Lab-Scale Nano-Filtration Membrane Performance With High Silica Concentration Feed Water

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Abstract

Water scarcity emphasizes the necessity of water recovery from saline sources, which are plentifully available. Nanofiltration (NF) is widely implemented in water reclamation treatment particularly in industrial field. However, the presence of silica hinders the possibility of obtaining a high recovery in a NF membrane system. In this work, the effect of silica concentration on the scaling of a NF membrane was investigated. Three types of feed water (demineralized water, water with 100 mg L^{-1} and 300 mg L^{-1} of SiO_2) were fed to a single stage nanofiltration element. All cases were carried out at room temperature, pH values ranged 7 - 8 and a constant recovery of 11±1% for 24 hours continuously. The mass transfer coefficient (MTC) was used as method to determine the scaling of the membrane module. Results showed that no silica scaling occurred during the system runtime. Nonetheless, for all cases, an initial decrease in MTC could be detected. On the one hand, based on some literature reports, it is likely that a part of silica (mainly colloid) had precipitated on the membrane surface forming a more porous layer than the precipitation of polymerized monomeric silica. But to verify this argument, a further autopsy method is demanded. On the other hand, concentration polarization may also contribute to the initial decline in mass transfer coefficient. But the reason for the decrease for demi-water case remains unknown. In brief, Nanofiltration membrane can operate at a silica concentration up to 300 mg L^{-1} for 24 hours in lab-scale. The guideline (120 mg SiO_2 L^{-1}) suggested in the industrial standard (ASTM, D4933-08) was judged to be relatively conservative. For more practical purpose, further study is needed.

Keywords: Nanofiltration, brine, silica scaling, high silica concentration

1. Introduction

Water scarcity is an undeniable fact around the globe (Haidari et al., 2017). Rapidly growing population, environment deterioration and global climate change accentuate the water scarcity around the globe (Qu et al., 2013). Under such an intractable situation, water recovery from saline water resources is considered as an effective solution to cope with this water utility stress. Especially in industry, water reclamation is widely applied to reduce the degree of dependency on fresh water (Bixio et al., 2006) particularly fresh groundwater resources (Heijman et al., 2009). In the last decade, zero liquid discharge (ZLD), a concept about wastewater management strategy that aims at eliminating the liquid discharge in the form of wastewater and maximize water usage efficiency, has drawn worldwide interest (Tong & Elimelech, 2016). At the same time eliminating liquid discharge leads to the production of saline waste streams (brines), which calls for solutions to manage these streams. For instance, within the European Union (the EU), this concept is embodied by a grant agreement (No. 730390) of which the main goal is to re-design a water and minerals circular economy, known as zero brine project (EASME, 2016). Accordingly, achieving a higher water recovery (>95%) is one of the tasks of this project.

Nano-filtration (NF) is usually applied as a pretreatment to reverse osmosis (RO). In industry, both technologies are prevalently adopted in water reclamation systems, due to their small footprint and high outflow quality (Qu et al., 2013; Voorthuizen et al., 2005). However, fouling (mineral scaling and bio-fouling) of NF and RO systems is almost inevitable, which limits the attainable product water yield (Bacchin et al., 2006; Greenlee et al., 2009; McCool et al., 2013). Among all the foulants, silica is especially a challenge since it is hard to be removed neither by backwash nor by harsh chemical cleaning once this scaling occurs (Koo et al., 2001; Lin et al., 2014). In addition, harsh chemical cleanings are threats to membrane lifespan, especially for polymeric membranes where the membrane skin layer is sensitive to chlorination and oxidants.

As a result, three conventional approaches are applied to alleviate the impact of silica

scale formation during water reclamation: (1) precondition: to treat ions below a certain level, regardless the concentration of silica, for example, to keep aluminum and iron concentration below 0.05 mg L^{-1} (Antony et al., 2011); (2) pretreatment: to reduce the silica concentration, like by coagulation at high pH, softening using soluble magnesium compounds (Latour et al., 2014); (3) simply, by using antiscalants to disturb the scaling process (Ning, 2002; Salvador Cob et al., 2015). Hitherto, researches on this topic have mainly concentrated on method 1 and 3 but restricted on the second method.

According to ASTM (American Society for Testing and Materials) standard (D4933-08), it is recommended to operating a RO system with a SiO_2 concentration in the concentrate flow below a certain level. At a temperature of 25 ℃, this is 125 mg SiO_2 L^{-1}, which means for a membrane system with a recovery of 90%, SiO_2 concentration in the feed flow should be maximum around 12 mg SiO_2 L^{-1} ; or for a system with a recovery of 50%, it should be 60 mg SiO_2 L^{-1}. It can be seen that, obtaining a higher recovery means increasing scaling potential, unless intensive pretreatment is applied or proper antiscalants are used. The scaling as the consequence of higher recovery will lead to more frequent replacement of membrane modules and increase of operational costs. While most treatment plants adjust their recoveries based on the ASTM value (120 mg SiO_2 L^{-1}). Few literature showed a possibility of operating NF/RO installation function with feed water in higher SiO_2 concentration than that suggested by ASTM standard without scaling.

In a study focused on flux of a RO system, the system was fed with water containing calcium, magnesium and various SiO_2 concentrations (100 ~ 500 mg $SiO_2 L^{-1}$). It was found that the flux drop is inversely related to the initial concentration of silica given a plausible reason that deposit of silica in a high SiO_2 concentration is more porous than that in a low SiO_2 concentration (i.e. scale in a low SiO_2 concentration is more severe than scale formation in a high SiO_2 contaminated solution) (Sheikholeslami et al., 1999; Sheikholeslami et al., 2000). The same authors showed in later research (2001) that an increase of initial silica concentration (200 ~ 600 mg $SiO_2 L^{-1}$) enhances the silica polymerization process (i.e. scaling formation). The performance of membrane differs

from various pH values suggesting pH above 9.5 or below 5.5 is preferable if the feed includes SiO_2 but in absence of cations (Sheikholeslami et al., 2001). A 500-hours-long monitoring batch experiment reported by Koo et al. (2001) showed that at silica concentration of 300 mg L^{-1} or lower, the solution remains meta-stable for a long time but polymerization will occur at silica concentration greater than 300 mg L^{-1} even without cations (Ca^{2+} and Mg^{2+}). Haidari et al. also demonstrated that no scaling is observed in the absence of antiscalant and with a feed water containing up to 280 mg L^{-1} of SiO_2 during 8 hours running time. A recent article, in which a real-time monitoring of RO membrane performance was conducted, demonstrated that silica scaling takes place significantly earlier than suggested by the detection of measurable flux decline (Thompson et al., 2017). It can be seen that the upper level of feasible silica concentration in the feed water is still under discussion.

In this research, the quantity of membrane-produced water by NF membrane under constant recovery was studied and much effort was put into the membrane performance with respect to silica scaling. The goal of this study was to investigate whether nanofiltration membrane can operate at high silica concentration up to 300 mg SiO_2 L^{-1} without decrease in performance (i.e. constant recovery) within 24 hours. It should be noted that 24 hours was set as the runtime for each lab-scale experiment, which was a rather short time for full scale experiments or for real situation to detect silica scaling. However, it was still considered as a valid indication for future research on high silica concentrated wastewater treatment by NF/RO method. Mass transfer coefficient (MTC) was used to judge the membrane performance. Meanwhile, it was assumed that the silica scaling will immediately result in MTC drop. Afterwards, results and discussion were presented followed by a conclusion with some recommendations for future study.

2. Theoretical background

2.1. Silica chemistry, solubility and scaling

Silica, $(SiO2)_n$, is the generic name given to compounds that are composited by silicon dioxide (SiO_2). According to the molecular structure, silica is classified as crystalline silica

and amorphous silica (for any silica lacking crystal structure) (Iler, 1979). Silicate exists when silicon dioxide is bond with other metal elements. Once silica or silicate is presented in water, it exists in many forms and can be further categorized into dissolved silica (prevalently known as monomeric silica or reactive silica), unreactive silica including colloid silica and particulate silica (usually called suspended silica) (Ning, 2002; Sheikholeslami et al., 1999). In the literature, monomeric silica is also variously named as silicic acid, orthosilicic acid, monosilicic acid or silicic monomer. Solubility of silica increases by temperature and at temperature of 25 ℃, it is about 125 mg L^{-1} (ASTM, D 4993-08). Solubility is also sensitive to pH . The lowest silica solubility happens at pH range from 7 to 8. The stability of monomeric silica depends on the silica concentration. In low silica concentration, it is meta-stable and the polymerization proceeds slowly, whereas this process is accelerated with increasing concentration. Silica ions tend to bond with each other or other (in)organic ions forming polysilicic acid. If the aggregation continues, more highly polymerized colloidal silica will be present which can further form particulate silica, although in a very low rate (Iler, 1979; Sahachaiyunta et al., 2002; Semiat et al., 2003). This is often the case in silica-supersaturated solutions. Moreover, its un-ionized property makes it hard to be removed by pretreatment and even provokes fouling in processes like softening or ion exchange (Sheikholeslami et al., 1999).

Fouling takes place in NF/RO systems if the dissolved silica exceeds its solubility. In the literature, silica fouling is divided into two types: the precipitation fouling and particulate fouling (Semiat et al., 2003; Sheikholeslami et al., 1999; Sheikholeslami et al., 2000). The former is the precipitation of polysilicic acid on the membrane surface forming an impervious glasslike film. Thus, this kind of fouling is widely known as scaling. The other type of fouling refers to accumulation of colloids that exist initially in the bulk solution. Accumulated silica colloids are subsequently deposited onto the membrane surface forming a film with a more porous structure.

Besides the initial silica concentration, pH of the solute and ambient temperature, the presence of other species also contribute to the formation of silica scale (Gabelich et al., 2005; Iler, 1979; Kimura et al., 2013; Thompson et al., 2017; Tong et al., 2017). In this

study, the working conditions were set to: the pH around 7.5±0.5 for the feed water, 25±5 ℃ for operating temperature and demineralized water as solute.

3. Materials and Method
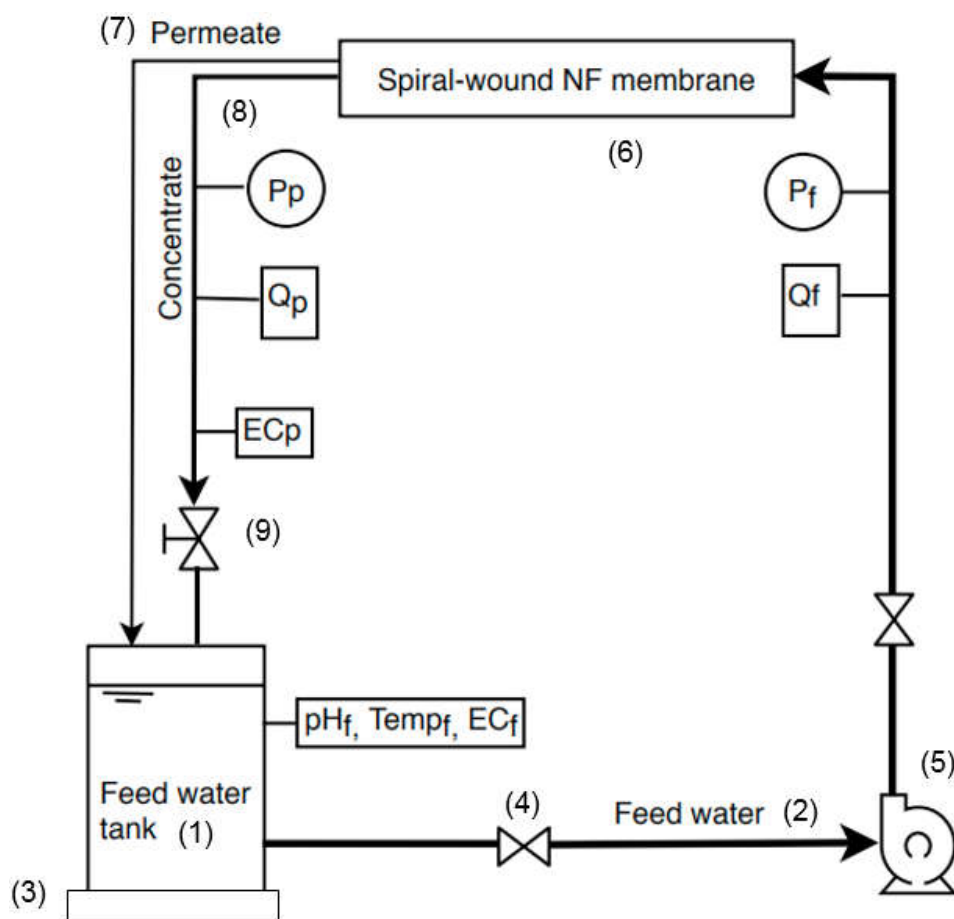
3.1. Apparatus and experimental procedure

Fig. 1 Experimental Setup

The experimental setup is shown in Fig. 1. All experiments were started at room temperature of 20 ℃. The feed water tank (1) was put on a wooden step (3) and covered with aluminum foil to limit the temperature variation caused by ambient changes. Feed water (2) was firstly pumped (5) to the NF membrane module (6). The generated permeate (7) and concentrate (8) flows were returned to the feed tank (1) to keep the same feed water concentration at all time.

During the operation period (24 hrs), temperature, electric conductivity and pH of feed water and electrical conductivity of concentrate flow were measured every 30 minutes. Meanwhile, flow rates of feed water and concentrate were collected with a frequency of 10 per second (default value of the software used for data logging) and the average was adopted for the following analysis. Moreover, the trans membrane pressure was determined by measuring the mean pressure at both the feed side and the concentrate side at the same measuring frequency as that of the flow rate.

In this study, recovery (11±1%) was directly achieved by applied pressure and feed flow rate, which were regulated via operating the rotational speed of the pump and the open degree of a valve installed in the concentrate stream. For all experiments, trans membrane pressure was about 1.5 ~ 2.0 bar (see table 1).

3.2. Nano-filtration membrane

The examined Nano-filtration membrane was a spiral-wound Nano-filtration membrane manufactured by DOW (DOW FILMTEC™ NF90-2540). It has a diameter of 61 mm (2.4 in inch) and is featured by an effective filtration area of 2.6 m^2; the maximum operating temperature is 40 ℃. The Molecular Weight Cut-off (MWCO) of this membrane is unknown. According to Salvador Cob et al.(2015), silica can be partly retained in the concentration stream, therefore, in this study it was assumed silica was at least 50% retained in the concentrate. Tested demi-water permeability at 25 ℃ was about 8.28 L m^{-2} h^{-1} bar^{-1}. An autopsy report provided by Haidari (personal communication) showed that for this type of membrane there are two envelops which create two feed channels for water purification, and the cross sectional area of the feed channel was 0.684 × 10^{-3} m^2 (0.9 m × 0.76 mm). It was assumed that the feed spacer porosity was about 80%.

3.3. Method of determining the operating condition

3.4.1. Cross flow velocity

Cross flow velocity was applied as a parameter to judge the applied feed flow rate whether it was sufficient to minimize the concentration polarization effect (an effect leads to an

initial rapid decline in flux) at an acceptable energy cost and without damaging the membrane with respect to shear force (Haidari et al., 2017; Heijman et al., 2009). The cross-flow velocity in the spacer-filled channel can be calculated by using eq. 1 (Haidari et al., 2017).

$$V = \frac{Q_f}{A} = \frac{Q_f}{W \cdot H \cdot \varepsilon} \qquad \text{Eq.1}$$

where, Q_f is feed flow rate in each feed channel, in $m^3 \ s^{-1}$; A is the cross section area of one feed channel; W is the width of channel in m (W = 0.9 m),; H presents the height of the channel in m (H = 0.76 $\times$ 10^{-3} m),; and ε refers to the spacer porosity ($\varepsilon = 80\%$). In this study, the NF membrane system operated at a constant cross flow velocity of about 0.15 m s^{-1} which provided a feed flow rate of circa 0.3 $m^3 \ h^{-1}$.

3.4.2. Recovery

Recovery is a parameter that indicates the ratio of permeate flow of the tested membrane system to the feed flow. In many industrial water treatment schemes, membrane elements are placed in a series to obtain their desired recovery. To simplify the complexity of experiments, a single element was used for testing and recovery was set to be constant at 11±1% (i.e. flow rates for feed flow and for concentrate flow were kept constant for experiment No.1 to No.3 by pressure adjustment); except experiment No.4 of which pressure was the same as experiment No.1.

3.4. Artificial feed water

To explore the performance of membrane under various silica concentrations, feed water containing 100 mg L-1 and 300 mg L-1 silica (SiO2) with pH (around 7) were tested respectively.

The chemical used for all experiments was sodium silicate solution (Na2O7Si3, SiO2 composition: 26.5%; density: 1.39 g mL-1 at 25 ℃) produced by Sigma-Aldrich Chemistry. The artificial feed water for all experiments was prepared by diluting this viscous sodium silicate solution with demineralized water (di-water, electric conductivity < 5 μS cm-1).

Meanwhile, hydrochloride, of which the concentration was 1.2 mol L-1, was utilized for pH adjustment (preferable within 7 to 8, as aforementioned). A summary of the quality of feed water is listed in Table 1.

Table 1 Characteristics of feed water

Experiment ID*	Feed composition	Silica concentration mg L^{-1}	Electric conductivity µS cm^{-1}	working condition recovery/pressure
No.1	Demineralized-water	-	5 ± 3	11±1% /1.5±0.05bar
No.2	$Na_2O_7Si_3$ + HCl	100	100 ± 20	11±1% /1.5±0.05bar
No.3	$Na_2O_7Si_3$ + HCl	300	500 ± 50	11±1% /1.9±0.05bar
No.4	$Na_2O_7Si_3$ + HCl	300	500 ± 50	7±0.3% /1.5±0.5bar

* the same membrane was used for all experiments. Experiment ID referred to the time sequence (i.e. demi-water case was conducted first followed by 100 mg SiO$_2$ L^{-1} case and ended up with 300 mg SiO$_2$ L^{-1} case).

To see a lower pressure will still cause a decrease in flux meaning that the silica accumulation, because the cross flow rate is lower than the retention time is longer. A more rapid decrease at the very beginning.

3.5. Theoretical analysis

3.6.1. Mass transfer coefficient

As pre-stated, mass transfer coefficient (MTC) was used to estimate the performance of the membrane system. A decrease in this parameter implies a deteriorated membrane workability which, is a sign of silica scaling. MTC, in m s^{-1} Pa^{-1} , is calculated based on eq. 2.

$$MTC = \frac{(Q_f - Q_c) \times TCF_{MTC}}{A \times NDP \times 3.6 \times 10^6} \qquad \text{Eq.2}$$

where, Q_f and Q_c are feed flow rate and concentrate flow rate, respectively in m^3 h^{-1}; A stands for the effective membrane surface, 2.6 m^2; TCF_{MTC} means temperature correction factor used to convert MTC to a more comparable normal temperature condition. This is considered due to the fact that temperature affects the water viscosity, which influences the ability of water to diffuse through membrane pore (e.g. mass

transfer). TCF_{MTC} is obtained via eq. 3.

$$TCF_{MTC} = 3200 \times \left(\frac{1}{T+273.15} - \frac{1}{T_{ref}+273.15} \right) \qquad \text{Eq.3}$$

where, T (in ℃) is the measured temperature and T_{ref} is the room temperature (20 ℃).In addition, in eq. 2 NDP (in kPa) , short of net driving pressure, represents the driving force for a membrane process and in some literature also called net trans membrane pressure (TMP_{net}). It can be computed by using eq. 4

$$NDP = \left(\frac{P_f + P_c}{2} - P_p \right) - \left(\frac{\pi_f + \pi_c}{2} - \pi_p \right) \qquad \text{Eq.4}$$

in which P and π are the static pressure and osmotic pressure, respectively, and the subscripts (f, c, p) refer to feed water, concentrate and permeate, respectively. In this study, P_p and π were assumed to be zero.

Both P_f and P_c were provided by two digital pressure meters. The osmotic pressure was obtained by using the following formula (see eq. 5)

$$\pi = EC_{20^o C} \times 1.0911 \times 0.0433 \times TCF_p \qquad \text{Eq.5}$$

in which TCFp is another temperature factor which takes into consideration the temperature impact on the solubility of $Na_2O_7Si_3$. Eq. 6 is used for calculating the TCF_p.

$$TCF_p = \frac{273.15+T}{273.15+T_{ref}} \qquad \text{Eq.6}$$

in which the parameters are the same as defined in eq. 3.

In this study, electric conductivity (EC) was used to indirectly measure the silica concentration of the feed water and to further determine osmotic pressure. This method is applied because determining silica concentration with available instruments was not either easy or accurate enough to perform (as it usually exclude inactive silica). However, it should be noted that the EC in the tested solution was the result of presence of sodium ion and chlorine ion, as silica ions are uncharged. Therefore, empirical coefficients,

1.0911 and 0.0433, were used to convert EC value to salt concentration in solutions.

Furthermore, an empirical formula (eq. 7) was used to standardize the measured EC to room temperature (℃)（personal communication）.

$$EC_{25^oC} = \left(1.6799 - 0.046193 \times T + 0.00060 \times T^2\right) \times EC_T \qquad \text{Eq.7}$$

where T is the measured temperature in ℃; ECT is the conductivity at temperature T.
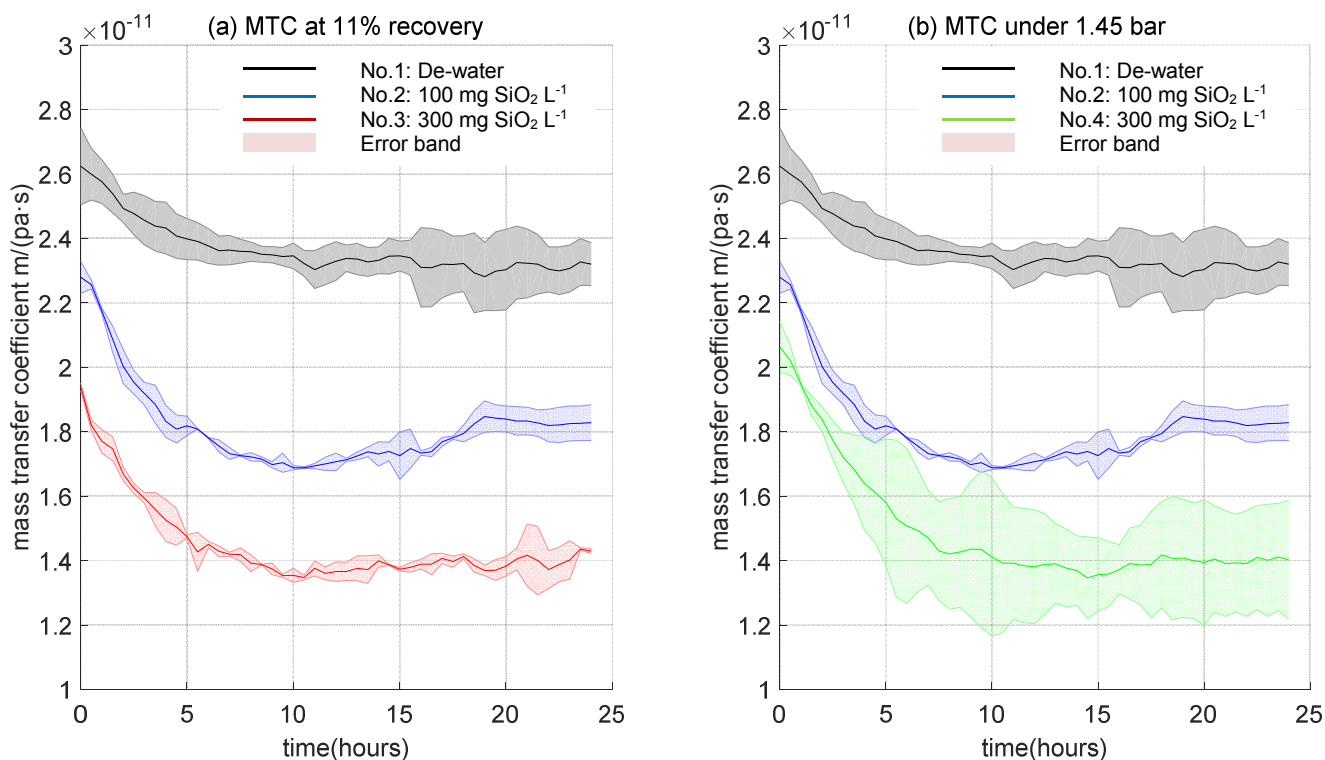
4. Results and discussion

Fig. 2 Mass transfer coefficient over the operation period (24 hrs) – Error band show the standard deviation from duplicate measurements except for experiment no.1 (triple).

Fig.2 shows the mass transfer coefficient (MTC) of the NF-membrane over time for demi-water, solution with 100 mg L^{-1} and 300 mg L^{-1} SiO$_2$, respectively. The x-axis represents the exam period from start of the experiment and y-axis represents the MTC of the membrane system during the 24-hours runtime duration. In Fig.2 (a), the constant

recovery module, no steep MTC decrease happened for all experiments after stabilization meaning no existence of serious silica scaling. This answer the question that the tested nano-filtrtion membrane can operate at constant without decrease in performance in 24 hours duration. Based on this future study could extent the experiment duration to a longer time for a more practical purpose.

As shown in Fig.2 the highest MTC appeared at the experiment fed with demi-water, around 2.3 m Pa-1 s-1. As the amount of silica in the feed solution increasing, a drop in the value of MTC can be detected: feed water with 300 mg L-1 silica exhibited the lowest MTC, approximately 1.4 m Pa-1 s-1, which is 39% lower than no.1 (demi-water) and 22% less than no.2 (100 mg SiO2 L-1). This is reasonable because the resistance for mass transfer is increasing when the silica concentration in feed water increases and mass transfer coefficient is inversely proportional to this resistance. Another plausible explanation could be that as assumed before (in section 3) silica was at least 50% retained in the concentrate meaning the concentration polarization can also contribute to the drop in flux when fed with more silica concentrated water. But since the silica concentration in the permeate was not measured in this study, it is difficult to quantify the impact due to concentration polarization, which could be a task for future study (for example, using ICP-MS). It would be also interesting to notice that the flux has already been different at t=0 hour. The reason for this phenomenon could be that the time for starting data logging and for running the set-up was not concurrent. Data logging started when pressure adjustment finished (to ensure the same recovery or flux for each experiment). Thus there was a time delay due to which the initial MTC had been different already.

In addition, for experiments no. 2 to no.4, MTC declined sharply almost immediately when experiments started and took about 6 hours to reach a relative steady state. This was consistent with results from Semiat et al. (2003) and Lin et al. (2014) who also reported an apparent decreasing trend at the very first few hours and a 6-hour stabilization time. The reason for this initial distinct decrease in MTC, could be that silica deposited on the membrane surface immediately when filtration started and this process continued for

about 6 hour until it reached a (dynamic) balance. When it comes to the silica sedimentation compounds, according to the chemistry of silica (section 3.1), the most likely species of the deposits would be the colloidal silica that is less harmful than monomeric silica. An autopsy research (method like XRD) on the development of deposited silica species in different time series would be needed and interesting for future study. Additionally, by comparing no.3 and no.4, both of them reached the same stable MTC value, around 1.4 m Pa^{-1} s^{-1} which probably means that the sedimentation rate of colloidal silica is independent from applied pressure.

However, noticeably, for experiment no.1, MTC also exhibited a decrease while the feed solution contained only demi-water and the membrane was new, though its descending slope was milder compared with the rate of the other three tests of which slopes were almost equal. Two plausible reasons could explain this. Firstly, demi-water is water with very low concentration of charged ions (i.e. low electric conductivity) which means it could also contain a certain amount of uncharged particles like organic materials. Secondly, as demi-water was not entirely pure water the concentration polarization effect (CP effect) may play an important role in the initial decline for demi-water test.

One last thing worth paying attention to is, as depicted in Fig.2 (a) and (b), MTC of test no.2 showed an increasing trend after 15 hours runtime and reached another relatively stable value until the end of the experiment. This was probably because after the first 15 hours of colloidal silica sedimentation, the silica concentration in the bulk solution had dropped meaning a decreasing in resistance for mass transfer. Besides, the layer formed by deposit silica was so porous that can allow water pass through easily so that the presented MTC was slightly higher and reached another steady state. However, for a more silica concentrated feed water (test no.3), there was no sign of such a phenomenon. An explanation for this could be that no.3 had a larger buffer of silica (300 mg L-1 silica) in the bulk solution than no.2 (100 mg L-1 silica) so that it takes a long time to detect the increase in flux due to less amount of silica in feed solution. If the explanation for this increase was considered true then a rising tendency of no.3 would also appear but later in time if the experiment continues.

5. Conclusion

This study investigated whether a commercial nano-filtration membrane can operate at high silica concentration up to 300 mg SiO_2 L^{-1} without decrease in performance during 24 hours and conclusions were drawn as follows:

- In lab scale, NF-90 membrane can keep its performance providing constant flux when fed with water containing up to 300 mg L^{-1} of silica within 24 hours.

- During the time for stabilization, decrease in mass transfer coefficient could be the result of silica sedimentation; the degree of drop in mass transfer coefficient relates to the concentration polarization effect and the initial silica concentration in feed solution;

- Future research can focus on extending the exam duration, type of deposit silica species and quantifying the concentration polarization impact by measuring the silica concentration in the permeate stream.

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