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Method Development of Fouling Index Measurement for Membrane Fouling Potential Evaluation

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Abstract: Modified Fouling Index (MFI) serves as an indicator for characterizing the fouling potential of a feedwater. This work focused on the development of membrane fouling index measurement method for the evaluation of membrane fouling potential. Crossflow sampler (CFS) was incorporated into the development of fouling index to investigate the influence of CFS on the MFI tests. *Colloidal silica* with the particle size of 70-100 nm was adopted as a model foulant. Results were verified based on the effect of colloidal silica concentration (i.e., 0, 50, 100 and 200 mg L) on the MFI and CFS-MFI values. Polyvinylidene fluoride (PVDF) ultrafiltration membranes with Molecular Weight Cut-Off (MWCO) of 150 and 100 kDa were employed to study the effect of membrane MWCO as the test membrane in the dead-end cell on the fouling indices. Through the experimental results, the best membrane for the colloidal silica particles was determined. The results indicated that the fouling indices through PVDF 100 membrane were higher than that of PVDF 150 due to the retention of particles on membrane with smaller MWCO. In terms of the silica concentration effects, the fouling indices increased significantly with the increase in feed silica concentration. The rate of increment for the fouling indices through PVDF100 membrane (R^2~0.99) was found to have a stronger degree of linear relationship with the silica concentration than PVDF150 (R^2<0.75), indicating that the PVDF100 membrane was a more appropriate test membrane for fouling index tests on model foulant of colloidal silica (70-100 nm).

Key words: Constant flux, fouling prediction, ultrafiltration, colloidal silica particle

INTRODUCTION

Membrane fouling is a persistent problem in this advanced technology. Membrane fouling potential can be predicted by determining the Fouling Indices (FI) of the feedwater. Existing fouling indices used to measure the particulate and colloidal fouling potential include Silt Density Index (SDI) and Modified Fouling Index (MFI). They are recognized as the popular fouling predictive tools in Reverse Osmosis (RO) applications. The reliability and effectiveness of fouling indices, however, are indeed questionable due to its deficiencies. For instances, the SDI and $MFI_{0.45}$ tests cannot simulate crossflow filtration characteristics as RO system and microfiltration (MF) membrane with pore size of 0.45 μm is too large to retain small particles (Shon *et al*., 2009). Boerlage *et al.* (2002) investigated the effect of membrane pore size on the MFI through ultrafiltration (UF) membranes. They revealed that the MFI_{UF} was significantly higher than the $MFI_{0.45}$ for tap water, implying that the relatively large particles were retained by the UF membrane.

Several researchers have further contributed to the development of fouling index for better prediction of fouling problem in membrane filtration process (Koo *et al.*, 2013). Javeed *et al.* (2006) integrated a crossflow sampler (CFS) operating under the similar crossflow conditions as RO process into the MFI test (i.e., CFS-MFI). Despite of the fact that extensive studies have been conducted to improve the experimental method, the homogeneity of tests is equally important as considering the CFS at the upstream and applying smaller pore size of membrane in dead-end cell. Such effects are usually overlooked in the measurement of FI.

This study focused on the development of membrane fouling index measurement method for the evaluation of membrane fouling potential. Crossflow

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sampler was incorporated into the development of fouling index to investigate the influence of CFS on the MFI tests. The MFI and CFS-MFI values were carried out using an identical setup to ensure homogeneity of measurements. The performances of the setup were tested on colloidal silica suspension with four different concentrations using two different types of UF membranes. Method of evaluation based on the comparison and validation of results are presented to demonstrate the acceptability of this developed method. The validation of the method was carried out against its sensitivity towards particle concentration, UF membrane pore size and hydrodynamic effect of CFS in evaluating the reliability of setup.

Background: The origin of modified fouling index was derived based on cake filtration theory and it can be used to model the TMP increase while maintaining a constant flux in membrane systems (Sim *et al*., 2010). The $MFI_{0.45}$ is performed when the feed is introduced into a pore size of 0.45 μm MF membrane installed within in-line filter holder in dead-end filtration mode. The $MFI_{0.45}$ measured at a constant flux is expressed in Eq. 1 as follows (Sim *et al*., 2010):

$$MFI = \frac{\mu I}{2\Delta P_0 A^2} \quad (1)$$

where, μ is the filtrate solution viscosity, I is the resistivity of cake, ΔP_0 is the standard pressure of 2×10^5 Pa (2 bar) and A is the membrane area. The cake resistivity, I can be determined from the slope of the linear portion in the graph of transmembrane pressure (TMP) versus filtration time as presented in Eq. 2 (Sim *et al*., 2010):

$$TMP = J\mu R_m + J^2\mu It \quad (2)$$

where, J is the constant flux, R_m is the membrane resistance and t is the filtration time. Figure 1 illustrates a typical plot of TMP versus filtration time, t for determining the cake resistivity, I. The phenomenon of TMP increase indicated that an additional applied pressure is required to maintain a constant flux due to the formation of cake on the test membrane (Sim *et al*., 2010). I is substituted into Eq. 1 to calculate the values of MFI. $CFS\text{-}MFI_{const.flux}$ is determined using the same procedures and equations Eq. 1 and 2 as the MFI with the condition CFS cell presents at the upstream of dead-end cell.

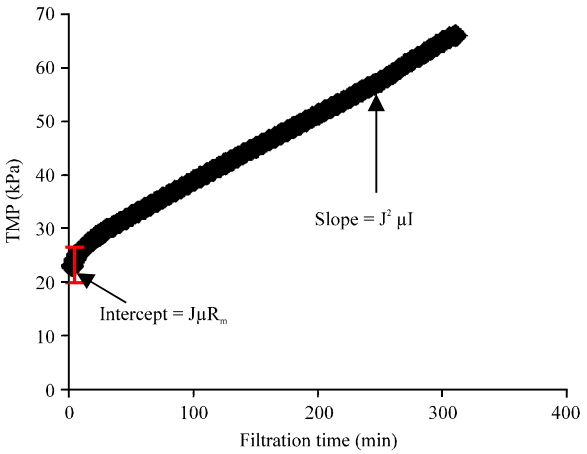


Fig. 1: TMP vs. t under constant flux filteration for determining the cake resistivity, I (Sim *et al*., 2010). Reprinted from (Sim *et al*., 2010) with permission from Elsevier

MATERIALS AND METHODS

Testing procedures and condition parameters: Figure 2 shows the schematic diagram of the equipment used for the MFI and CFS-MFI tests. The fouling index measuring device consisted of feed and collection tanks, a feed pump, a CFS cell (GE Osmonics©, Minnetonka, MN), flowmeters, pressure gauges, pressure sensors, a peristaltic pump (Cole-Parmer, Masterflex, US), a dead-end cell and data logging system. The effective membrane areas of CFS cell and dead-end cell were 0.0155 and 0.0095 m^2, respectively. All the experiments were commenced by pumping the feed suspension into the CFS cell using a piston-driven diaphragm pump (Hydracell pump, US). A peristaltic pump was installed in the CFS permeate line to withdraw permeate from the CFS cell and subsequently pumped into the dead-end cell. The crossflow velocity at the CFS cell was fixed at 0.39 m sec^{-1}. The constant flux at the dead-end cell was maintained at 30.9 $Lm^{-2} h^{-1}$ by using the peristaltic pump. The conceptual design flow of the MFI and CFS-MFI tests is presented in Fig. 3. For the test measurement of MFI, the feed was first introduced into the CFS cell which was not installed with any membranes, followed by a permeate flow into the dead-end cell for MFI measurement. The function of the CFS cell is to simulate the crossflow filtration effect in the FI measuring devices as in RO system. The readings from the pressure sensors at the dead-end cell were logged in the data logging system. The MFI and CFS-MFI values were calculated using Eq. 1. All the experiments were repeated for at least twice to ensure the results were reproducible.

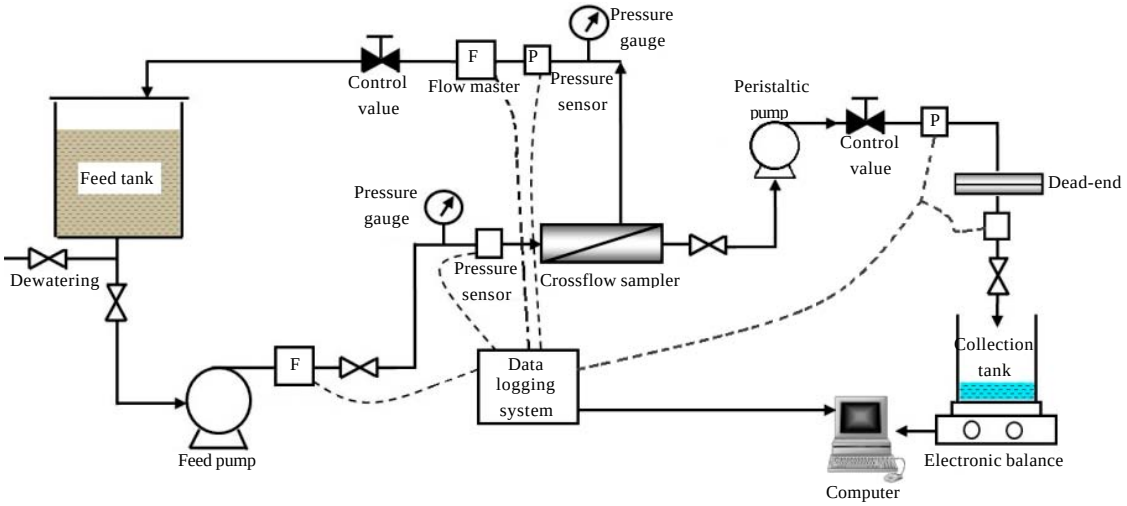
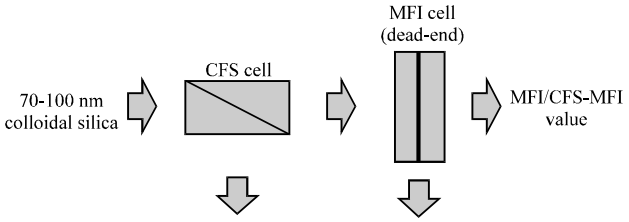


Fig. 2: Schematic diagram of the MFI and CFS-MFI setup under constant flux



Concentration (mg L^{-1})	Membrance tested	Membrance tested	Fouling index
0, 50, 100, 200	No membrance	150 kDa PVDF UF 100 kDa PVDF UF	MFI
	5µ PC MF		CFS-MFI

Fig. 3: A conceptual design flow of the MFI and CFS-MFI tests

Colloidal silica as fouling species: Feed suspension of colloidal silica was prepared by adding a buffer solution to obtain the desired concentration (i.e., 0, 50, 100 and 200 mg L^{-1}). The buffer solution was prepared by mixing 6.81 g of potassium dihydrogen phosphate (KH_2PO_4) with 467 mL of 0.1 M sodium hydroxide (NaOH) to produce a feed suspension of pH 8 (Sim *et al*., 2010). The buffer solution helps to enhance the solubility of colloidal silica in the feed suspension. The solubility of colloidal silica increases with the increase in pH greater than 7.8 (Karabelas, 2003). The mixture of suspension was then exposed to sonication treatment for duration of 10 min to ensure that the suspension was stable and free from any large aggregate (Hong *et al*., 2010).

Membrane: Flat sheet polyvinylidene fluoride (PVDF) UF membranes with the Molecular Weight Cut-Off (MWCO) of 150 kDa (denoted as PVDF 150) and 100 kDa (denoted as PVDF 100) were used in the dead-end cell. A polycarbonate (PC) MF membrane of 5 µm was employed in the CFS cell for the CFS-MFI measurement. The wettability of all membranes was measured using EasyDrop contact angle measuring instrument (KRÜSS GmbH, Hamburg) based on the standard sessile drop method. The properties of the membranes are shown in Table 1.

Table 1: Properties of membranes

Membrane	Material	Pore size/ MWCO	Contact angle, θ (°)[a]	Manufacturer
PC	Polycarbonate	5 (µm)	87.9	Membrane solutions®
PVDF150	Polyvinylidene fluoride	150 (kDa)	91.6	Amfor Inc.
PVDF100	Polyvinylidene fluoride	100 (kDa)	92.9	Amfor Inc.

[a]Determined through easyDrop contact angle measuring instrument

RESULTS AND DISCUSSION

MFI and CFS-MFI determinations with ultrafiltration membranes: A comparison was made between MFI and

Table 2: Comparison of MFI and CFS-MFI for different membranes tested with ultra pure water and silica suspension of 50, 100 and 200 (mg L^{-1})

	MFI		CFS-MFI	
Silica concentration (mg L^{-1})	PVDF 150	PVDF 100	PVDF 150	PVDF 100
0 (Ultra pure water)	290	340	307	409
50	3851	4062	2740	3344
100	3950	6831	2907	5029
200	4219	11680	3748	10455

CFS-MFI tests filtering with colloidal silica suspension by using two different types of UF membranes (i.e., PVDF 150 and PVDF 100 membranes). Table 2 presents consistent trends among all values of MFI and CFS-MFI. Higher fouling index values were observed when filtering a higher concentration of feed silica suspension. The high fouling index is mainly attributed to the increase of particles deposition on the membrane surface as fouling potential increased with the increase in the feed concentration (Park *et al.*, 2007). It can thus be concluded that the fouling indices are proportional to the concentration of silica foulant.

The fouling indices for the membrane with small MWCO would be higher than that of large MWCO. Since, a decrease in MWCO corresponds to a decrease in pore size of membrane, the UF membrane with small MWCO (i.e., 100 kDa) tends to retain more particles compared to the membrane with large MWCO (i.e., 150 kDa). The retention of particle would lead to fouling mechanisms such as adsorption, pore plugging and concentration polarization and this may eventually cause higher TMP in the dead-end cell. A small MWCO membrane would also create a considerably high total filtration resistance which in turn reduce the permeability flux of membrane (Koo *et al.*, 2012).

Method performance evaluation: Figure 4a and b shows the relationships between fouling indices and colloidal silica concentrations in the MFI and CFS-MFI test for the PVDF 150 and PVDF 100 membranes, respectively. Apparently, the MFI and CFS-MFI values for the PVDF150 (Fig. 4a) have poor correlations with the silica concentration. On the contrary, the MFI and CFS-MFI values for the PVDF100 UF membrane Fig. 4b were linearly proportional to the concentration of silica, with the R^2 values for linear regression were as high as 0.989 and 0.994, respectively. The results implied that the FI failed to reflect the fouling potential of this silica when the PVDF150 UF membrane was employed. It is thus justified to conclude that the PVDF100 UF membrane is a more appropriate test membrane than the PVDF150 UF membrane for this type of colloidal silica.

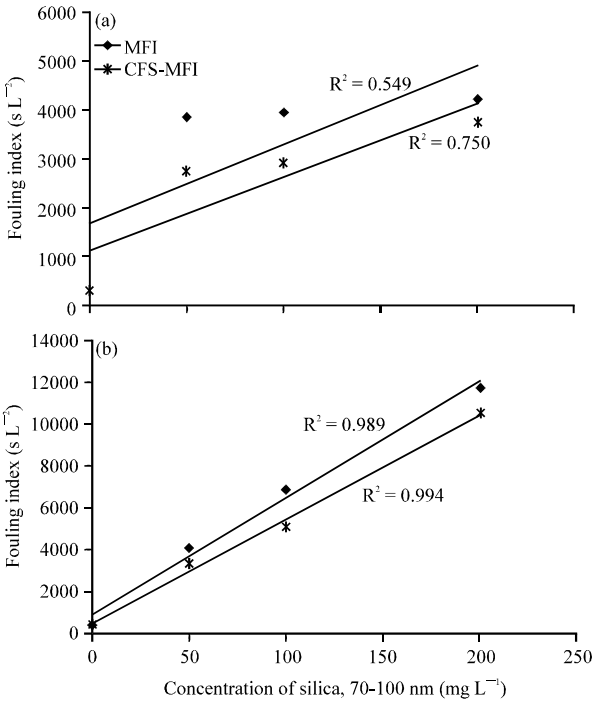


Fig. 4(a-b): Relationship between fouling indices and silica concentrations in the MFI and CFS-MFI tests. (Operating conditions: Crossflow velocity = 0.39 m sec^{-1}, flux = 30.9 Lm^{-2} h^{-1}, feed = 70-100 nm colloidal silica) (a) PVDF150 (b) PVDF100

CONCLUSION

A bench-scale fouling index measurement device was developed in this study. The performance of this fouling index measurement device was evaluated through a series of experimental tests. The effect of membrane employed (i.e., PVDF 150 and PVDF 100 UF membrane) on the MFI and CFS-MFI values was investigated. Colloidal silica with different concentrations was adopted as the model foulant in the tests. It was found that the fouling index increased significantly with the increase in the feed concentration. A plausible explanation to this observation is that the rise in silica loading may potentially create a much higher resistance of flow on the membrane. Higher

MFI-UF values were obtained for membrane with lower MWCO due to the capability of smaller pore size membrane to retain particles larger than the filter on the membrane surface. The results showed that the CFS-MFI values tend to be lower than the MFI values indicating that hydrodynamic effect plays an important role to simulate the crossflow filtration effect in RO system. From the R^2 values obtained, it is clearly shown that the PVDF100 UF membrane is a more appropriate test membrane for the MFI and CFS-MFI measurements due to higher linear regression between FI and concentration of silica.

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