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Implications of membrane fouling toward the removal of the pharmaceutical sulfamethoxazole by nanofiltration processes*

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Abstract: The influence of membrane fouling on the retention of the trace organic contaminant sulfamethoxazole by a nanofiltration (NF) process was investigated. Organic fouling caused a severe flux decline possibly due to pore blocking and adsorption directly after the commencement of the fouling layer development. Such membrane-foulant interactions were absent for colloidal fouling, which resulted in a more gradual flux decline. Membrane charge played a significant role in the separation process of inorganic salts, where the retention was the highest in a caustic environment (high pH) due to more swollen membrane material caused by the higher negative charge on the membrane. Organic fouling and a combination of colloidal and organic fouling led to a significant increase in the membrane negative charge. The influence of membrane fouling on solute retention was dependent on the fouling behaviour and the physicochemical properties of the model foulants, where the model foulants probably contributed to an increase in the retention of charged solutes due to enhanced electrostatic interactions. Organic fouling caused an increase in the retention of inorganic salts and sulfamethoxazole due to pore blocking. In contrast, colloidal fouling caused a decrease in the retention of inorganic salts due to cake-enhanced concentration polarisation. However, the presence of a colloidal fouling layer did not reduce the retention of sulfamethoxazole. A mixture of colloidal and organic matter improved the retention of inorganic salts. A similar conclusion can be inferred for sulfamethoxazole at pH 4 when the compound exists in a neutral form.

Key words: Nanofiltration (NF), Organic fouling, Colloidal fouling, Combined fouling, Trace organics, Water reuse
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1 Introduction

Recent advances in material and system design have made membrane filtration technology an increasingly cost-effective option for seawater desalination and wastewater reuse. In general, reverse osmosis (RO) and nanofiltration (NF) membrane technology requires lower energy consumption for the desalination of seawater than alternative thermal processes. Another important application of membrane filtration technology is in the area of wastewater reuse (Schäfer *et al.*, 2005). Water reuse (or water recycling) is the purification of reclaimed wastewater with the aim of providing further water supplies for beneficial use. Water reuse has been acknowledged “as a major solution to resolve the issue of water scarcity” (Schäfer *et al.*, 2005). A major advantage of wastewater reclamation in comparison with seawater desalination using membranes is the low osmotic pressure of reclaimed water in comparison with that of seawater. As a result, NF and low pressure RO membranes can be a cost-effective solution for the purification of reclaimed wastewater.

The shift toward water reuse has been rising globally in recent years. This is driven mainly by the increasing demand for both potable water as well as water for other purposes such as irrigation and industrial consumption. The process of producing potable water from reclaimed wastewater must be very

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reliable due to a wide range of pollutants present in wastewater. In addition, it is necessary to minimise the steps required to eliminate all possible and unknown pollutants. Compared with other water treatment technologies, NF/RO membrane filtration can play a key role in the removal of trace organic contaminants for water reuse purposes (Bellona *et al.*, 2004; Nghiem *et al.*, 2004; López-Muñoz *et al.*, 2009; Wu *et al.*, 2010a; 2010b).

During the operation of a low-pressure membrane filtration system, fouling can occur due to the deposition of various organic, inorganic and colloidal matter on the membrane surface. These constituents are ubiquitous in all types of wastewater. Bacteria, which can receive nutrients from dissolved organic matter in reclaimed wastewater, can also colonise on the membranes surface to cause biofouling (Schäfer *et al.*, 2005). As membrane fouling is inevitable during the filtration of reclaimed wastewater, it has been the subject of many investigations over the last few decades. Membrane fouling can lead to a decline in permeate flux, hence, in increasing cost to operate the water reuse system. Recent investigations have also revealed that fouling can also alter the separation performance of the membrane filtration process, resulting in either the improvement or deterioration of the permeate water quality. Given the significant interest in trace organic removal in water reuse application, several research groups have recently investigated the impact of fouling on the removal of these trace organic contaminants by NF membranes (Plakas *et al.*, 2006; Xu *et al.*, 2006; Nghiem and Hawkes, 2007). These studies highlight the complexity of the influence of membrane fouling on trace organic retention by NF membranes and the underlying mechanisms (Plakas *et al.*, 2006; Xu *et al.*, 2006; Nghiem and Hawkes, 2007; Verliefde *et al.*, 2009; Nghiem *et al.*, 2010). Xu *et al.* (2006) observed that retention behaviours of ionic, hydrophilic non-ionic and hydrophobic organics by fouled NF membranes were quite different from one another. Ng and Elimelech (2004) reported that colloidal fouling of RO membranes caused a considerable decrease in the retention of the natural hormones estrone and estradiol. It is noteworthy that most if not all studies reported in the literature to date used a single foulant or secondary treated effluent without a detailed characterisation of the fouling constituents to simulate membrane fouling. The effect of membrane fouling caused simultaneously by more than one type of foulant on the retention of trace organics has rarely been examined before. In fact, systematic investigations of membrane fouling caused simultaneously by both organic matter and colloidal particles such as the one reported by Li *et al.* (2007), are still very rare in the open literature.

This paper aims to study the influence of a combination of organic and colloidal fouling on the separation of sulfamethoxazole, which was selected as a model trace organic contaminant by an NF process. The experiments were conducted with the commercially available membrane NF270. Organic and colloidal fouling, as well as a combination of both, was simulated under controlled experimental conditions. The effects of membrane fouling on the retention of sulfamethoxazole were examined and analysed in relation to the membrane and foulant characteristics. Based on these results, the effects of combined membrane fouling on the retention of sulfamethoxazole were delineated.

2 Materials and methods

2.1 Nanofiltration membrane

An NF membrane, namely NF270, was used. The membrane was supplied by Dow FilmTec (Minneapolis, MN) as flat sheet sample and was stored dry. The NF270 membrane is a thin-film composite piperazine-based polyamide membrane with a microporous polysulfone supporting layer. The properties of this membrane are shown in Table 1.

2.2 Zeta potential analysis

The surface charge was measured with a SurPASS electrokinetic analyser (Anton Paar GmbH,

Table 1 Properties of the NF270 membrane

Pure water flux (L/(m^2·h·kPa))[a]	Average pore diameter (nm)[b]	MW cut-off (g/mol)[c]	NaCl retention (%)[d]
0.17	0.84	155	20–40

[a] Artug and Hapke, 2006; [b] Nghiem *et al.*, 2004; [c] Boussu *et al.*, 2006; [d] according to the manufacturer

Graz, Austria) equipped with a clamping cell. Potassium chloride (KCl) was used as the background electrolyte at a concentration of 1 mmol/L and the operation pressure for the measurement was set to 30 kPa. The streaming potential was calculated using the Fairbrother-Mastin approach. The pH adjustment was made with hydrochloric acid (HCl) and potassium hydroxide (KOH) by means of automatic titration. Prior to each measurement the background electrolyte solution was used to rinse the system for 100 s at 10 kPa. All measurements were conducted at room temperature ((25±1) °C).

Prior to the zeta potential measurement, the fouled membranes were dried in air. The dry membranes were then soaked in Milli-Q water for 24 h prior to the measurement. This resulted in a stable surface condition, which was able to resist the wall shear stress during measurement. Because zeta potential measurement of the fouled membrane at high pH would result in the dissolution of the foulants from the membrane surface, the measurement was conducted at pH up to 9. This technique is not suitable for colloidally fouled membranes because the colloidal fouling layer can be removed by the wall shear stress during the measurement.

2.3 Chemicals and reagents

Humic acid and the colloidal silica Ludox HS-30 were selected as the model foulants to simulate the behaviours of organic and colloidal fouling, respectively. The colloidal silica was supplied at 30% suspension in water and was stored at 4 °C.

Analytical grade sulfamethoxazole was selected as a trace contaminant. Sulfamethoxazole is a widely prescribed antibiotic and is frequently detected in raw sewage, secondary treated effluent and wastewater impacted water bodies. The properties of sulfamethoxazole are presented in Table 2.

A stock solution of 1 g/L of sulfamethoxazole was prepared by dissolving the compound in pure methanol. The stock solution was stored at −4 °C and was used within one month. A 10 L background electrolyte, containing sodium chloride (20 mmol/L), calcium chloride (1 mmol/L), and sodium bicarbonate (1 mmol/L) was used for the filtration experiments to simulate a natural environment. Analytical grade sodium hydroxide (NaOH) and hydrochloric acid (HCl) were used for feed water pH adjustment. All chemicals used in this study were supplied by Sigma-Aldrich (Castle Hill, Australia). All aqueous solutions were prepared with laboratory grade Milli-Q water.

Table 2 Properties of sulfamethoxazole

Molecular formula	Solubility[a,b] (mol/L)	MW[a] (Da)	Dissociation constant[a]	$\log K_{\text{ow}}$ at 25 °C
$C_{10}H_{11}N_3O_3S$	0.062	253.28	1.39 & 5.81	0.659

[a] SciFinder Scholar; [b] at pH 8 and 25 °C

2.4 Membrane filtration setup and experimental protocol

A laboratory-scale cross-flow NF system was used. The NF system consisted of a stainless steel plate-and-frame membrane cell with an effective surface area of 40 cm^2 (4 cm×10 cm), a stainless steel feed reservoir (10 L), and a high pressure pump (Hydra-cell, Wanner Engineering Inc., Minneapolis, MN). The concentrate flow rate was monitored by a rotameter. The permeate flow was measured using a digital flowmeter (Model 5025000, GJC Instruments Ltd., UK) and the data was automatically recorded by a personal computer. Feed pressure and cross-flow velocity were controlled by means of a bypass valve and a back pressure regulator. The temperature of the feed water was controlled with a PID control refrigerator (Neslab RTE7, Thermo Fisher Scientific, Canada).

Membrane fouling and subsequent retention testing experimental protocol was conducted in three steps: compacting, fouling development and retention measurement. First, the membrane was compacted using Milli-Q water at 1800 kPa for approximately 2 h until a stable baseline flux was obtained. The electrolyte was introduced to the feed reservoir and the permeate flux was adjusted to 117 L/(m^2·h) prior to the addition of the model foulants. The cross-flow velocity was set to be 30.4 m/s. The fouling layer was then allowed to develop for 18 h and the feed solution was kept at pH 8.

After the development of the fouling layer, the permeate flux was adjusted to 38.6 L/(m^2·h). The selected trace contaminant was spiked into the feed reservoir to make up a concentration of 750 μg/L. The solution pH was raised to approximately pH 10 by addition of a proper volume of 1 mol/L NaOH and then incrementally dropped to pH 4 using stepwise additions of 1 mol/L HCl. Prior to sample collection at each pH value, the system was equilibrated for 1 h. A small volume of feed and permeate samples was taken for analysis at specified time intervals. The temperature of the experimental solution was kept constant at (20±0.1) °C. Both permeate and concentrate were recirculated back to the feed reservoir throughout the entire experiment. To examine the retention of the trace organic contaminants by the clean membrane, a similar protocol excluding the fouling development step was adapted. Analysis of trace organic concentrations in feed and permeate samples were conducted immediately using a high-performance liquid chromatography (HPLC) system (Shimadzu, Kyoto, Japan), equipped with a Discovery C18 column (with diameter, length and pore size of 4.6 mm, 150 mm and 5 mm, respectively). The HPLC system consisted of an auto sampler (SIL-ADvp) a high-pressure pump (LC-10ATvp), a degasser (DGU-14A), a UV-Vis detector (SPD- 0Avp) and a controller (SCL-10A), and a personal computer. Acetonitrile and water were used as the mobile phase. The eluent flow rate was 0.7 ml/min and detection was made with a wavelength of 280 nm. Observed retention is defined as $R=100\times(1-C_P/C_F)$, where C_P and C_F are permeate and feed concentrations, respectively.

3 Results and discussion

3.1 Simultaneous organic and colloidal fouling

Fig. 1 shows the evolution of the membrane permeate flux over time during the fouling layer development phase. Colloidal fouling did not affect the flux as much as organic fouling. A combination of both foulants led to a more severe flux decline as observed with humic acid or silica colloids alone. The role of humic acid fouling on the flux decline (Nghiem and Coleman, 2008; Nghiem *et al.*, 2008a) and from colloidal fouling has been discussed in a previous study (Nghiem *et al.*, 2008b).

Membrane fouling involving the organic foulant humic acid appeared to follow two distinctive stages (Fig. 1). In the initial stage, significant flux decline was observed during approximately the first 400 min of the filtration process. The flux decline became gradual and subsequently reached an almost stable state at the end of the experiment. It has been concluded that the first stage was initiated from humic acid adsorption and pore blocking due to small flock particles (Nghiem *et al.*, 2008a), which could be a major cause of severe membrane fouling (Nghiem *et al.*, 2006). The second stage of fouling occurred probably due to a continuous build-up of the fouling layer on the membrane surface. It is unlikely that silica colloids can absorb into the membrane and cause pore blocking due to its hydrophilic nature and larger particle size compared to the nominal membrane pore diameter of the NF270 membrane. The hydrodynamic diameter of the Ludox HS-30 silica was estimated to be approximately 9 nm (Boussu *et al.*, 2007). In contrast, the nominal pore diameter of the NF270 was reported to be 0.84 nm (Nghiem *et al.*, 2004). Therefore, in the absence of pore blocking and adsorption, the permeate flux decline due to silica colloidal fouling was gradual and quite small (Fig. 1).

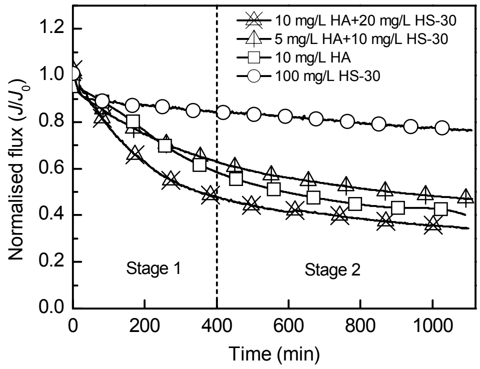

Fig. 1 Normalised permeate flux of the NF270 membrane as a function of time. The background electrolyte contained 20 mmol/L NaCl, 1 mmol/L $NaHCO_3$ and 1 mmol/L $CaCl_2$. Cross-flow velocity, permeate flow and temperature was 30.4 cm/s, 38.6 L/(m^2·h) and (20.0±0.1) °C, respectively

A mixture of colloidal and organic matter led to a more severe permeate flux decline in comparison to that caused individually by either of the foulants. A higher concentration of both organic and colloidal matter led to a much more severe permeate flux decline. This is particularly prevalent in the initial stage of fouling where a sharp flux decline could be observed. Interestingly, the build-up of the fouling layer in the second stage was almost similar for both mixtures and did not seem to be dependent on the concentration of the foulant mixtures (Fig. 1).

3.2 Effects of organic/colloidal fouling on solute separation

The influence of membrane fouling on the retention of inorganic salts (measured by conductivity) was investigated by comparing the retention values under clean and fouled conditions. With an average pore diameter of 0.84 nm, the NF270 can be classified as a loose NF membrane. Therefore, the transport of inorganic salts can occur via both convection and diffusion. The membrane surface charge can play an important role in the transport via convection. Fig. 2a shows the zeta potential of the NF270 membrane as a function of pH. The membrane surface charge was negative throughout the pH range. The influence of the membrane surface charge on the retention of inorganic salts by the virgin membrane can be seen in Fig. 2b. Conductivity retention was the lowest under acidic conditions at pH 4.5 (11%) and the highest under caustic conditions at pH 10 (28%). Elimelech *et al.* (1997) attributed this phenomenon to a swelling of the membrane with increasing background pH, caused by the interactions between the increasingly negatively charged functional groups of the membrane polymeric matrix. This mechanism reduces the membrane pore size and subsequently results in an increase in retention at high pH when the membrane surface is most negatively charged (Elimelech *et al.*, 1997). In fact, according to Freger (2004), membrane swelling is especially prominent for semi-aromatic membranes.

It can be further seen in Fig. 2b that membrane fouling caused a considerable variation in the retention of inorganic salts. Note that the effect of induced charge (membrane swelling) with increasing pH could still be observed on fouled membranes and that salt retention by the prefouled membranes increased with increasing pH. As discussed above, humic acid fouling could cause pore restriction and thereby limit solutes to cross the membrane via pores. In addition, Xu *et al.* (2006) observed an increased negative membrane charge after the membrane was fouled by organic matter and referred this to the accumulation of the negative charged fraction of the foulants on the membrane surface. Results reported here are consistent with those reported by Xue *et al.* (2006). Organic fouling and a combination of organic and colloidal fouling led to a more negatively charged membrane surface. With increasing pH, the negative charge of the membrane fouled with humic acid alone increased significantly. This is due to the additional dissociation of carboxylic groups, which are present in the humic acid. In the presence of colloidal matter, the effect of pH on the membrane surface charge is slightly reduced, indicating that the charge of the colloidal matter is likely to be the dominant factor. Hence, humic acid and colloidal fouling could possibly contribute to further electrostatic interactions and therefore could aid in further increasing solute retention. Membrane fouling caused by a combination of both humic acid and silica colloid led to an increase in salt retention most likely because of pore restriction and enhanced electrostatic interactions. Conversely, fouling caused only by colloids is known to induce the cake-enhanced concentration polarisation (CECP) effect, which could explain a lower conductivity retention by the colloidally fouled membrane as can be seen in Fig. 2b. CECP results in higher concentrations

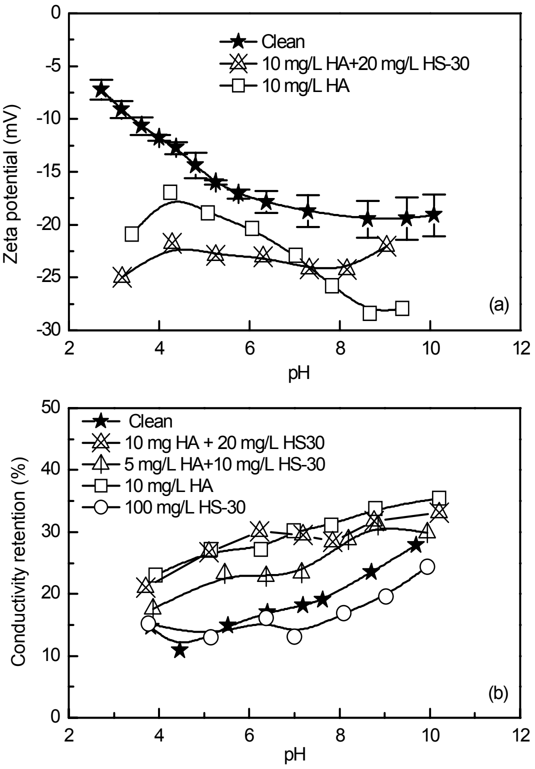

Fig. 2 (a) Zeta potential of the clean and fouled NF270 membrane as a function of pH. The error bars show standard deviation of three repetitive measurements. The background electrolyte was 1 mmol/L KCl; titration was made with 100 mmol/L KOH and 100 mmol/L HCl; (b) Impact of organic and colloidal fouling on conductivity retention as a function of pH. Experimental conditions were the same as shown in Fig. 1

of inorganic salts on the feed side of the membrane surface due to hindered back diffusion. Subsequently, CECP could lead to an increase in the transport of inorganic salts across the membrane (Hoek and Elimelech, 2003).

Consequently, as shown in Fig. 2b, the colloidally fouled NF270 membrane indicated a notable decrease in conductivity retention. This is especially obvious in basic environments. After the fouling layer was developed with a mixture of both organic and colloidal matter, an increase in conductivity retention was observed. This increase was slightly lower than the one observed for the membrane fouled with humic acid alone. Hence, CECP seems to be almost negligible in presence of humic acid.

3.3 Effects of organic/colloidal fouling on sulfamethoxazole retention

The retention of sulfamethoxazole is expected to vary as a function of the background solution pH because the retention is dependent on the charge of sulfamethoxazole. Fig. 3a shows the ratio of the molecular fraction of different charged species of sulfamethoxazole as a function of pH. When the solution pH coincides with the second pK_a value (5.81) of the compound, sulfamethoxazole exists in both neutrally and negatively charged forms at the same fraction. As the solution pH increases to beyond this pK_a value, sulfamethoxazole can be deprotonated further and the fraction of negatively charged species increases sharply to 100% at approximately pH 8. Conversely, as the solution pH decreases to below this pK_a value, sulfamethoxazole can transform to a neutral form, which becomes the dominant species at pH 4. When the solution pH decreases further to below its first pK_a value (1.39), sulfamethoxazole can acquire a positive charge. With a logK_{ow} value of 0.659 (Table 2), sulfamethoxazole can be classified as quite hydrophilic. Hence, adsorption of this compound into the membrane and into the fouling layer can be neglected.

Fig. 3b shows the retention of sulfamethoxazole by the clean and previously fouled NF270 membrane. The highest retention of sulfamethoxazole (98%) could be achieved in a strong caustic background solution (pH 10), where sulfamethoxazole is negatively charged. Below its pK_a value of 5.81, where sulfamethoxazole is neutral, the retention was quite low (approximately 15%).

Fig. 3b also indicates the role of membrane fouling caused by humic acid, colloidal particles, and a mixture of both on the retention of sulfamethoxazole. In a caustic environment (above pH 8) the retention of the negatively charged sulfamethoxazole shows no significant variations.

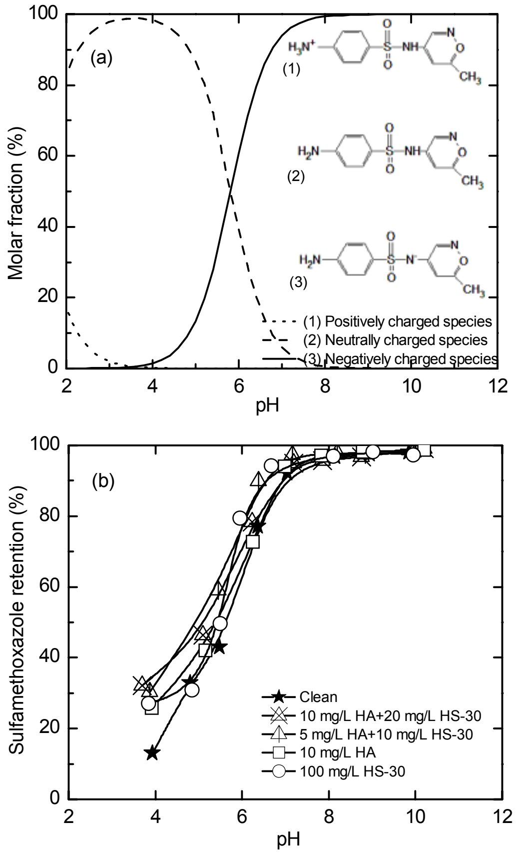

Fig. 3 (a) Speciation of sulfamethoxazole as a function of pH. Sulfamethoxazole pK_a values of 1.39 and 5.81 were used for calculation; (b) Impact of humic acid, colloidal fouling and a combination of both on the retention of sulfamethoxazole. Experimental conditions were the same as shown in Fig. 1

Conversely, at the vicinity of the compound's second pK_a value and under neutral conditions, humic acid fouling resulted in an increase in sulfamethoxazole retention possibly due to pore restriction. Organic fouling and a combination of organic and colloidal fouling caused a significant increase in the negative charge of the membrane, which resulted in an increase in membrane swelling and the narrowing of the membrane pores. Therefore, a slight increase in retention of the neutral sulfamethoxazole by the silica colloidally fouled membrane could be observed (Fig. 3b). It is noteworthy that in another study, a significant decrease in sulfamethoxazole retention by colloidally fouled membranes was reported (Nghiem *et al.*, 2008b). The reported retention ability for sulfamethoxazole by a colloidally fouled NF270 membrane was 60% in caustic environment and 0% in acidic environment, which was not observed in this study. This was probably due to differences in the protocols and concentrations. Membrane fouling with colloids and humic acid caused the highest retention of sulfamethoxazole in the lower pH range. This was most likely due to pore restriction and induced negative charge to the membrane. It can be furthermore assumed that the fouling layer caused by a combination of both humic acid and silica colloids is very compacted, which would mask any CECP effect. Consequently, a reduction in sulfamethoxazole retention by the fouled NF270 membrane involving the silica colloids was not observed in this study.

4 Conclusions

Results reported here indicate that membrane flux decline was dependent on the model foulants. In particular, organic fouling caused a severe flux decline due to pore blocking and adsorption directly after the start of the fouling layer development. Adsorption and pore blocking were absent for the colloidal fouling process, hence, the flux decline was found to be only slight and gradual throughout the experiment. A combination of organic and colloidal fouling led to a severe flux decline in comparison to that caused by either of the individual foulants alone. Membrane charge played a significant role in the separation process of inorganic salt, where the retention was the highest in caustic environment (high pH) due to more swollen membrane material caused by the higher negative membrane charge. Organic fouling and a combination of both organic and colloidal fouling caused a significant increase in the negative membrane charge. The influence of membrane fouling on the retention of inorganic salts and sulfamethoxazole was found to be dependent on the fouling behaviour and the physicochemical properties of the model foulants, where the model foulants probably contributed to the tendency to increase the solute retention by electrostatic interactions. Organic fouling caused an increase in solute retention due to pore blocking. In contrast, colloidal fouling caused a decrease in the retention of inorganic salts due to CECP. A mixture of colloidal and organic matter improved the retention of salt and neutral sulfamethoxazole. Pore blocking by humic acid was, in this case, thought to be the predominant mechanism, which seemed to be able to mask partly the CECP phenomenon usually observed on colloidally fouled membranes.

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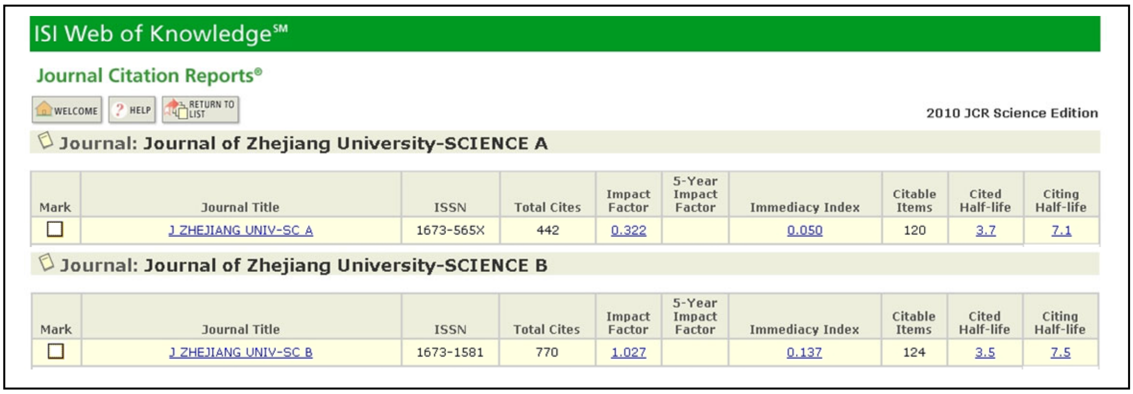

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