



Case Study of a Small Scale Reverse Osmosis System for Treatment of Mixed Brackish Water and STP Effluent

I Nyoman Widiasta* & Retno Dwi Jayanti

Department of Chemical Engineering, Diponegoro University, Jl. Prof Sudarto-Tembalang, Semarang 50239, Indonesia

*E-mail: widiasta@undip.ac.id

Abstract. A case study on utilizing reverse osmosis (RO) technology to fulfill fresh water needs at a mall and a hotel has been done on Bali Island, Indonesia. A mix of brackish water and sewage treatment plant (STP) effluent was used as feed water in the RO system. The system used 36 membrane elements (CSM RE 8040 BLN) arranged into two stages: 8 pressure vessels (PVs) in the first stage and 4 PVs in the second stage, each loaded with 3 membranes. The objectives of this research were to assess the cleaning effectivity in the plant, to evaluate the cleaning of 1 membrane element using a CIP system, and to assess the use of the membrane for filtration in the pre-treatment system. SEM and FTIR analysis indicated that the foulants on the membrane surface were dominated by organic foulants and inorganic deposits. To clean the discarded membrane the proposed method used NaOH solution (pH 12 and pH 13) and citric acid (pH 2 and pH 3). All membranes displayed a dramatic decline in rejection of about 80%. Based on the rejection tests of SO_4^{2-} , Cl^- , turbidity reduction approached 100%. It can be concluded that an RO membrane that has undergone selectivity decline can be re-used as a filtration membrane in the pre-treatment system.

Keywords: domestic wastewater; chemical cleaning; ion rejection; reuse; RO membrane.

1 Introduction

Reverse osmosis (RO) technology is used to reclaim domestic wastewater. Fouling and scaling are still the major problems in RO application since they can cause membrane performance degradation and flux decline, and influence the product water quality [1-3]. Fouling and scaling depends on several factors, i.e. membrane morphology, feed water composition, and operation conditions [4-8]. Pre-treatment can be done in RO application to control membrane fouling and scaling by removing impurities from the feed water [3,9,10]. Unfortunately, pre-treatment is not capable to perfectly remove all impurities from feed water and even causes fouling of the membrane [9,11]. Chemical membrane cleaning is an alternative way to control fouling on the membrane [12]. This can be done when the membrane fouling and scaling causes the flux decline to approach 5-10% [13-15]. In RO application, chemical membrane cleaning is more often

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selected to control membrane fouling and scaling than for pre-treatment. This is because chemical cleaning is considered effective to control membrane fouling and scaling in large RO applications.

Previous researches reported that chemical membrane cleaning can already provide almost 100% recovery of membrane flux so it is able to extend membrane lifespan [1,16-21]. The cleaning agents that are usually used are ethylene diamine tetraacetic acid (EDTA) [17,21], alkali solutions like sodium hydroxide (NaOH) [16,18], acid solutions like hydrogen chloride (HCl) [18] and citric acid [16]. Ang, *et al.* [21] used 2 mM (pH 11) of EDTA solution and 10 mM (pH 10) of SDS solution at 40°C for 60 minutes to clean an RO membrane for domestic wastewater reclamation. The results indicated that recovery of the permeate flow rate reached 100%. The rejection recovery was not measured. Ang, *et al.* [17] used EDTA solution for 60 minutes without considering the effect of temperature on RO membrane cleaning. The permeate flow rate recovery reached only 91%.

RO membrane cleaning for domestic wastewater application that combines an acid and alkali solution was done by Li, *et al.* [18]. This was conducted using HCl (pH 2.5) for 1 hour, then continuing with NaOH (pH 11.5) for 1 hour, and finishing with acidic cleaning by HCl solution at the same operation condition. This recovered about 95% of the permeate flow rate and 98% of membrane rejection after cleaning. Ochando-Pulido, *et al.* [16] cleaned an RO membrane using citric acid at the first stage and NaOH-SDS in 30-35°C for 25 minutes at the second stage. The recovery of the permeate flow rate was 85.1% after cleaning. The pH of the cleaning solution was not considered. Moreover, SEM characterization showed types of organic foulants, iron colloid ($\text{Fe}(\text{OH})_3$), CaCO_3 , CaCl_2 , and CaSO_4 scale.

Membrane rejection is important to be measured to know the cleaning efficiency apart from the permeate flow rate recovery. This process retrieves fresh water that is the desired product from RO application, so not only the quantity but also the quality of the product is an important parameter from this application. The cleaning solution's pH is another parameter that should be given attention in view of the chemical stability of the membrane towards cleaning solution pH. In this research, the influence of cleaning solution pH on permeate flow rate recovery and rejection in the water treatment plant (WTP) system of effluent/brackish water was observed. The combination of citric acid and NaOH solution at outside guidance pH is a novelty of this research compared to previous researches.

A membrane element from CSM (RE-8040-BLN) was used in this research. Periodic cleaning cannot fully restore rejection or recover flow rate, thus

decreasing the membrane's lifespan, and it also cannot be used for the same application. Therefore, the objectives of this research were to evaluate the chemical cleaning of a membrane element using a cleaning in place (CIP) system, to characterize membrane fouling and scaling, and to analyze the influence of membrane cleaning on flow rate recovery and rejection conducted at extreme pH.

2 Process Design

This research used the membrane element of an effluent/brackish water WTP system. The plant system design is shown in Figure 1. The RO system consisted of RO feed tank, feed pump, cartridge filter, high pressure pump, 8 pressure vessels (PVs) at the first stage and 4 PVs at the second stage. Each PV was loaded with 3 membranes.

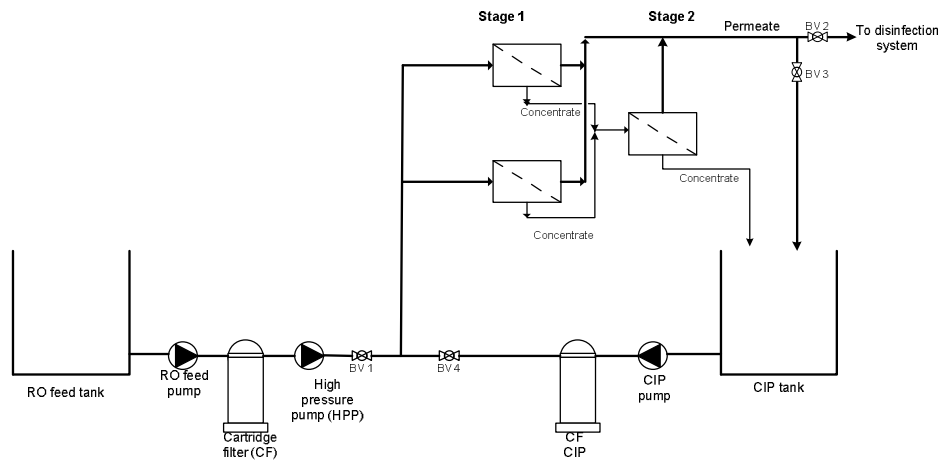


Figure 1 RO system design.

Cleaning was conducted by the CIP system using NaOH+EDTA and citric acid+HCl once a month. Both permeate flow rate and pressure drop were used to measure the cleaning efficiency. Therefore, this research also analyzed the normalized permeate flow rate and pressure drop in the system before and after cleaning. The normalized permeate flow rate was measured based on Eq. (1):

$$\text{normalized product flow} = \frac{[(AAP_s - \Delta\Pi_s)]TCF_s}{[(AAP_a - \Delta\Pi_a)]TCF_a} \times \text{actual flow} \quad (1)$$

AAP = average applied trans membrane pressure

$$AAP = \frac{P_{\text{feed}} + P_{\text{reject}}}{2} - P_{\text{permeate}}$$

P_{feed} = feed pressure

P_{reject} = concentrate pressure

P_{permeate} = permeate pressure

$\Delta\Pi$ = difference between the osmotic pressure on the membrane feed and permeate side

TCF = temperature correction factor

's' = standard condition (startup condition)

'a' = actual condition

3 Material and Methods

3.1 Materials

3.1.1 Membrane Element

All membrane elements (CSM RO Membrane) were obtained from the mall and hotel reclamation plant in Kuta, Bali, Indonesia after 1 year of operation. They were CSM RO Membranes. During operation these membranes were cleaned once a month using EDTA+NaOH at the first cleaning and citric acid+HCl at the second cleaning.

3.1.2 Chemicals

All chemicals used were NaOH (technical grade) in flake form and citric acid (food grade) as cleaning agent. MgSO_4 (technical grade) and CaCl_2 (technical grade) were used as filtration test solution. All chemicals were obtained from local producers in Indonesia.

3.2 Methods

3.2.1 Procedures

This research was conducted using a cleaning in place (CIP) set, shown in Figure 2. The experimental set consisted of a spiral wound membrane element, pump, CIP tank, pressure indicator, and pressure indicator valve. The cleaning procedures were divided into 3 steps as described below.

1. Flow rate measurement before cleaning

The procedure before cleaning consisted of measuring the flow rate of the water at 8 bar and room temperature at which the measurement was done after steady state was reached.

2. Cleaning procedure

Cleaning was conducted by circulating the NaOH solution (pH 13) through the membrane element at atmospheric pressure and room temperature for 60 minutes. After 60 minutes, the pumps were turned off and the membrane was kept submersed for 60 minutes. The pump was turned on again and NaOH solution was circulated for 60 minutes. After the membrane element was rinsed using aquadest, the cleaning process was continued using citric acid solution (pH 2) at the same operational conditions as for alkaline cleaning.

3. Flow rate measurement after cleaning

The flow rate of the water was measured after cleaning at 8 bar and room temperature. Membrane rejection of feedwater and product water both before and after cleaning was obtained by measuring total dissolved solid (TDS) using a TDS meter (hold) TDS-3 HM Digital. Flow rate measurement was done by measuring the time required to obtain 2 L of product.

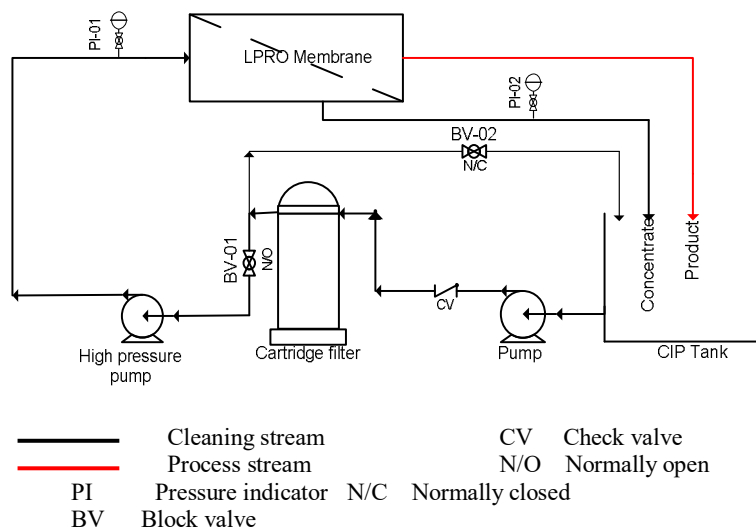


Figure 2 Experimental set-up.

4. Foulant characterization

A membrane element was autopsied and cut into pieces of 3 x 4 cm. The functional groups of foulants on the membrane surface were characterized using an IR Prestige 21 (Shimadzu, Japan) while the deposited substances on the membrane surface were analyzed using an SEM-EDX JSM-6510LA (Jeol, Japan).

3.2.2 Ion Rejection

Magnesium sulphate solution (2000 ppm) and CaCl_2 solution (500) ppm were used for rejection testing as divalent anion and monovalent anion respectively. Each solution test was circulated through the membrane element at 8 bar for 60 minutes. After circulation both feed and product sample were collected for analysis. The SO_4^{2-} and Cl^- ions were analyzed by Water Multichecker Lamotte Smart 3 Colorimeter.

4 Results and Discussion

4.1 Start up Data

The WTP system of effluent STP/brackish water was operated using 36 membrane elements (CSM RE-8040-BLN), arranged into two stages, 8 PV in the first stage and 4 PV in the second stage, which were loaded with 3 membrane elements in each PV. Startup analysis was conducted to ensure that the system had already operated well based on the set-up design. The data were also used as initial data when the system started operation. The system was observed at 7 pm and 8 am. The data included inlet pressure, concentrate pressure, product flow rate, TDS of product and feed water, concentrate flow rate, and recirculation flow rate. The system was fed with feed water at 1450 ppm of TDS. The feed pressures were conditioned at 6.6 bar at the first stage and 5.8 bar at the second stage, resulting in 80 ppm of TDS and 26 m³/h of product flow rate (Table 1). The system was designed to produce the permeate at 25-30 m³/h of flow rate, so it can be concluded that the product flow rate at the startup measurements was still within the range based on the design.

Table 1 Startup data.

Observation	P inlet stage 1 (bar)	P inlet stage 2 (bar)	ΔP	P concentra-te (bar)	Product flow (m ³ /h)	TDS of product (ppm)	Feed TDS (ppm)	Concent-rate flow (Lpm)	Recircula-tion flow (Lpm)
Sat, May 3 rd 2014 (07.00 pm)	6.6	5.8	0.8	5.0	26	80	1450	140	200
Sun, May 4 th 2014 (08.00 am)	6.4	5.9	0.9	5.0	27	80	1450	145	200

4.2 Long-term Performance

Figure 3 shows the normalized product flow rate for 2 years of operation. Cleaning was conducted using NaOH+EDTA and citric acid+HCl. Intensive membrane cleaning was conducted once a month, aimed at restoring the

membrane flow rate decreased by fouling or scaling. Cleaning in the first month of operation was conducted when 85% of the permeate flow rate from startup condition remained. Permeate flow rate recovery after cleaning was about 60–100%. Membrane replacement was conducted on September 2015 because of inadequate permeate rejection. Therefore, the permeate flow rate that resulted in the second year of operation was lower than during the first year of operation. The permeate flow rate is a result of the system pressure. Figure 4 shows the pressure drop in the system before and after cleaning during 2 years of operation. The cleaning guidance from membrane manufacturer CSM [9] recommends that cleaning should be done when the pressure drop reaches 15% from the initial value, i.e. 0.8 bar. Based on Figure 4, after membrane replacement, the pressure drop was lower than the initial value. An increment in pressure drop after cleaning was shown on September 2015 and May 2016. This may be caused by irreversible fouling on the membrane surface.

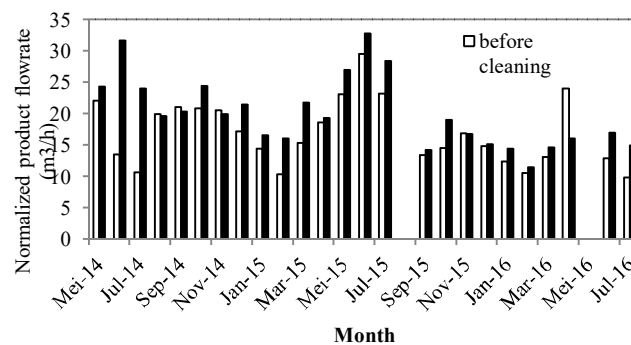


Figure 3 Normalized product flow rate for 2 years operation.

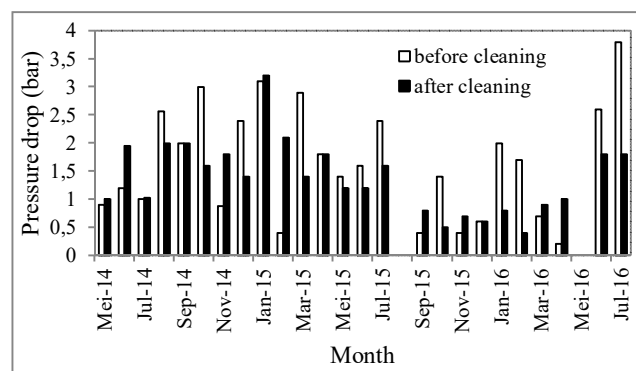


Figure 4 Pressure drop during 2 years of operation.

4.3 Membrane Fouling and Autopsy

FTIR and SEM-EDX analysis were used to find the foulant components on the membrane surface in view of finding a suitable chemical agent to remove them. The FTIR analysis showed numerous sharp peaks around $600\text{--}700\text{ cm}^{-1}$, suggesting that the membrane contained a variety of functional groups, such as carbonyl, amide, and carboxylate [22]. The broad band around $3300\text{--}3400\text{ cm}^{-1}$ indicates free O-H or N-H hydrogen bonds. The functional groups around 3300 cm^{-1} were probably N-H stretching vibration and carboxyl group ($-\text{COOH}$) of polyamide (Figure 5) [23]. Melián-Martel, *et al.* [24] state that hydroxyl absorption at above 3000 cm^{-1} are hydroxyl groups of polysaccharide. The band at 609 cm^{-1} indicates that sulphates were contained in the deposit [23]. The active layer of the membrane was shown at 1656 cm^{-1} , indicating amide I group ($\text{C}=\text{O}$) [25], while the amide II group (N-H) that should be visible at 1540 cm^{-1} did not appear in the FTIR. Another band that could be characteristic of the polyamide group is $\text{C}=\text{C}$ ring vibration at 1490 cm^{-1} [25].

The foulant components on the membrane surface were identified by SEM-EDX analysis (Figure 6). The foulant components were: C 60.66%, O 18.00%, Na 4.97%, Mg 0.42%, Al 1.73%, Si 3.83%, S 6.43%, Cl 2.39%, Ca 0.21%, and Fe 1.37%. The high C and O concentrations on the membrane surface were probably part of organic foulants and/or microbes [24]. Based on the FTIR and SEM results, the fouling layer on the membrane surface was formed by the accumulation of organic and inorganic compounds.

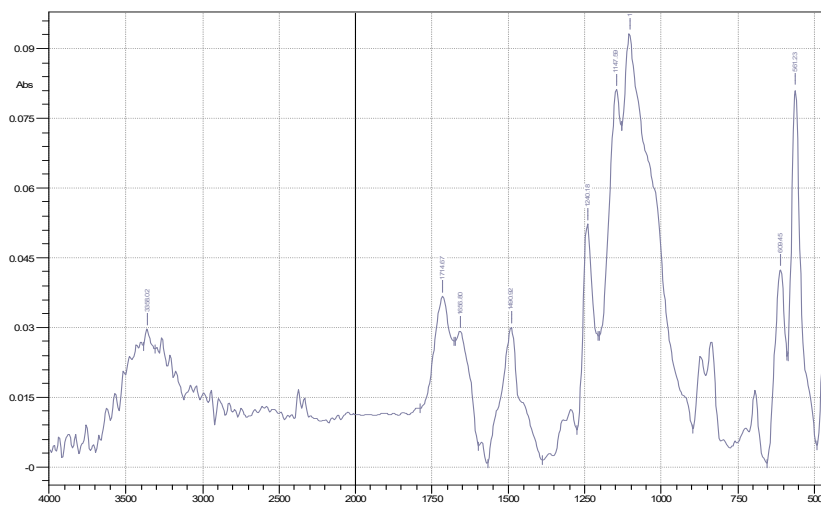


Figure 5 IR spectra of fouled membrane.

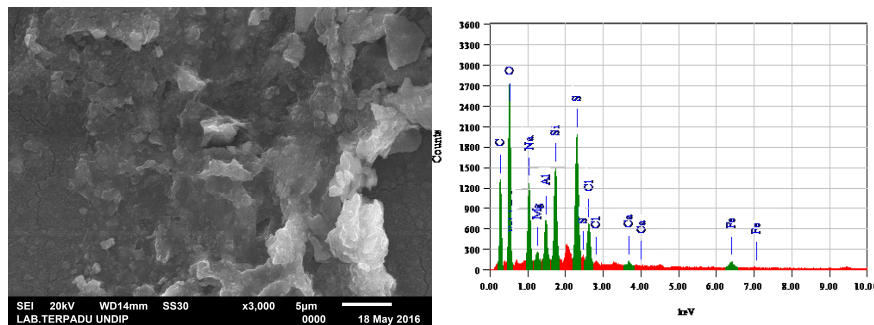


Figure 6 SEM analysis result of fouled membrane (left), foulant composition obtained by Electron Dispersive X Ray (EDX) (right).

4.4 Membrane Rejection and Flow Rate

The cleaning agent that was used in this research had to be able to dissolve the organic foulants on the membrane surface. A number of previous researches have reported that the presence of organic foulants on the membrane surface affects the flow rate due to the chemical composition of the feedwater and the strength of the foulant-foulant interaction [20]. Knowledge of the organic fouling mechanism on the membrane surface can be used as a basis to choose the cleaning procedure. The fouling of a membrane during filtration of a solution containing organic compounds occurs primarily due to adsorption of the organic deposits on the membrane surface. Initially, the organic deposits on the membrane surface reduce the available area for membrane filtration. The initial deposit leads to an increase in the hydrophobicity of the membrane surface.

A fouling monolayer is quickly formed on the membrane surface. Subsequent accumulation of organics on the membrane surface due to convective deposition leads to a fouling layer that makes the hydrophobicity of the membrane surface increase further [26,27]. The fouling layer on the membrane surface can also cause an increase in hydraulic resistance [27] and flow rate decline [20]. The occurrence of fouling on all membranes in this research was attributed to the low flow rate, at an average of 0.4 m³/h, and membrane rejection at an average of 70-80% (Figures 7 to 9).

Cleaning effectivity is affected by 2 mechanisms: the chemical reaction between the cleaning agent and the foulants on the foulant layer and the mass transfer from the cleaning agent in the bulk phase into the fouling layer and the mass transfer of foulants from the fouling layer into the bulk phase [21]. Cleaning in this research was conducted by circulating cleaning agent through the membrane, allowing the concentrate stream to flow. To know the optimum

condition based on the type and the pH of the cleaning solution, this research observed the effect of cleaning agent combination on rejection and flow rate. The cleaning agents used were NaOH (pH 13), NaOH (pH 12), citric acid (pH 2), and citric acid (pH 3). Table 2 shows cleaning methods used in this research.

Table 2 Membrane cleaning methods.

Methods	Alkaline pH	Acid pH
Alkaline – acid – alkaline	13	2
Acid – alkaline – acid	12	3
Alkaline – acid – alkaline	12	3

Membrane cleaning was conducted with 3 levels of cleaning (alkaline, acid, alkaline). The cleaning guidance from the membrane manufacturer suggests that if the color of the cleaning agent changes, then cleaning should be conducted again, using the same solution [9]. This can be seen in the Figure 7, in which both alkaline and acid cleaning were conducted twice. The membrane had rejection decline after the first and second alkaline cleaning, which then increased after acid cleaning and decreased after the third alkaline cleaning. Sodium hydroxide is a cleaning agent that dissolves foulants by hydrolysis and solubilization.

Sodium hydroxide is a strong base solution that can help remove particles that are deposited on the membrane surface, primarily organic matters and microbes attached to the active layer of the membrane [16]. The strong base of NaOH can serve as hydrolysis, saponification, and solubilization agent of organic foulants [28]. The presence of hydroxyl ions enhances the disaggregation of the fouling layer by increasing the pH and the ionic strength as well as the solubility of the organic compounds [19].

The increment in the pH value by the addition of the caustic cleaning reagent (NaOH) increases the electronegativity of the organic foulants deposited on the membrane surface due to the deprotonation of the carboxylic and the phenolic functional groups of organic pollutants [4]. It causes an increment of the deposit repulsion force on the membrane, weakens the adhesion force between the membrane and the foulants, and foulant removal. The fouling layer becomes denser, enabling penetration of the cleaning reagent, and can enhance the mass transfer, therefore leading to an increase in cleaning effectivity (flow rate) of the membrane [4]. This mechanism can be observed through the flow rate increment after alkaline cleaning (Figure 7).

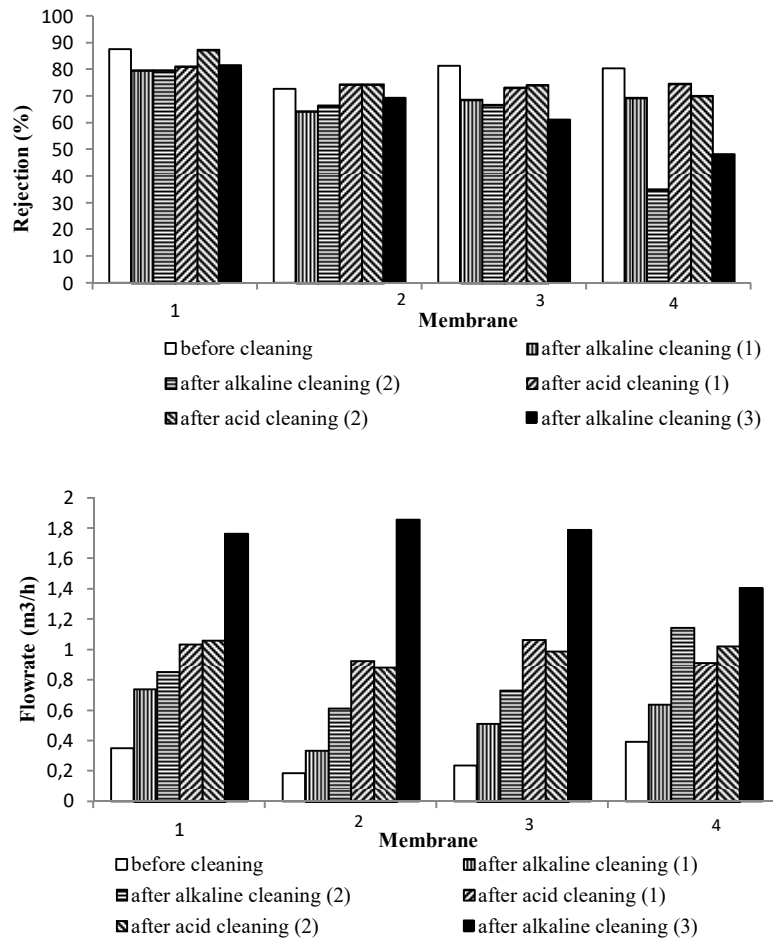


Figure 7 Membrane rejection and flow rate after alkaline cleaning pH 13 and acid cleaning pH 2 (operation conditions: atmospheric pressure and room temperature).

In this research the effect of cleaning stage variation was observed because it also has an important influence on cleaning effectivity. Cleaning was conducted in 2 series, i.e. acid cleaning (citric acid pH 2) in the first series, without cleaning reagent replacement; alkaline cleaning (NaOH pH 13) in the second series; followed by acid cleaning (citric acid pH 2). This variation showed a change in rejection and cleaning effectivity (Figure 8). Citric acid can hydrolyze some organic matter and also be very effective in cleaning colloidal iron ($\text{Fe}(\text{OH})_3$) and carbonate precipitates on the membrane surface given that it is able not only to dissolve and remove them, but also lead to the complexation of

the iron ions by forming a bidentate mononuclear complex more easily removable from the membrane surface [19].

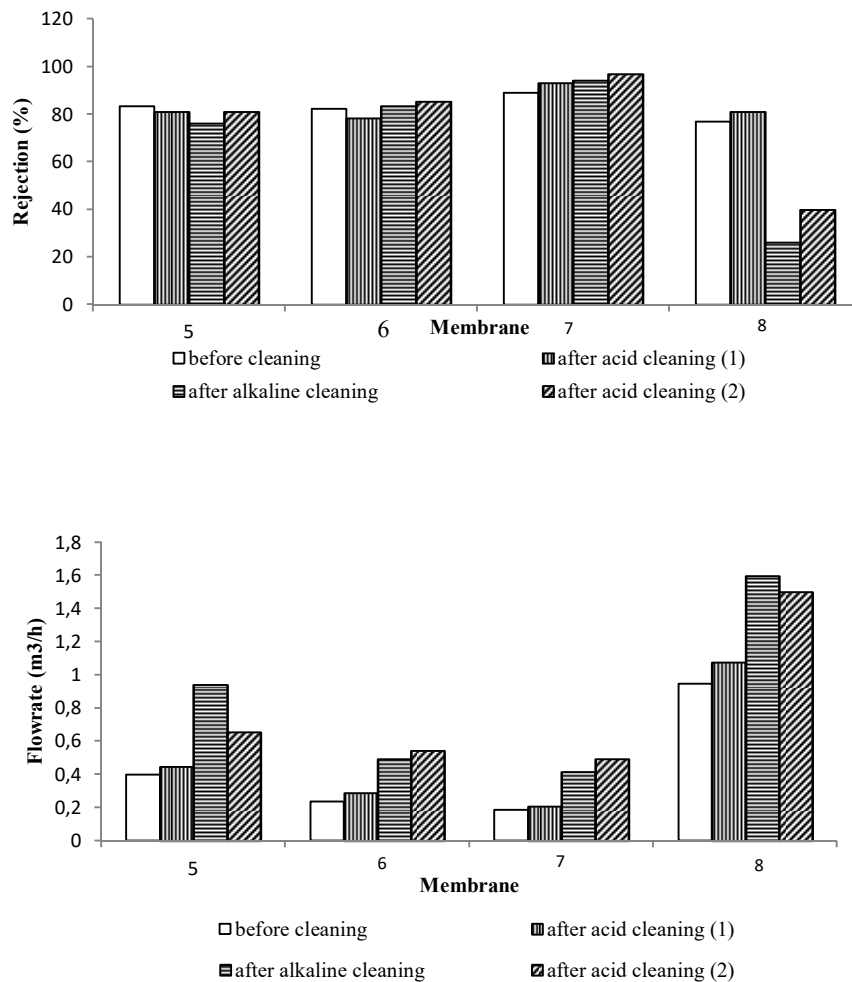


Figure 8 Membrane rejection and flow rate after acid cleaning pH 3 and alkaline cleaning pH 12 (operation conditions: atmospheric pressure and room temperature).

Figure 9 shows the membrane rejection change and the cleaning effectivity after alkaline cleaning using NaOH pH 12. All membranes displayed a decline in rejection after cleaning. Another factor that can explain this rejection decline is

caustic exposure at some cross-linked polyamides, resulting from the dissolution of oligomeric materials that fill or plug the larger pores of these. Once the material is removed from these large voids in the PA film, there can be a great increase in salt passage. The membrane selectivity decline may also be caused by caustic reagents, which leads to swelling on the polyamide membrane [28]. It can be concluded that to enhance cleaning effectivity as well as flow rate, a caustic reagent can be used, but only for hazard fouling.

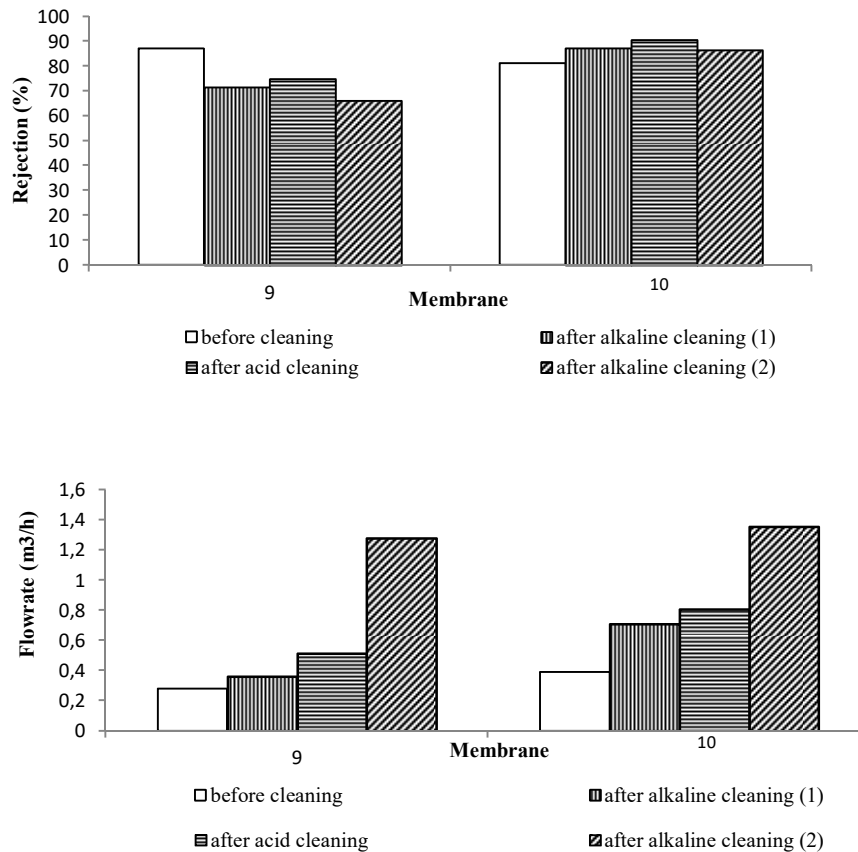


Figure 9 Membrane rejection and flow rate after alkaline cleaning pH 12 and acid cleaning pH 3 (operation conditions: atmospheric pressure and room temperature).

4.5 Monovalent and Divalent Ion Rejection

Membrane cleaning can lead to membrane selectivity decline. It was observed that all membranes had rejection decline but the membrane permeabilities were increased. As a result, the membrane cannot be used for the same application. Several studies have observed that the RO membrane properties had changed and then used them as filtration membrane in the pre-treatment system [29-31]. To know the membrane performance after cleaning, rejection tests were done using salt solution containing monovalent and divalent cations as well as monovalent and divalent anions. Magnesium sulphate and calcium chloride solutions were used in this research. The capability of a membrane in rejecting monovalent anions is shown by the rejection value of Cl^- ions while the capability of a membrane in rejecting divalent anions is shown by the rejection value of SO_4^{2-} ions. Table 3 compares the total rejection and the component rejection. Based on the Table 3, the membranes were more effective for rejecting divalent ions than monovalent ions. This is with regard to molecular size and membrane charge.

Table 3 Comparative Value of Total Rejection and Component Rejection

Test solution	Total rejection (%)	Cl^- rejection (%)	SO_4^{2-} rejection (%)
MgSO_4	69.61	-	67.83
CaCl_2	72.5	58.61	-

The polyamide is an amphoteric and hydrophilic polymer that possesses dissociable carboxylic groups [32]. Exposure of caustic reagent leads to a negative charge on the membrane surface. Chloride and sulphate ions have the same charge as the membrane, so anion removal can occur due to the electrostatic repulsion mechanism caused by negative charge density on the membrane surface [33]. The lower values of Cl^- rejection are due to the Cl^- ion charge having less charge and higher mobility than SO_4^{2-} [16]. The one thing that is a concern in our research result is that MgSO_4 rejection was less than 90% of the MgSO_4 rejection on the nanofiltration membrane. García-Pacheco, *et al.* [31] explain this as the borderline between the nanofiltration and the ultrafiltration membrane. Moreover, the membranes reduced the turbidity to almost 100%. Although this research has shown that using RO membranes as filtration membrane has promising potential, further study is required into the use of those membranes in an appropriate application.

5 Conclusions

Knowledge of foulant composition and the cleaning mechanism are important to achieve an effective cleaning procedure. Based on the SEM and FTIR analysis results from this research, almost all foulants that contaminated the membrane

surface were organic and inorganic compounds. Alkaline cleaning was effective, i.e. it led to a more significant cleaning effectivity increment, but the selectivity of the membrane declined. The filtration test of the membranes showed that SO_4^{2-} ion rejection was more effective than Cl^- ion rejection and could reduce the turbidity to almost 100%. Although this research showed that using RO membrane as a filtration membrane has promising potential, further study is required into the use of such membranes in an appropriate application.

Acknowledgments

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