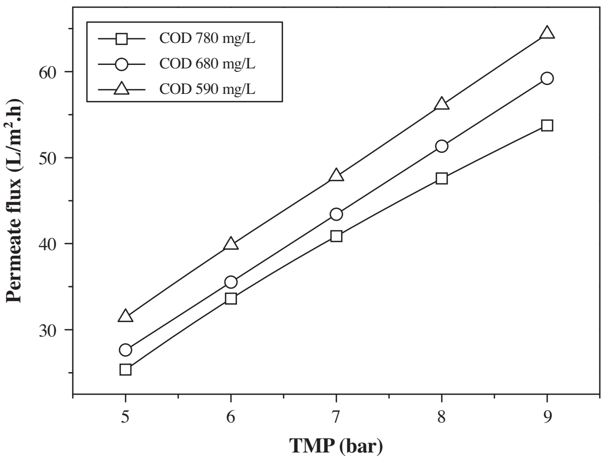

Fig. 4. Effect of the TMP on the permeate flux at different COD feed concentrations.

on the permeate flux at different concentrations of COD. When the COD concentrations were 590 and 680 mg/L, the permeate flux linearly increased with the TMP, indicating a limited influence of polarization concentration and membrane fouling. As the concentration increased to 780 mg/L, the permeate flux did not linearly change with the TMP. When the TMP was higher than 6 bar, the increasing tendency of permeate flux slowed down, indicating that the concentration polarization and membrane fouling had a negative effect on the permeability of the membrane.

Fig. 3 also shows that the rejection of the conductivity increased from 89.57 to 94.38% along the feed concentration. The enhancement of rejection may be caused by the accumulation of organic molecules on the membrane surface. Additionally, Ujang and Anderson [27] reported that with the higher feed concentration, the charge density on membrane surface also increased, which led to a higher rejection rate for conductivity.

As shown in Fig. 3, the rejection of COD first increased and then decreased with the increasing feed concentration. The highest rejection (98%) was obtained for the concentration of 660 mg/L. At the lower concentration (<660 mg/L), the main influential factor to the rejection was accumulation of molecules on the membrane surface, which produced a higher rejection as mentioned in Section 3.2. Moreover, when the concentration was higher than 660 mg/L, the effect of concentration polarization became significant, which was the main reason for the drop of COD rejection. Compared with COD, the ammonium molecule had a lower size to pass through the membrane, indicating the effect of membrane fouling was limited to its rejection. However, when the feed concentration was raised, the ammonium rejection dropped under the influence of concentration polarization.

3.4. Effect of operating temperature rejection of permeate characteristics

As clearly indicated in Fig. 5, COD, ammonium, and conductivity decreased when the temperature rose up, which showed an opposite tendency with the permeate flux. Similar behaviors were also observed by Figoli et al. [28] and Brandhunder and Amy [29] when they treated arsenic-containing wastewater. This was mainly attributed to an increase in the diffusivity of all the contaminants with temperature, which consequently increased the diffusive transport across the membrane [27]. Additionally, with an increase in temperature, the membrane pore size enlarged, which also resulted in a higher membrane flux and retention rate drop.

3.5. Effect of pH on permeate flux and rejection of permeate characteristics

In Fig. 6, the effects of pH on permeate flux and rejection is depicted. The permeate flux first decreased with the increasing pH value and reached the lowest value at pH 7.24. While increasing the pH value (higher than 7.24), the permeate flux began to increase. However, an opposite result was observed by Qin et al. [30] in the treatment of combined rinse water, and the critical point at which permeate pH would be equal to feed pH was about 6. The main reason for the phenomenon was explained as the

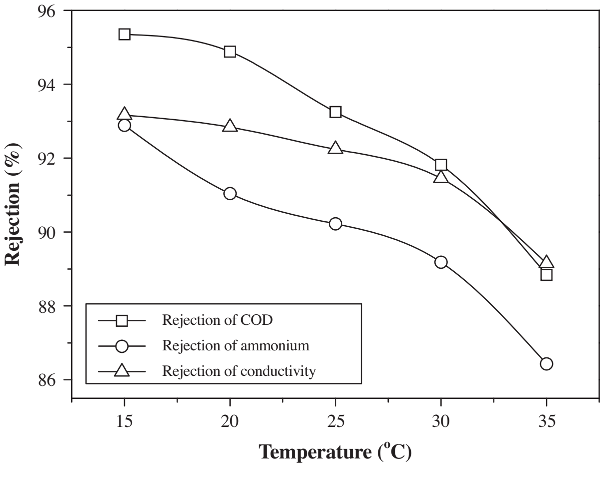

Fig. 5. Effect of feed temperature on rejection rates (feed COD concentration = 430 mg/L; ammonium concentration = 28 mg/L; conductivity = 515 μs/cm; TMP = 7 bar; andpH = 8.40).

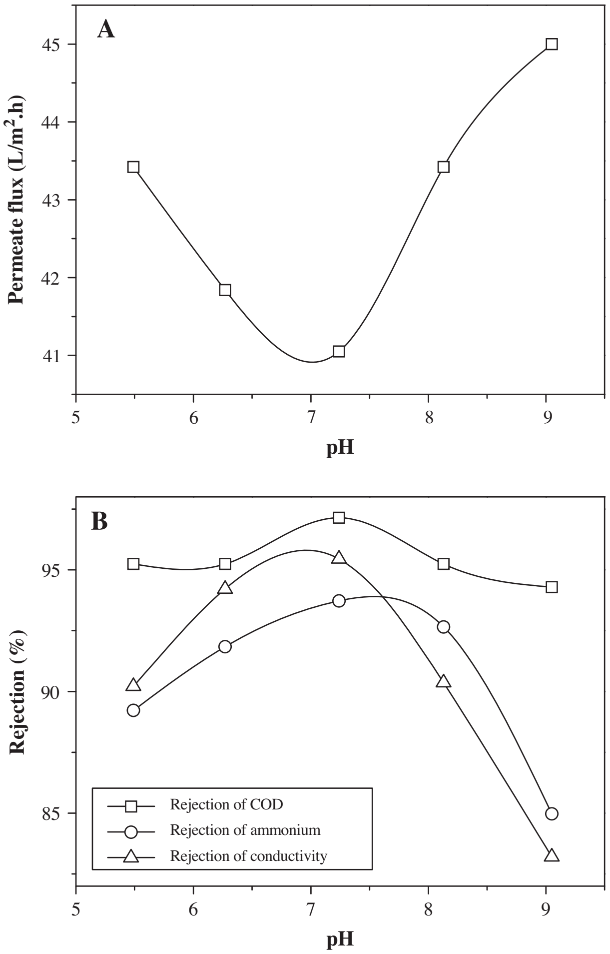

Fig. 6. Effect of pH on: (A) rejection rates; (B) permeate flux (feed COD concentration = 420 mg/L; ammonium concentration = 30 mg/L; conductivity = 508 μs/cm; TMP = 7 bar; and temperature = 25℃).

interactions of variable conjugate acid–base pairs exiting at the changing pH stages. Moreover, it was reported that the electric surface charge on NF membrane stayed zero at a critical pH value, meaning it reached the isoelectric point. The NF membrane surface was negatively charged at pH above the isoelectric point and positively charged at pH below the isoelectric point [31]. As assumed from the feed and effluent pH values in Table 2, the pH at isoelectric point in the present experiment was lower than 5. However, while the feed pH was higher than 7, the permeating pH was even higher. The reason may be that as the pH value becomes higher, the amount of OH^- in the feed increases, while the H^+ is attracted by the negative charge on the surface. So, more anions are needed to penetrate the membrane to keep electric neutrality. Thus, OH^- prior to passing through the membrane pore, due to its higher diffusion coefficient than other anions, causes a higher pH value in the permeating solution.

Table 2
Comparison of feed and permeating pH (mean value ± standard deviation)

Feed pH	Effluent pH
5.49 ± 0.05	4.55 ± 0.05
6.27 ± 0.09	5.96 ± 0.10
7.24 ± 0.06	7.32 ± 0.07
8.13 ± 0.17	8.50 ± 0.12
9.05 ± 0.12	10.49 ± 0.15

In our experiment, being with a neutral feed pH (7.24), functional groups on the membrane surface were at the non-ionization state, which caused the accumulation of molecules on the membrane surface. Consequently, the permeate flux decreased causing a higher rejection rate. Regardless of acidity or basicity of feed water, most of the functional groups were at the ionization state, strengthening the electrostatic interaction and thus, improved the enhancement of permeate flux and decline of rejection.

3.6. Analysis of MWD

Fig. 7 shows the MWD analyzed samples of feed water (COD = 420 mg/L) and permeating water (COD = 32 mg/L). Two peaks, marked 3,060 Da and 844 Da, were observed in succession at 21.010 and 22.961 min, respectively, indicating that there were still reasonable quantities of organics with high molecular weight remaining after the EGSB-A/O treatment (Fig. 7). However, after NF technology, there was only one peak that appeared at 21.505 min with a molecular weight of 2,208 Da. Compared with the feed water, the peak area reduced dramatically from 748,475 to 58,080 (data not shown) after NF treatment. Moreover, the different retention times of peaks in the two samples indicates a few by-products were formed in the permeating water, which was further confirmed in Section 3.7.

3.7. GC–MS analysis of BIWW

Table 3 enlists the GC–MS analysis data of feed water and permeating water. The compounds were grouped according to their principal functional groups. The percentages of reduction were calculated on the basis of their peak areas, according to the following equation:

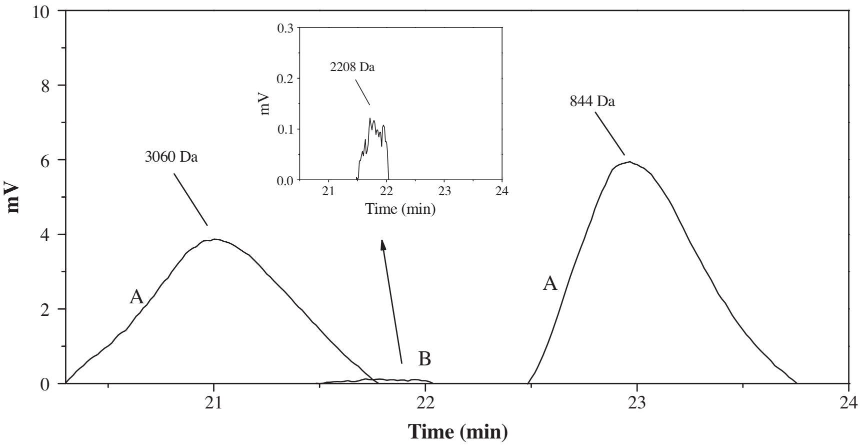

Fig. 7. MWD analysis of: (A) feed water; and (B) permeating water.

Table 3
GC–MS analysis of feed and permeating water

Compounds	Feed water	Permeating water	Reduction (%)
Alkane derivatives			
Pentadecane	942,779	–	100
Hexadecane	1,853,635	–	100
Undecylcyclopentane	2,084,503	–	100
Heptadecane	1,820,438	–	100
Octacosane	1,142,433	–	100
Octadecane	1,775,042	–	100
Hexadecane,2,6,10,14-tetramethyl-	911,769	–	100
Benzene derivatives			
1,2-xylene	2,231,359	–	100
Phenylethylene	1,025,679	–	100
Phenol,3,5-bis(1,1-dimethylethyl)-	3,102,593	–	100
2,6-Di-tert-butyl-4-methylphenol	2,132,846	–	100
2-Amino-5-methylbenzoic acid	–	1,155,094	B[a]
Ester derivatives			
2-(2-Butoxyethoxy)ethyl acetate	1,635,421	–	100
Dimethyl phthalate	2,832,105	–	100
Dibutyl phthalate	2,129,374	555,558	74
Alcohol derivatives			
1-Eicosanol	1,450,034	–	100
Linalool	–	224,536	B[a]
Ethanol,2-(dodecyloxy)-	–	310,362	B[a]
Aldehydes Derivatives			
Vanillin	–	454,900	B[a]
Decanal	–	242,955	B[a]

[a] B stands for by-product.

$$\text{Reduction} = \frac{\text{Feed water (peak area) - Permeating water (peak area)}}{\text{Feed water (peak area)}} \times 100\%$$

As observed, the contaminants in BIWW were mainly alkanes, benzene, and ester derivatives. Additionally, organics with bad smell and deep color like benzothiazole, 5-chlorovaleric acid, and diallyl sulfide, which contained elements of sulfur and chlorine, were also detected in the feed water.

After NF treatment, most of the compounds in feed water were removed completely, and the amount of the compound types had dramatically decreased. Benzene became the major contaminant in the NF effluent, in which the 2-amino-5-methylbenzoic acid content was about 22.07%. The main colored molecules and harmful organics in the feed water were almost rejected, with just a small amount of aldehyde and alcohol derivatives left in the effluent as the by-products.

4. Conclusion

The rejection of color was observed to be always higher than 99%, without significant influence of operating parameters. TMP was the main influential factor of the permeate flux, the change of viscosity; polarization concentration and accumulative mass caused by different operating conditions also affected permeate flux to a certain extent. The rejection of COD, ammonium, and conductivity reached at the maximum values of 98, 95, and 94%, respectively. It was also affected by polarization concentration and accumulative mass on membrane surface. Additionally, the molecule size created a great difference between rejection of COD and ammonium. Moreover, the rejection rate of COD, ammonium, and conductivity obtained could still be kept higher than 90, 84, and 83%, respectively.

The MWD results indicated that the NF module effectively removed most of the macromolecular organics, and the GC–MS data showed that the main colored molecules and hazardous organics, such as benzenes, alkanes, and esters were almost rejected and a small amount of aldehyde and alcohol derivatives left in the effluent as the by-products.

On the basis of the experimental results, NF can be considered a reasonably advanced treatment to remove contaminants in BIWW. Additionally, the effect of operating parameters cannot be ignored in order to produce permeating water of high quality.

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