

Normalization Method for Earlier Foulant Detection on A Reverse Osmosis Membrane Surface

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ABSTRACT

Operating Brackish Water Reverse Osmosis (BWRO) unit which a membrane technology in a water treatment system, operating parameters found to alter causing permeate flow and salt passage to shift, which leading to inaccurate evaluation of membrane condition. The observed operating parameters must be standardized in order to discern between such typical events and performance variations owing to fouling or issues called Normalization. Among normalization parameters, the objective of this work is to evaluate RO membrane condition using Normalized Permeate Flow (NPF) method at constant recovery setup applied in an RO plant facility of a factory located in Cilegon, Banten, Indonesia. The NPF show distinguish result compared to actual permeate flow (Qpa). Qpa tends to stable or a slow deterioration at 7%, which leading inaccurate conclusion since Qpa is spot data. In contrast, NPF shows 27% drop of membrane performance since its first run until the end of running cycle and the membrane must be immediately clean. In conjunction with NPF, applied pressure (Pfa) tended to increase up to 23.7%. Such higher Pfa is required to solve the osmotic pressure on feed-brine surface of the membrane even with the same amount of product. The higher osmotic pressure is caused by foulant on the membrane surface even with constant raw water concentration. Chromatic Elemental ImagingSM (CEISM) and Energy Dispersive Spectroscopy (EDS) analysis methods are used to understand type of foulant on the membrane surface. CEI detected a high weight percent of silicon as the primary foreign inorganic elements present on the membrane surface, while EDS analysis detected a layer of silicate at 40.22% wt. coated the membrane surface evenly.

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Introduction

In recent years there has been considerable growth in the utilization of reverse osmosis (RO) processes in major desalination plants ^[1]. Reverse osmosis is among the finest levels of filtration available. The RO membrane generally acts as a barrier to all dissolved salts and inorganic molecules, as well as organic molecules with a molecular weight greater than approximately 100. Water molecules, on the other hand, pass freely through the membrane creating a purified product stream. Rejection of dissolved salts is typically 95% to greater than 99%, depending on factors such as membrane type, pressure, temperature, recovery, and feed composition ^[2,17].

A factory located in Cilegon City, Banten Province, Indonesia commit to the company vision by conducting sustainability improvement program.

Optimizing Brackish Water Reverse Osmosis (BWRO) Cap. 2 x 120 m³/hour of feedwater by maintaining BWRO membrane lifetime up to 4 years considered as one of challenging improvement program. In addition, raw water characteristics that is used as feedwater containing high silica concentration in the feedwater. Need extra effort from the whole related parties to ensure achievement of the program.

The following table shows typically water analysis used in the RO system as feedwater

Table 1. Composition of RO feedwater

Parameters	Unit	Value
pH		7.15
Electrical conductivity	uS/cm	354.68
M Alkalinity as CaCO ₃	ppm	30.92
Chloride as Cl	ppm	16.83
Total Hardness as CaCO ₃	ppm	66.67
Total Iron as Fe	ppm	0.07

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Parameters	Unit	Value
Silica as SiO ₂	ppm	123
Silt Density Index	-	2.26
Residual Chlorine as Cl ₂	ppm	0.04

In an RO system, the most common sparingly soluble salt encountered are CaSO₄, CaCO₃, and Silica. Scaling of RO membranes may occur when sparingly soluble salts are concentrated within the element beyond their solubility limit [2,21].

Reverse osmosis (RO) systems are capable of separating dissolved ions from a feed stream based on salt diffusion mechanism. In RO systems, feed water is split into two streams: one with a (very) low salinity and one with a high salinity. The low salinity stream is known as permeate or product water while the high salinity stream is known as concentrate, brine, or reject [2,3,15].

In operating RO system, operating parameters found to alter causing permeate flow and salt passage to shift, which leading to inaccurate evaluation of membrane condition. The observed operating parameters must be standardized in order to discern between such typical events and performance variations owing to fouling or issues. Normalization is the process of comparing actual performance to a reference performance while accounting for the effects of operating conditions. The data is analyzed to see if the performance of the membrane system has altered over time. This is referred to as normalization as ASTM D4516 method [4].

The concepts of normalization can be used to calculate normalized productivity or normalized permeate flow (NPF) based on three commonly facts: permeate flow increases proportionally with increased net pressure, permeate flow increases with increased feed temperature, and at constant pressure and temperature, permeate flow decreases over time due to fouling, and increases over time because of membrane deterioration [5].

The objective of this work is to evaluate RO membrane condition using NPF method at constant recovery setup applied in an RO plant facility of a factory located in Cilegon, Banten, Indonesia.

Methods

Calculation of Normalized Permeate Flow (NPF)

Normalization is a comparison of the actual performance to a given reference performance while the Influences of operating parameters (e.g. ressure, temperature, recovery rate, and feed concentration) are taken into account [2,19]. It involves referring the plant operation back to a standard condition, which

can be defined as the operation condition when the RO plant shows the reference performance [6,19]. The reference performance may be the designed performance or the measured initial performance. Normalization with reference to the designed system performance is useful to verify that the plant gives the specified performance, while normalization with reference to initial system performance is useful to show up any performance change between day one and the actual date [2,6,15]. Among several normalization parameters, this work focused on NPF using Eq (1) suggested by ASTM D4516 [7,20].

$$Q_{ps} = \frac{P_{fs} - \frac{\Delta P_{fbs}}{2} - P_{ps} - \pi_{fbs} + \pi_{pa}}{P_{fa} - \frac{\Delta P_{fba}}{2} - P_{pa} - \pi_{fba} + \pi_{pa}} \cdot \frac{TCF_s}{TCF_a} \cdot Q_{pa} \quad (1)$$

where Q_{pa} = permeate flow at actual conditions, Q_{ps} = permeate flow at standard conditions (normalized permeate flow), P_{fa} = feed pressure at actual conditions (kPa), P_{fs} = permeate pressure at standard conditions (kPa), P_{pa} = permeate pressure at actual conditions (kPa), P_{ps} = permeate pressure at standard conditions (kPa), $\Delta P_{fba}/2$ = one half device pressure drop at actual conditions (kPa), $\Delta P_{fbs}/2$ = one half device pressure drop at standard conditions (kPa), π_{fba} = feed-brine osmotic pressure at actual conditions (kPa), π_{fbs} = feed-brine osmotic pressure at standard conditions (kPa), π_{pa} = permeate osmotic pressure at actual conditions (kPa), π_{ps} = permeate osmotic pressure at standard conditions (kPa), TCF_a = temperature correction factor at actual conditions, and TCF_s = temperature correction factor at standard conditions.

TCF is generally affected by the RO membrane, and it is ideal to obtain TCF from the membrane manufactures. Since the TCF from membrane manufacturers is not known, TCF is calculated using a membrane-independent equation (TCF₁) [7].

$$TCF_1 = 1.03^{(T-25)} \quad (2)$$

where T is temperature (°C).

For the osmotic pressure is calculated using equation (3) [2].

$$\pi_{fc} = \frac{C_{fc}(T+320)}{491000} \quad (3)$$

Where π_{fc} is empirical osmotic pressure equation and C_{fc} is feed-brine concentration (mg/l).

For feed-brine concentration is calculated using equation (4)

$$C_{fc} = C_f \cdot \left(\frac{\ln\left[\frac{1}{(1-R)}\right]}{R} \right) \quad (4)$$

Where C_f is feed concentration and R is operational recovery. Salt rejection is assumed to be 100% [2].

As the osmotic pressure and TCF are quite site-specific, it is important to select proper equations reflecting the characteristics of feed water and RO membrane used in the field for better normalization performance. The better normalized performance means that the NPF data group has a lower variance (or standard deviation) during an operation period without fouling.

Data Collection

Data was collected in an RO plant facility of a factory located in Cilegon, Banten, Indonesia since October 2021 to March 2022 operational period. Analysis of the data is carried out with Microsoft Excel result in a trending chart. Laboratory analysis was also conducted to analysis type of foulant on the membrane surface by using Chromatic Elemental ImagingSM (CEISM) and Energy Dispersive Spectroscopy (EDS)

Results and Discussions

NPF data for fouling detection

The NPF data are produced as a result of normalization using ASTM D4516 method (Eq. 1). Figure 1. presents NPF and actual permeate flow or Q_{pa} . Since the membrane experienced several cleaning actions and to do a proper evaluation, the chart is divided into two regimes called A dan B regime to identify membrane performance at each cleaning cycle. In the A regime, Recovery was maintained at constant state at 50% resulted in Q_{pa} 56.17 m³/h at first run after cleaning in place (CIP) action, while NPF at 58.05 m³/h.

Along with the operational run, there was no change in recovery until the end of the cycle. However, feedwater flow was re-set to gain more permeate water which not affect to NPF trend. Deterioration of Q_{pa} was 7.0% based on the observation along the running cycle, while NPF shows higher deterioration, 27% drop since its first run.

At this point, the membrane must be immediately cleaned. Furthermore, in the B regime, Q_{pa} showed stable value instead which no permeate flow drop along the run. However, NPF shows differently. The membrane performance dropped by 17.7% and the membrane must be immediately cleaned.

According to Figure 3. Typical problem symptoms of reverse osmosis membrane^[8]. Typical last stage scaling possibly to occur such as CaSO₄, BaSO₄, CaCO₃, and SiO₂.

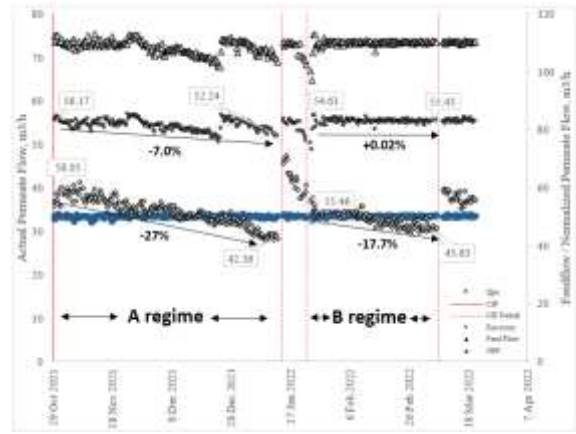


Figure 1. Profile NPF at constant recovery

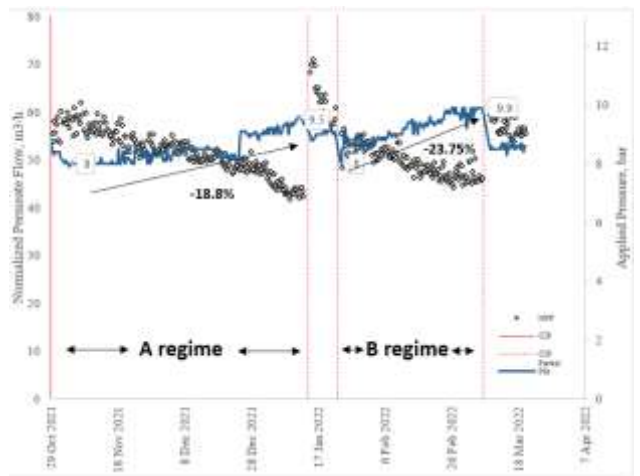


Figure 2. Profile NPF vs Applied pressure

Observation	Normalized Permeate Flow	Normalized Recovery Ratio	Feed Concentration	Possible Cause	Resolution	Correction Action
↗	↘	↗	↘	Metals fouling	Analysis of metal ions in cleaning solutions or deposit during reverse	Improved pretreatment to remove metals Sed cleaning
↗	↘	↗	↘	First stage Colloidal fouling	EDS measurement of feed Analyze deposit during membrane autopsy	Optimized pretreatment system for colloidal removal Feed 100-10 High pH cleaning with anionic detergent formulation
↗	↘	↗	↘	Last stage Scaling CaCO ₃ , CaSO ₄ , BaSO ₄ , SiO ₂	Analysis of metal ions in cleaning solution	Proper scale inhibitor dosage and/or reduce pH Reduce recovery Cleaning
↗	↘	↗	↘	Last stage Scaling SiO ₂	EDS/Energy Dispersive X-ray Spectroscopy of membrane surface	Strong acid cleaning Reduce recovery
↗	↘	↗	↘	First stage Colloidal fouling	Check presence of silica in spool, vessels and ends of elements Element autopsy	Remove TIC component of PE with addition of EDPA, check dose or formulation Clean with alkaline silicate surfactant
↗	↘	↗	↘	Stable All stages	Analyse deposit during membrane autopsy	Optimization of pretreatment system (e.g. coagulation pre-treat) Revers/activated carbon treatment Clean with high pH detergent
↗	↘	↗	↘	All stages but start first stage Chlorine contact attack	Chemical analysis of feed Follow test on membrane flat sheet during membrane autopsy	Check chlorine feed equipment and disinfection equipment
↗	↘	↗	↘	First of last stage Release of membrane by uncontrolled crystals	Microscopic/white analysis of feed during membrane autopsy	Improved pretreatment Check all filters for media leakage Adjust fine particulate dosage Reduce recovery
↗	↘	↗	↘	Stable All random O-ring leaks, seal of side glass leaks	White test Vacuum test, Colloidal material passage	Replace O-rings Repair or replace the elements

Figure 3. Typical problem symptoms of reverse osmosis

To prevent scaling of sparingly soluble salt in a concentrated water along the membrane, type of antiscalant and non oxidizing biocide chemical were dosed at certain dosage but not being further discussed in this work.

As RO operation proceeds, the silica level in the concentrate stream increases and often reaches saturation, which can cause deposits of silica, or precipitation of metal silicates on the membrane surface (Scaling). Silica fouling is very difficult to remove from RO membrane, and eventually leads to performance deterioration such as permeability loss and premature system shutdown^[9].

The following table shows recorded water analysis of brine stream.

Table 2. Water analysis of brine stream

Parameters	Unit	Value
pH		7.5
Electrical conductivity	uS/cm	1,924
T-Hardness as CaCO ₃	ppm	92,67
Silica as SiO ₂	ppm	170

Silica scaling occurs when its concentration exceeds the allowable solubility. Crystalline silica has a low solubility of 5–6 mg/L, whereas the solubility of amorphous silica ranges from 120-150 mg/L at 25°C^[10]. In the water treatment system, the solubility of amorphous silica is affected mainly by temperature, pH, and the presence of other ions^[11]. The presence of multivalent cations such as hardness, iron, and aluminum reduce the apparent solubility of silica due to their interactions with the silicate anion. Particularly, calcium and magnesium catalyze the polymerization reaction of dissolved silica. This means that higher concentrations of total hardness leads to a faster drop in dissolved silica level^[11, 12].

Effect of fouling on the pressure increase

Figure 2 presents NPF drop and its correlation to the increment of applied pressure. In the A regime, applied pressure (Pfa) increased from 8 bar at its initial run to 9.5 bar at end of cycle, the increment was 18.8%, while the NPF as the aforementioned at 27% drop. While in the B regime, the Pfa increased from 8 bar at its initial run to 9.9 bar at the end of cycle, the Pfa increment was 23.75%

According to Figure 3. Typical problem symptoms of reverse osmosis membrane^[8]. It is clearly observed that Pfa increased in conjunction

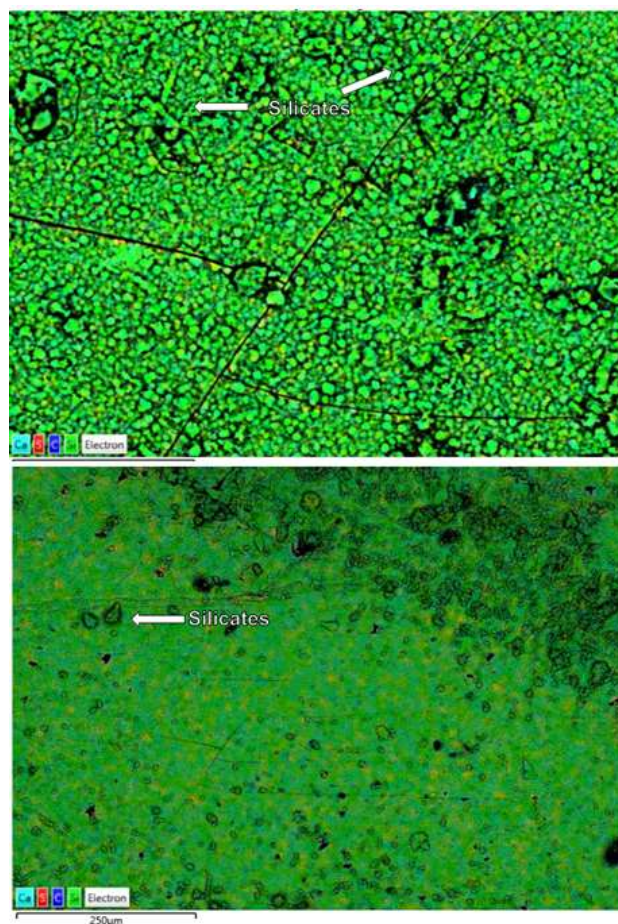


Figure 4. Foulant analysis by CEISM

with NPF drop. Higher Pfa was required to solve osmotic pressure on the membrane surface^[2,14,18]. Osmotic pressure at initial run obviously low, then required lower Pfa. While, at the end of the cycle, osmotic pressure increased due to foulants on the membrane surface (specifically on the feed-brine side) even with constant raw water concentration.

Laboratory foulant analysis

Figure 4. presents laboratory analysis of foulant found on the membrane surface by using Chromatic Elemental ImagingSM (CEISM). CEI detected a high weight percent of silicon as the primary foreign inorganic elements present on the membrane surface. The layer appeared to be relatively evenly distributed across the membrane surface. The fouling layer was relatively thick preventing the signal from the membrane, represented by sulfur (red), from being observed through the foulant. Low amounts of carbon (dark blue) and calcium (cyan) were scattered across the membrane surface. Carbon is more likely contributed by the membrane materials.

Energy Dispersive Spectroscopy (EDS) analysis was also conducted to observed relative

concentrations of foulant on the membrane. The following table shows EDS analysis result

Table 2. EDS analysis of foulants

Parameters	Unit	New Membrane	Value
Carbon	% wt	55 – 75	3.28
Oxygen	% wt	15 – 20	55.88
Sulfur	% wt	5.5 – 6.5	0.43
Silicon	% wt	-	40.22
Calcium	% wt	-	0.19

EDS analysis detected a layer of silicate coated the membrane surface evenly. The low amount of sulfur detected indicates a relatively thick foulant layer. Trace amount of calcium was also detected.

Conclusion

Normalization is an important technique to properly operate BWRO process because it can be a tool for early fouling detection compared to actual instrumental reading. Researchers in this work applied a normalization method a Brackish Water Reverse Osmosis (BWRO) facility in a industry with installed capacity 2 x 120 m³/hour of feedwater. The NPF show distinguish result compared to actual permeate flow (Q_{pa}). Q_{pa} tends to stable or a slow deterioration which leading inaccurate conclusion since Q_{pa} is spot data. In contrast, NPF shows declining membrane performance since its first run until the end of running cycle. In conjunction with NPF, applied pressure (P_{fa}) tended to increase as well. Such higher P_{fa} is required to solve the osmotic pressure on feed-brine surface of the membrane even with the same amount of product. Higher osmotic pressure is caused by foulant on the membrane surface even with constant raw water concentration, Chemical treatment is used in this work such as antiscalant and non-oxidizing biocide. CEISM and EDS analysis methods are used to understand type of foulant on the membrane surface. CEI detected a high weight percent of silicon as the primary foreign inorganic elements present on the membrane surface, while EDS analysis detected a layer of silicate coated the membrane surface evenly.

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Author Contributions

Conceptualization, MK Ummam; methodology, MK Ummam; software, MK Ummam; validation, MK Ummam; formal analysis, MK Ummam; investigation, MK Ummam; resources, MK Ummam; data curation, MK Ummam; writing—original draft preparation, MK Ummam; writing—review and editing, MK Ummam, TY Hendrawati; visualization, MK Ummam, TY Hendrawati.; supervision, TY Hendrawati.; project administration, TY Hendrawati.; funding acquisition, TY Hendrawati. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest

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