

CHEMICAL CLEANING OF NANOFILTRATION MEMBRANES FOULED BY ORGANIC MATTERS

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Abstract

Membrane fouling is a term to describe non-integral substance on membrane surface which results in rapid decline of permeation flux and deteriorate the performance of membrane. Chemical cleaning agents especially like alkaline cleaners are most widely employed to restore the membrane performance. This research mainly investigated the potential use of sodium hydroxide (NaOH) and sodium hypochlorite (NaOCl) as the chemical cleaning agents to restore the permeate flux of organically fouled nanofiltration (NF) membranes under varying applied pressure and flow condition. The performances of the cleaning protocols were quantified using flux recovery and resistance removal. The results demonstrated that NaOCl is more effective than NaOH. This observation is also in line with FTIR analysis in which the transmittance intensity showed by FTIR spectra of NaOCl is higher than that of NaOH. The results also reported that higher flux recovery and resistance removal were achieved when the fouled NF membranes were cleaned with higher concentration of chemical agents and applied pressure. However, the improvements of flux recovery and resistance removal by increasing the applied pressure were found insignificant at higher applied pressure range (16 to 18 bar) than the lower applied pressure range (i.e. 12 to 14 bar). This research plays an important role by identifying the key parameters that could restore the flux of organically fouled NF membranes significantly.

Keywords: Cleaning agent, Flux recovery, Process variable, FTIR.

1. Introduction

Since 1970s, membrane plays an important role in treating the polluted water, as well as for human consuming and industrial usage. Unfortunately, this advanced technology often experiences membrane fouling which is an inevitable hurdle to

the expansion of its application [1]. Fouled membrane requires an extensive cleaning to maintain high flux.

Membrane fouling is commonly defined as an undesirable formation of deposits, results in the rapid decline of the permeation flux with time and cause poor membrane performance. Generally, foulants are typically colloidal materials that made up from organic (humic acids, protein, carbohydrate), inorganic (clays, silica salt, metal oxides, etc.), biological (virus, bacteria, microbial attachment) in the presence of nutrients on membrane surface [2]. Membrane fouling will result in poor performance of this system and the increase in the operation cost due to energy and time spent on the membrane filtration [3]. The extent of membrane fouling typically depends on four factors, that is membrane material, solution conditions, operating conditions and protein properties [4]. Reversible fouling is a type of temporary membrane fouling which can be removed easily with certain cleaning methods, while irreversible fouling is defined as permanent fouling as foulants still remain after the cleaning [1, 5].

Cleaning of membrane simply means the removal of all non-integral substances (foulants) by chemical, biochemical or physical means. Kim et al. [6] reported that optimisation of the cleaning process requires knowledge of how operating conditions affect the cleaning process and subsequent performance of cleaned membranes as well as the stability of cleaning agents. In general, cleaning may remove the deposited cake layer which in return will improve its permeation flux significantly [7]. Fouled membranes could be cleaned by either physical and chemical or combination of both methods [8]. Physical cleaning method adopted the mechanical forces to detach foulants from the membrane surface. Meanwhile, the effectiveness of chemical cleaning method relied on chemical reaction occurred to deteriorate the strong cohesion forces between the fouling components and the adhesion forces between the foulants and the surface of membrane [9].

Sodium hydroxide (NaOH) is widely used in membrane cleaning. NaOH consists of hydroxyl group (-OH) which is responsible for dissolution of slightly acidic natural organic matters (NOM) and promotes cleavage of polysaccharides and proteins into smaller sugars and amides [10]. Besides, -OH group helps in expanding the NOM molecules and facilitates mass transfer of cleaning agent to reach membrane surface. However, the concentration of NaOH as the cleaning agent has to be controlled at an optimum value in order to attain maximal cleaning efficiency, with considerations of cost and membrane integrity [11].

Sodium hypochlorite (NaOCl) is the most common type of oxidant which used as cleaning agent for organically fouled nanofiltration (NF) membranes. However, NaOCl is not universally adopted due to possible damage to some membranes as not every membrane is chlorine resistant. Besides, generation of chlorinated organics have impacts to the health and environmental [12]. In fact, combination of oxidants and alkaline agents theoretically has better removal on organic foulants than oxidant cleaning agents alone.

This paper investigates the ability of NaOH and NaOCl as the chemical cleaning agents to restore the permeate flux of organically fouled NF membranes under varying applied pressure and flow condition. The performances of cleaning protocols adopted were subsequently evaluated using flux recovery and resistance removal. Furthermore, Attenuated Total Reflectance-Fourier Transform Infrared

(ATR–FTIR) was performed to investigate the deposition of organic foulants on the test membranes and their behaviours after the chemical cleaning. This output of this research is expected to benefit the membrane technology industry by identifying the key parameters that could optimally restore the flux of organically fouled NF membrane.

1.1. Cleaning efficiency quantification

Flux recovery and resistance removal are used to evaluate the effectiveness of different cleaning protocols. The flux recovery and the resistance removal can be estimated using Eq. (1) and (2), respectively [13].

$$\text{Flux recovery} = \left(\frac{J_{wc} - J_{wf}}{J_{wi} - J_{wf}} \right) \times 100\% \quad (1)$$

where J_{wc} is permeate flux after cleaning, J_{wf} is permeate flux after fouling and J_{wi} is permeate flux before fouling.

$$\text{Resistance removal} = \left(\frac{R_f - R_c}{R_f} \right) \times 100\% \quad (2)$$

where R_f is membrane resistance after fouling and R_c is membrane resistance after cleaning. R_f can be calculated from the water flux after filtering with suspension as presented in Eq. (3).

$$R_f = \left(\frac{\Delta P}{\mu \sum J_{wf}} \right) - R_m \quad (3)$$

R_m is membrane resistance which can be estimated from the initial water flux as shown in Eq. (4).

$$R_m = \left(\frac{\Delta P}{\mu \sum J_{wi}} \right) \quad (4)$$

The resistance, which is remained after cleaning, R_c can be calculated from the water flux after chemical cleaning as depicted in Eq. (5).

$$R_c = \left(\frac{\Delta P}{\mu \sum J_{wc}} \right) - R_m \quad (5)$$

where ΔP is transmembrane pressure and μ is viscosity of the fluid.

2. Materials and Methods

The following subsections will describe the methods and materials (cleaning agents) used in recovering water flux of organically fouled membrane. Filtration system setup and protocol of cleaning process will also be described in detail.

2.1. Organically fouled membrane preparation

The organically fouled membranes were prepared by filtering new NF polyethersulfone (PES) membranes with commercial typed Aldrich humic acid (AHA) at the concentration of 10 mg/L. This commercial AHA with the model of 53680 was supplied by Sigma Aldrich. Some important physical and chemical characteristics of this AHA have been mentioned in [14]. Stock solution of AHA (0.2 g/L) was prepared by dissolving the powdered form of AHA in 0.05 M sodium bicarbonate (NaHCO_3) solution. Before dissolving the mixture, NaHCO_3 was adjusted to pH 8.5 with 0.1 M NaOH to enhance its solubility [15]. Foulant suspension was prepared by adding deionized water to the stock solution to obtain the desired concentration of 10 mg/L.

2.2. Filtration setup and experiments

The filtration setup consisted of a nitrogen cylinder tank, a pressure regulator, a self-fabricated feed reservoir, a stirred cell (Sterlitech HP4750), a magnetic stirrer and an electronic balance. The effective membrane area of the stirred cell is 0.00146 m². Nitrogen gas was used to pressurise the cell to the operating pressure. The feed solution was fed continually from the feed reservoir connected to the stirred cell to replenish the permeate. The permeate flux was continuously measured gravimetrically by an AND GF-10K electronic balance.

Filtration tests were conducted via deionised water before and after the fouling process to measure the differences of their pure water fluxes. The water fluxes before fouling and after fouling were labeled as J_{wi} and J_{wf} , respectively. The applied pressure to measure the water flux was fixed at 14 bar. All filtration experiments were conducted at room temperature of about 25 °C at 400 rpm stirring speed and repeated for at least twice to ensure the results were reproducible.

2.3. Membrane cleaning protocol

Prior to the NaOH (pH 12) cleaning, the NF membranes were fouled with 10 mg/L AHA as described earlier. The organically fouled NF membranes were subsequently cleaned by filtrating NaOH solution with the concentration of 0.5, 1.0, 2.0, and 3.0 wt% under the applied pressure of 12, 14, 16, and 18 bar for a duration of 30 min. Under the identical concentration of the cleaning agent and applied pressure, the cleaning cycle was repeated for backwash flow condition to evaluate and compare the cleaning efficiency of fouled membranes between normal and backwash flow conditions.

The water flux, J_{wc} was determined after the chemical cleaning process. NaOH was chosen as the cleaning agent in this study because it was generally known to have rapid cleaning result and overall cleaning effectiveness [16]. To compare the cleaning efficiency with another type of chemical cleaning agent, similar membrane cleaning protocols were adopted for NaOCl (pH 11) with the concentration of 0.2, 0.5, 1.0, and 2.0 wt%.

2.4. Analytical methods

FTIR analyses of samples were carried out using an ATR-FTIR spectroscopy (Nicolet 6700, Thermo Scientific, Waltham, MA) to determine major functional groups of the virgin and chemically cleaned NF membranes. The adsorbed chemical compounds were analysed by the ATR-FTIR in the transmittance mode for a wavenumber ranged from 400 to 4000 cm⁻¹.

3. Results and Discussion

The findings of this work will be divided into several subsections and discussed according to effect of NaOH concentration, effect of operating pressure, effect of filtration direction and effect of type of cleaning agent.

3.1. Effect of NaOH concentration

The effect of NaOH concentration on the cleaning efficiency of the organically fouled NF membranes under normal and backwash flow conditions is presented in Fig. 1. The percentages of flux recovery and resistance removal were consistent with the concentration of NaOH in which they increased with the increase in NaOH concentration. Higher flux recovery and resistance removal were observed when the fouled NF membranes were cleaned with higher concentration of NaOH. Increasing the NaOH concentration means increasing the capacity of the cleaning agent to loosen and dissolve the foulant from the fouling layer. At high NaOH concentration, the NaOH could easily dissolve the organic foulant, which are deposited on the membrane surface as a cake or a gel. In addition, the cleaning chemical concentration can affect both the equilibrium and the rate of the reaction.

The cleaning chemical concentration plays a key role not only to maintain a reasonable reaction rate but also to overcome mass transfer barriers imposed by the fouling layer. In practice, the cleaning chemical concentration is usually high enough to ensure a desirable reaction rate. Typically, NaOH cleans organically fouled membranes by hydrolysis and solubilisation. Solubility of the organic foulant increases by few orders of magnitude when carboxylic functional groups of the organic foulant are completely deprotonated at basic conditions [17]. Consequently, higher concentration had resulted in a much better removal of the foulant. Similar trend has been observed by other researchers [13, 18, 19]. On the other hand, this effect is insignificant at a lower concentration of NaOH (0.5 wt%). This is due to limited ability of cake removal by the cleaning agent.

On the contrary, the flux recovery and resistance removal of membranes under backwash flow condition were consistently increased with increasing NaOH concentration. The backwashing method is a reversed filtration process in which the chemical cleaning agent is fed from the permeate side to the filtration membrane. With the water flushing from the permeate side to feed side, it will expand the fouling layer accumulated on the surface of the filtration membrane, and then the foulants will be fluidised and been removed [20].

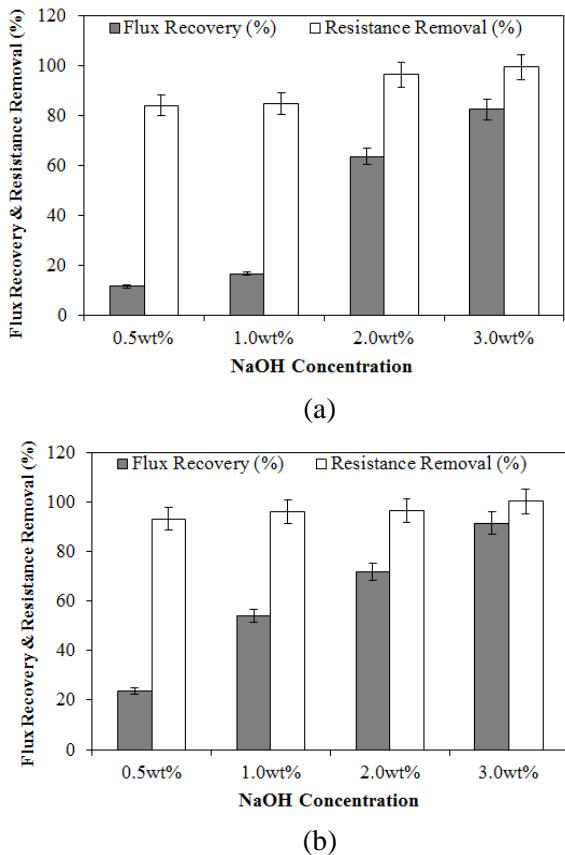


Fig. 1. Effect of NaOH concentration on cleaning efficiency of the organically fouled NF membranes under different flow conditions, (a) normal and (b) backwash.

3.2. Effect of applied pressure

The effect of applied pressure on the cleaning efficiency of the organically fouled NF membranes with different applied pressures ranging from 12-18 bar is presented in Fig. 2. Higher flux recovery and resistance removal were achieved with the increase in applied pressure. This is because higher applied pressure will potentially increase in the hydrodynamics of mass transfer from the fouling layer to the bulk solution [13]. This phenomenon will promote the collision of particles and the breakdown of particles into smaller sizes, which eventually lead to their easy removal [13].

Furthermore, the rate of increment for the flux recovery and resistance removal of the fouled NF membranes which were cleaned with NaOH at different applied pressures under backwash flow condition at higher applied pressure range (16 to 18 bar) were found insignificant compared to the lower applied pressure range (i.e. 12 to 14 bar). The flux recovery was found increased from 35% to 63% at the applied pressure of 12 to 14 bar. On the contrary, the flux recovery for the applied pressures of 14 to 18 bar has experienced a marginal increase (from 63%

to 74%). Similar trend was observed for resistance removal. The resistance removal was found hovering around 98-99% at the applied pressures of 14-18 bar. It seemed to have a threshold applied pressure between 14-18 bar. Beyond this threshold pressure, the improvements in resistance removal were found to be of marginal significance. This is due to the nearly completed removal of the reversible deposits by NaOH at the stipulated pressure ranges. In this case, the increment of the applied pressure is less beneficial.

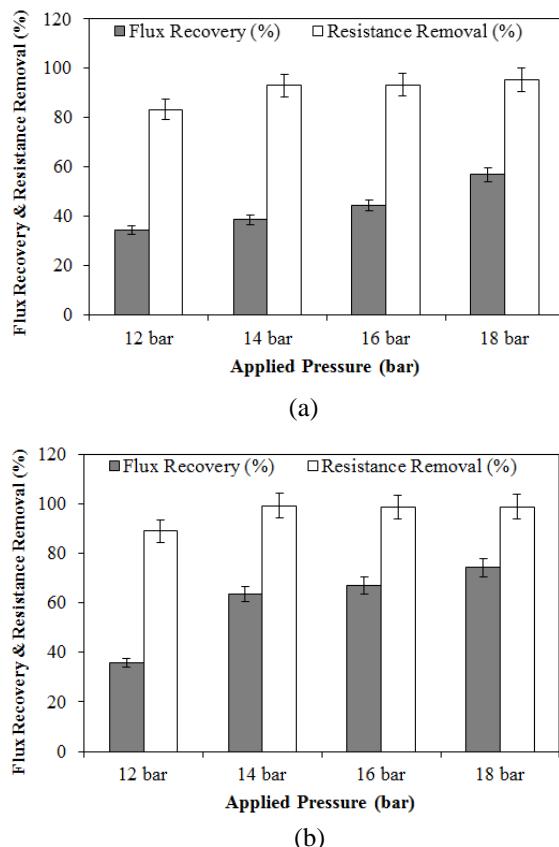


Fig. 2. Effect of applied pressure on cleaning efficiency of organically fouled NF membranes under different flow conditions, (a) normal and (b) backwash.

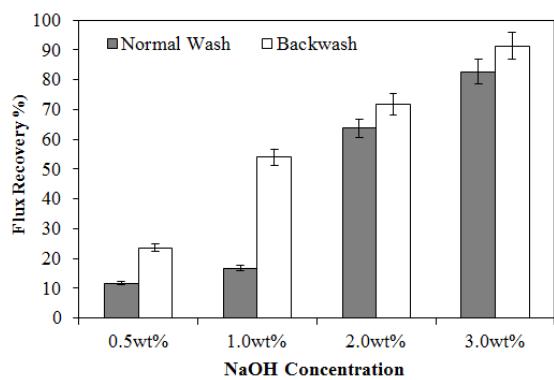
When comparing the flux recovery and resistance removal obtained, it was found that the percentage of flux recovery was relatively lesser than the percentage of resistance removal for most of the conditions studied. It is expected that irreversible membrane fouling has been occurred in this study. Kimura et al. [21] demonstrated that organic matter played an important role in the development of the irreversible fouling in their study. Their results reported that consecutive use of two (or more) different agents in chemical cleaning of membrane was expected to be more effective in restoring pure water flux than cleaning with a single cleaning agent. In our study, the use of single agent in

chemical cleaning could be the reason why completely restoration of flux could not be achieved for organic fouling.

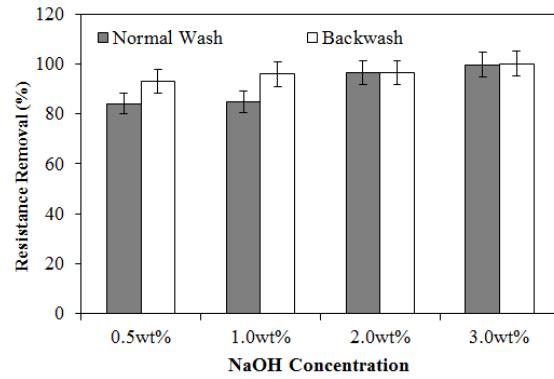
3.3. Effect of filtration direction subject to variation in NaOH concentration

The effect of filtration direction subject to variation in NaOH concentration is presented in Fig. 3. It was found that, within the concentration range investigated, the flux recovery and resistance removal for the backwash flow condition always higher than that of the normal flow condition. The flux recovery at backwash flow condition reached a pseudo-maximum around 91% for 3.0wt% NaOH as compared to the normal flow condition (i.e. 82%).

The flux recovery at normal and backwash flow conditions lied in a wider range of 11-82% and 23-91%, respectively. Meanwhile, the resistance removal obtained from normal and backwash flow conditions hovering around a narrower range of 84-99% and 93-100%, respectively. Further discussion on backwash mechanism that facilitates the cleaning efficiency will be explained in Section 3.4.



(a)



(b)

Fig. 3. Effect of filtration direction subject to variation in NaOH concentration, (a) flux recovery and (b) resistance removal.

3.4. Effect of filtration direction subject to variation in applied pressure

Figure 4 compares the cleaning efficiency of NaOH between the normal and backwash flow conditions at different applied pressures. The flux recovery and resistance removal for backwash flow condition were found to be always higher than that of the normal flow condition. This can be explained by the effect of backwash flow condition would have probably lead to their easy removal of foulants deposited on the membrane surface. When backwash flow condition is applied, the pores of membranes are flushed inside out. The pressure on the permeate side of the membrane is higher than the pressure within the membranes, causing the pores to be cleaned easily [22]. This phenomenon could be claimed as backwash process in microfiltration and ultrafiltration membranes. Zhang and Ma [23] has reported that reversible fouling, caused by a gel layer resulted from reversible concentration polarisation, can be removed by means of strong shear force of backwashing. This cleaning process is commonly denoted as chemically enhanced backwashes. The action of reversing the flow will create turbulence during backwash and eventually will contribute to the disruption of the fouling layer on the surface of membrane [8, 24].

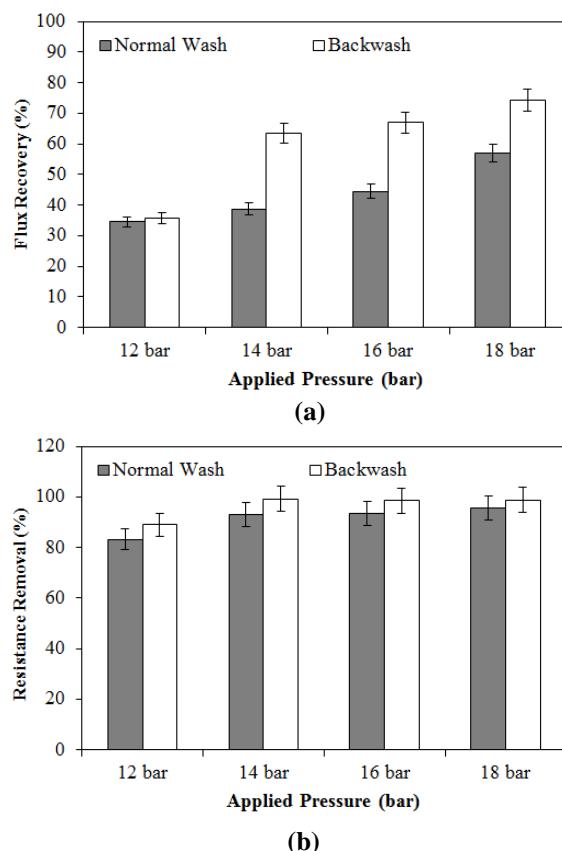
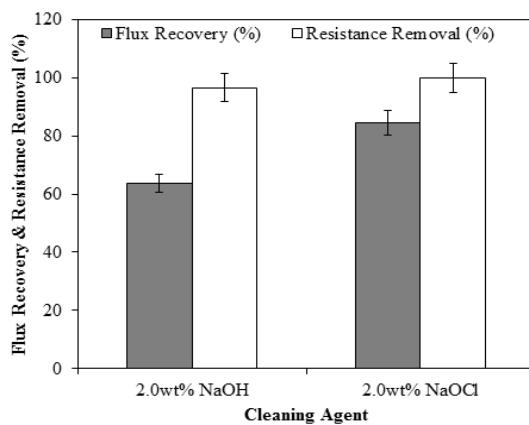


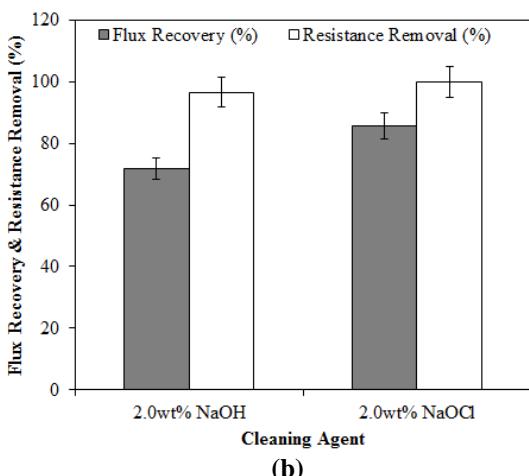
Fig. 4. Effect of filtration direction subject to variation in applied pressure,
(a) flux recovery and (b) resistance removal.

3.5. Effect of cleaning agent type

Figure 5 shows the cleaning efficiency of the organically fouled NF membranes cleaned with NaOH and NaOCl (2.0wt% each) under flow conditions of normal and backwash. NaOCl showed higher cleaning efficiency of 84.5% compared to 63.7% for NaOH. NaOCl is an oxidising agent which is able to remove organic foulants through oxidation process [25]. Foulants deposited on the membrane surfaces were easily detached from the surface of membrane by the oxidative environment of NaOCl. This finding was supported by Yoon [26] who found that using NaOCl as the cleaning agent before acids is more effective in removing organic foulant. Exposure of the fouled membrane with NaOCl also tends to decrease the contact angle of PES membrane due to gradually increase of its relative hydrophilicity after oxidative chemical cleaning [27].



(a)



(b)

Fig. 5. Effect of cleaning agent type on cleaning efficiency of the organically fouled NF membranes under different flow conditions, (a) normal wash and (b) backwash.

3.6. ATR-FTIR spectroscopy

Transmittances of ATR-FTIR of the organically fouled NF membranes and the chemically cleaned NF membranes were compared to analyse the changes in the functional groups of the membranes resulting from the membrane interactions with the cleaning agents and the organic foulant. Fig. 6 presents the ATR-FTIR spectra of the virgin NF membrane, fouled NF membranes, and chemically cleaned NF membranes with NaOH and NaOCl (2.0wt% each) separately under backwash flow condition. The peak bands at 1580 and 1650 cm^{-1} signify aromatic bands characteristic of the PES NF membrane. In comparison with the FTIR spectra of virgin and the fouled NF membranes, a significant transmittance was observed after the exposure to the AHA. For instances, the intensity of the peak at 1646 cm^{-1} , attributed to C=O stretching (carbonyl group), was lower in the organically fouled NF membrane. The absorption peaks such as a broad absorption peak at 3396 cm^{-1} were identified as -OH stretching and the distinct peak at 685 cm^{-1} were denoted as C-H bending [28]. The decrease in transmittance intensity implied that the virgin membrane has been coated with organic foulants [29].

The ATR-FTIR spectra of the fouled NF membranes and NF membranes cleaned with NaOCl revealed that the carbonyl peak caused by the organic foulants mentioned earlier (1646 cm^{-1}) have shifted to higher wavenumber (1740 cm^{-1}) attributed to oxidative degradation of organic foulants by the cleaning agent of NaOCl [25]. Owing to NaOCl is an oxidant, the oxidation of organic compounds would generate more oxygen containing functional groups such as aldehyde, ketone, and carboxylic acids once the organically fouled membrane is exposed to NaOCl solution [30]. In addition, the degraded functional groups would be more susceptible to hydrolysis at high pH level of 11. The presence of these functional groups generally improves hydrophilicity of their parent compounds. As a result, the oxidation would help to reduce the adhesion of foulants to the membranes. Generally, this oxidation degradation happened when oxygen is absorbed at certain parts on the organic compounds. With this oxygen absorption, an increase in oxygen containing groups such as carbonyls assigned to C=O stretch at 1740 cm^{-1} could be observed [31].

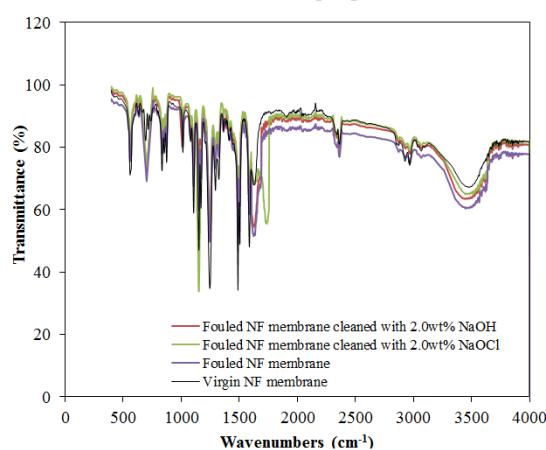


Fig. 6. ATR-FTIR spectra comparison of virgin NF membrane, fouled NF membranes, chemically cleaned NF membranes with NaOH and NaOCl under backwash flow condition.

The spectra of membrane cleaned with 2.0 wt% NaOH/NaOCl are also presented in Fig. 6. Obviously, the spectra of cleaned membrane showed almost identical patterns as the fouled membrane except the increase in their transmittance intensity due to the removal of foulants on the membrane surface by NaOH/NaOCl. The observed of identical patterns in spectra signifies that the alteration in chemical composition of membrane is insignificant when it is cleaned using NaOH/NaOCl. On the other hand, the higher transmittance intensity detected for the membrane cleaned with NaOH is also in line with the flux recovery obtained from experiment as it was found to be quite effective (FR=71.69%) in removing organic foulants.

4. Conclusions

A series of experimental runs were carried out to investigate the cleaning efficiency of the organically fouled NF membranes by different NaOH and NaOCl concentrations at different applied pressures under normal and backwash flow conditions. The performance of the cleaning protocols was quantified using flux recovery and resistance removal. The following findings are drawn from this study:

- It was found that higher flux recovery and resistance removal were observed when the fouled NF membranes were cleaned with higher concentration of NaOH. This is because higher concentration of NaOH could easily dissolved the organic foulant, which are deposited on the membrane surface as a cake or a gel.
- Higher flux recovery and resistance removal were achieved with the increase in applied pressure. Higher applied pressure tends to enhance the collision of particles breakdown into much smaller sizes.
- The flux recovery and resistance removal for the backwash flow condition always higher than that of the normal flow condition due to the action of reversing the flow could promote a strong shear force of backwashing.
- FTIR analysis of samples demonstrated that NaOCl was more effective than NaOH in removing organic foulants based on the intensity of transmittance measured. The T% of NaOCl was found consistently greater than that of NaOH, particularly obvious at the intensity of the three peaks of the fouled membrane were found increased after cleaning with NaOCl.

These findings have vital implications for identification of the key parameters that could restore the flux of organically fouled NF membranes significantly. In conclusion, this research plays an important role for the design of chemical cleaning protocols for the PES NF membranes used in surface water treatment.

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