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Investigation of SWRO membrane failures of two different configurations

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ABSTRACT

Membrane autopsy is a useful tool for identification of operational problems. In the procedure, membranes are cut open and their surfaces are examined. Chemical, physical and microbiological analyses are used to determine the type of foulant present. In addition, intrinsic viscosity measurement and degree of acetylation studies are carried out depending on the chemical nature of membranes to find out the extent of degradation. Other techniques such as scanning electron microscope (SEM) and Energy Dispersive X-ray (EDX) are also used to augment the autopsy. Two cases studies are presented. Cellulose triacetate (CTA) hollow fine fiber (HFF) SWRO membranes and thin film composite (TFC) spiral wound (SW) SWRO membranes were examined to identify root causes of performance decline. Both types of membranes are used in commercial SWRO plants operating along the Red Sea coast. Autopsy revealed that HFF membrane element was fouled by a mixture of organic matter, fine silt and iron oxides, whereas biological fouling was absent. The presence of fine silt on the membrane surface indicates inadequacy of pretreatment. The SW membrane element fouled by organic matter as well as iron in addition to corrosion product. Moreover, autopsy of a chemically cleaned SW membrane proved that cleaning was efficient in removing most (87%) of the foulants, especially the inorganic portions, except for some of the organic matter, which remained on the membrane surface.

Keywords: RO; Seawater; Fouling; Autopsy; SWRO; TFC; HFF; CTA

1. Introduction

The reverse osmosis (RO) process is emerging as a competitor to conventional thermal desalination processes due to technical advances which reduce cost of water produced by RO. Almost 56 million m^3/d of desalinated brackish and seawater is produced by RO which accounts for nearly 65% of the world's total installed desalination capacity [1]. Membranes form the heart of RO processes. Occasionally, these membranes get affected by fouling and/or by membrane degradation affecting plant performance and its ability to deliver the required quantity and quality of product water. Fouling is a phenomenon by which unwanted materials get deposited on the membrane surface. This results in reduced product flow or higher energy demands in addition to occasional reductions in product quality. Fouling is due to deposition of colloidal particle from feed water or organic matter or other suspended matter and or due to growth of biological species. Also, sometimes it could be due to chemical scaling from the precipitation of saturated salts on the membrane surface. The phenomenon of membrane degradation could happen due to oxidation or hydrolysis of membrane polymers depending on membrane chemical composition. Although an experienced operator may have some idea regarding type of problem from the deviation in process parameters, it is extremely difficult to specifically diagnose the problem. Proper identification of the problem, whether fouling or membrane degradation, is the key to take corrective actions. Thus, autopsy becomes an essential tool to properly identify the actual cause for performance deterioration of membranes. Autopsy is a destructive procedure where representative

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membranes are identified and removed from membrane racks and subjected to various detailed analyses involving physical, chemical and biological analyses. Hence, autopsy becomes a very useful tool for identification of poor membrane performance causes, which can help in selection of effective remediation measure to mitigate problems facing RO plant. These measures include problem prevention by improving pretreatment process or proper selection of chemicals for membrane cleaning to remove foulants from membrane surfaces without adversely affecting membrane performance.

Desalination technologies research institute (DTRI) has been offering autopsy services to RO plants throughout Saudi Arabia. The institute works on different kinds of RO and NF membranes to identify the problems faced by RO plants and offers solution [2–9]. The types of membrane examined include both brackish water RO as well as seawater RO (SWRO) of spiral wound (SW) and hollow fine fiber (HFF) configurations as well as nanofiltration (NF) membranes used for seawater pretreatment. These membranes are of different chemical composition such as polyamide, cellulose acetate as well as thin film composite (TFC) polymers.

Polyamide based spiral wound (SW) thin film composite (TFC) membrane and cellulose triacetate (CTA) based hollow fine fiber (HFF) membrane are the two types of SWRO membranes presently available in the market. SW TFC membranes are the most commonly employed RO membrane in SWRO desalination plants worldwide [10–12]. Whereas, HFF CTA membranes are extensively used in several desalination plants in Saudi Arabia, mainly due to its claimed ability to tackle biofouling, which is one of the major challenges in the region [12–15]. This was due to the superior chlorine resistance of HFF CTA membrane, thereby preventing the growth of microorganisms and algae by chlorine disinfection. Chlorination of feed water is a commonly used strategy to prevent biological fouling in RO systems [13]. However, polyamide SW TFC membranes are susceptible to degradation by chlorine, which warrants de-chlorination of the feed water to the membrane [10,12]. Chlorine is capable of destroying not only biofilm forming microorganisms but also the organic matter deposited on the membrane surface [13,14]. Hence, HFF CTA membrane exposed to chlorine is expected contain lesser organic/biofilm formation on the membrane surface compared to the SW TFC membrane.

Membrane autopsy procedure has been widely used to identify fouling pattern in SWRO membranes [16–22]. The advantage of membrane autopsies is that accurate information about the composition could be obtained. Also, autopsy could be used to assess the chemical cleaning efficiency of RO membranes. In this study, autopsy procedure was used to identify fouling pattern of two different types of membranes which were operated on Red sea coast. The feed seawater for both membranes were obtained from open intake and employed almost similar pre-treatment scheme except that, in the case of HFF CTA membrane, it was exposed to chlorine for one hour in a day of operation. Also, autopsy was used to evaluate the effectiveness of chemical cleaning on foulant removal from SW TFC membrane. Thus by presenting two case studies, the present study aims to highlight the importance of membrane autopsy procedure in identifying possible cause of fouling as well as in verifying effectiveness of chemical cleaning of SWRO membranes.

2. Autopsy procedures

The autopsy procedure can include visual inspection, scanning electron microscope (SEM), energy dispersive X-ray (EDX), and various chemical analysis.

2.1. Visual inspection

The visual inspection includes exterior examination of membrane for any mechanical damage such as cracks, splits or evidence of physical abuse. Deposits, if available, are collected for further examination. The membrane is then cut open to observe any unusual patterns or deposits on membrane leaves, spacers and other parts. During the examination, foulant deposits on the surface are carefully scraped off for further chemical and biological analyses. Also, representative pieces of membrane are cut from different locations for estimating density of foulants.

2.2. Scanning electron microscope (SEM) and energy dispersive X-ray (EDX)

Representative samples collected from different parts of the membrane are dried under vacuum at low temperature prior to performing SEM and EDX analyses. SEM provides detailed magnified image of morphological features of foulants deposited on the surface of the membrane. SEM provides evidence of biological organisms and/or scale deposits. EDX provides chemical elemental compositions and elemental distributions of foulants of either biological or chemical origins.

2.3. Chemical analyses

Foulants collected from membrane surfaces are subjected to various wet chemical analyses and instrumental analyses using inductively coupled plasma (ICP-Mass) and atomic absorption spectroscopy (AAS) to quantitatively determine major inorganic components. Total organic content is estimated by loss of ignition at 550°C.

2.4. Intrinsic viscosity measurement

Intrinsic viscosity measurements are carried out on polyamide based membranes as well as cellulose acetate based membranes. Measurements to assess the incidence of membrane polymer chain scission by means of oxidation and/or by hydrolysis. Prior to the intrinsic viscosity measurements, membrane fibers are cleaned and rinsed overnight using flowing distilled water. The membrane samples are then dried (in an oven at 105°C) for 6 h. Intrinsic viscosity [η], which is related to the polymer molecular weight, is determined following standard procedure [4]. Ubbelohde type capillary viscometer is used at constant bath temperature. After determining the efflux time (t_o) for the solvent dimethyl sulfoxide (DMSO), the efflux time (t_i) is determined for the known concentration of the sample. By

successive dilution using the DMSO solvent, measurements are made at different concentrations for each sample. The intrinsic viscosity [η] is determined as intercept of the plot of η_{sp}/c against c, where c is concentration (*g/dl)* of membrane sample and η_{sp} is specific viscosity. The specific viscosity can be obtained from relative viscosity (η_{rel}) as $\eta_{rel}-1$. The η_{rel} can be determined from the efflux time of solvent t_o and sample t_i as $\eta_{rel} = t_i/t_o$.

2.5. Degree of acetylation measurement

For cellulose acetate based membranes, the loss of acetylation is determined. With slight modification to the standard ASTM procedure [4], the acetylation degree of membrane fibers, which is normally expressed as percent of acetyl group in the polymer, can be determined. Initially, membrane fibers collected from the autopsied elements are cleaned overnight using distilled water to remove any deposits on the fibers and then dried for 6 h at 105°C. A known amount of membrane sample (0.7 g) is then added to 70 ml of acetone in an Erlenmeyer flask and stirred using a magnetic stirrer, for 1 h followed by addition of 5 ml of DMSO. After 30 min of additional stirring, 1N NaOH is added in excess (15 ml) to the membrane sample solution and kept stirring for another 1 h. After adding hot (60°C) distilled water for washing down the sides of the flask and continuing stirring for another 10 min, the un-reacted excess amount of NaOH is titrated against standard sulfuric acid (0.5 N) using phenolphthalein indicator. When the pink color disappears completely, excess (0.2–0.3 ml) sulfuric acid is added and re-titrated with 0.1 N NaOH. For each sample, a duplicate is also carried out as well as two blank. The acetylation degree is then calculated as the percentage weight of combined acetic acid formed during hydrolysis by the excess NaOH to the total weight of membrane polymer.

3. Two case studies

Two cases on problems faced by different SWRO plants which utilize different types of membrane in terms of composition and configuration will be examined.

3.1. Cellulose triacetate (CTA) HFF SWRO membrane

The first example is a CTA HFF SWRO membrane used in a SWRO plant along Red Sea coast which showed poor performance after a few months of operation. The symptom was a decline in plant production and recovery ratio and a slight increase in salt passage. The plant was in operation for about four years and received feed from an open intake where chlorination was applied on feed seawater to maintain a residual chlorine level of about 0.5 ppm in the pretreatment. The pretreatment consists of conventional gravity type dual media filters (DMF). Ferric chloride was dosed as coagulant and sulfuric acid was added to pretreatment system to maintain pH to about 6.5. Sodium bisulfite (SBS) was added downstream of cartridge filter for dechlorinatuion except for an hour in a day so as to expose the RO membrane to residual chlorine. The plant was operated at a recovery of 35%. Chemical cleaning of the membranes are carried out at interval of 6 mo period. 2% citric acid solution with pH of 4 adjusted by adding ammonium hydroxide was used for chemical cleaning. Upon inspection, the membrane was found to be free from any visible defects (Fig. 1). Moreover, the outer fibers of the membrane was found to be very clean free from deposits and looked like new (Fig. 2).

However, the fibers in the center portion of the element were found to be reddish brown in color (Fig. 3). The color intensifies closer to the feed tube (Fig. 4). Additionally, lot of

Fig. 1. A view of CTA HFF SWRO membrane before dissection.

Fig. 2. A closer view of membrane showing very clean outer fibers.

Fig. 3. A view of middle portion of the membrane with discoloration.

Fig. 4. A view of center portion of the membrane closer to the feed tube.

anthracite particle were observed on the fibers near the feed tube (Fig. 5), indicating defects in cartridge filters.

A distinct color difference among the fibers obtained from different sections (outer, middle and inner) is quite clear as shown in Fig. 6. This pattern is typical in hollow fine fiber (HFF) membrane where most of foulants get trapped on the fibers close to the feed tube. These foulants were found to loosely adhere to the fibers. Additionally, an unpleasant odor was detected indicating possible organic or biological fouling origin.

The foulant deposits collected from the inner fibers were subjected to chemical analyses and the results are given in Table 1. The primary organic matter forms about only 31.3% indicating absence of biofouling. In typical biofilms, total organic matters accounts for more than 50% of the dried foulant deposit [23]. Among the acid soluble portion of foulants, iron (Fe) constitute major inorganic component amounting to 15.6%. Iron could be either from the coagulant dosed or due to corrosion of stainless steel piping which is evident from the presence of chromium (Cr) and nickel (Ni).

SEM and EDX analyses were conducted on gold coated fibers collected from outer, middle and inner portion of the membrane. SEM and its corresponding EDX spectra of outer fiber are shown in Fig. 7. The EDX spectra reveal the absence of any inorganic foulant deposits on the fibers.

Fig. 5. A view of inner portion of the membrane close to the feed tube showing anthracite particles.

Fig. 6. Distinct color difference among the membrane fibers collected from outer, middle and inner portion of the membrane (from left to right).

SEM and its corresponding EDX spectra of fibers obtained from middle portion are shown in Fig. 8, where the foulant deposits is found to be mainly consisting of Si, Al, Mg and Fe as seen in Fig. 8. The deposits look like a mixture of fine silt and iron oxide.

Inner fiber also shows similar SEM and EDX to that of middle fiber as can be seen in Fig. 9. Moreover, distinct features of biofouling are absent in SEM images.

Percentage of acetyl content of outer, middle and inner fibers are 59.4, 59.7 and 59.9%, respectively revealing very slight hydrolysis of the membrane polymer.

Table 1
Chemical analyses of foulant deposit collected from the CTA HFF SWRO membrane

S. No.	Parameter	Weight %
1	Primary organic matter	31.3
2	Iron	15.6
3	Chromium	1.2
4	Magnesium	1.0
5	Nickel	1.0
6	Aluminium	0.8
7	Potassium	0.4
8	Calcium	0.3

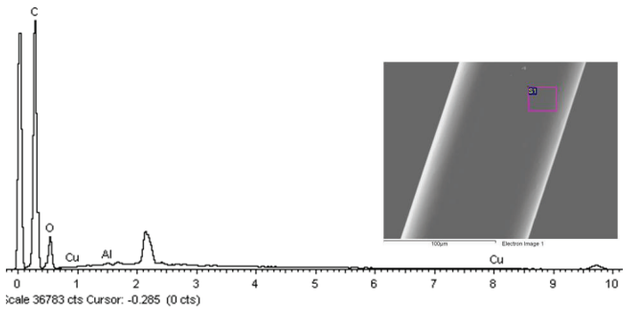

Fig. 7. Outer Fiber: EDX Spectrum and SEM of relatively clean outer fiber free from any foulant.

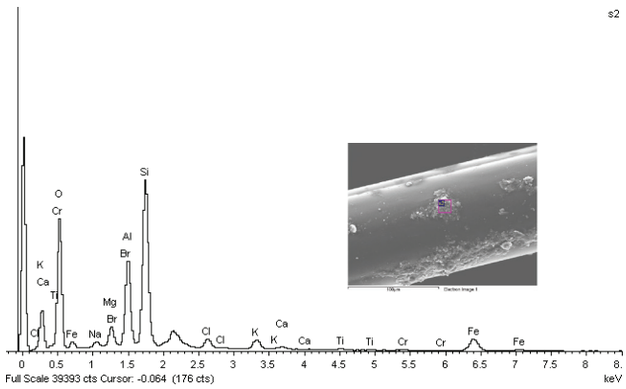

Fig. 8. Middle Fiber: EDX Spectrum and SEM of middle fiber showing EDX of foulant deposit.

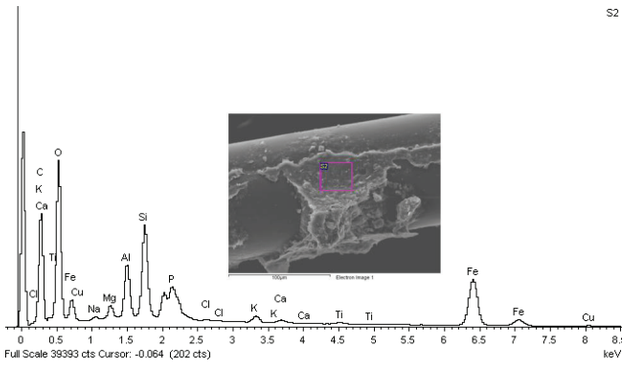

Fig. 9. Inner Fiber: EDX Spectrum and SEM of inner fiber showing EDX of foulant deposit.

Typical percentage of acetyl content in a virgin membrane fiber is in the range of 59.9 to 60.7% according to the membrane manufacturer. Intrinsic viscosities of outer, middle and inner fibers are 1.69, 1.65 and 1.57 dl/g, respectively revealing absence of membrane polymer degradation.

The autopsy reveals that the membrane element was fouled by a mixture of organic matter, fine silt and iron oxides. Biological fouling was absent as evident from lower organic content of less than 50%. SEM images did not indicate any biological activity. Iron oxides could have originated from corrosion of stainless steel parts as evident by the presence of Cr and Ni and also due to the coagulant usage. Presence of fine silt on the membrane surface indicates inadequacy of pretreatment in preventing particulate fouling. Hence, the pretreatment has to be enhanced to handle particulate fouling. Also, presence of anthracite particles indicates failure of cartridge filter.

3.2. Spiral wound TFC SWRO membrane

Two spiral wound membranes operated in a SWRO plant located along the Red Sea was investigated for their poor performance and to test the effectiveness of chemical cleaning performed to restore the performance. One of the membranes was before chemical cleaning (BCIP) and other was immediately after cleaning (ACIP). The plant was in operation for about 7 mo only and received feed from an open intake where chlorination was applied on feed seawater to maintain a residual chlorine level of about 0.3 ppm in the pretreatment. The pretreatment consists of conventional pressure type dual media filters (DMF). Ferric chloride was dosed as coagulant and sulfuric acid was added to pretreatment system to maintain pH to about 6.8. Sodium bisulfite (SBS) was added downstream of cartridge filter for dechlorinatuion. The plant was operated at a recovery of 41%. Chemical cleaning of the membranes are carried out 3 mo after the initial startup. High pH cleaning using EDTA (1%) followed by low pH cleaning using 2% citric acid was carried out.

Upon inspection, both membranes (BCIP & ACIP) were found to be free from any visible defects. However, particles of different sizes were seen on feed side end of the both membranes (Fig. 10), whereas brine side end

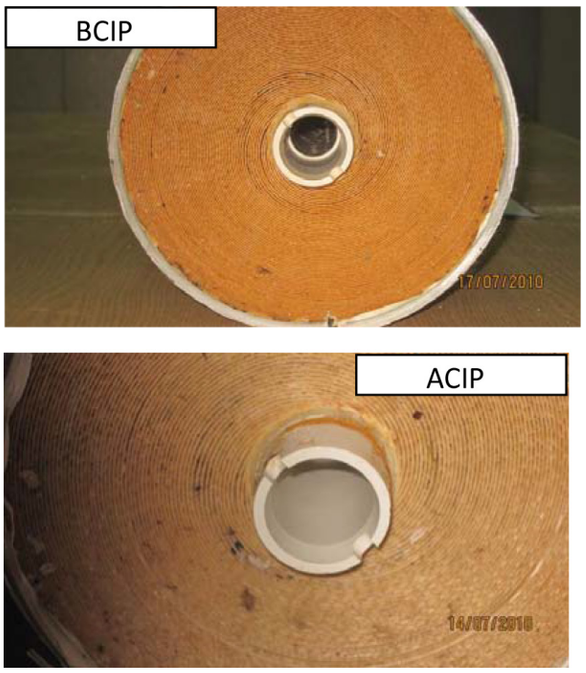

Fig. 10. Feed side of membrane elements (both BCIP & ACIP) showing anthracite as well as sand particles.

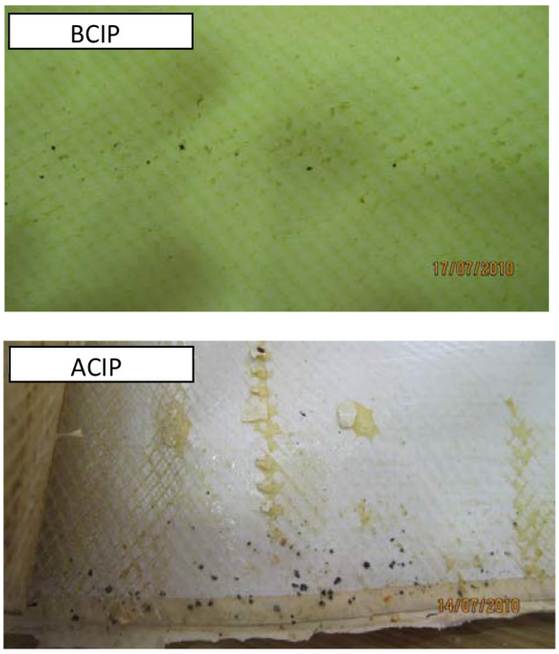

Fig. 11. Membrane element (BCIP & ACIP) showing lots of sand and anthracite particles on membrane surfaces.

of both elements were very clean, indicating that these particles escaped cartridge filters and reached the membrane element. Moreover, detailed investigation of both membranes leaves also revealed the presence of sand and anthracite particles on both membranes surfaces (Fig. 11) thus demanding thorough investigation of cartridge filter unit as well as pretreatment.

Further investigation of the BCIP membrane leaves reveal that there is a uniform coating of reddish brown color foulant on all leaves (Fig. 12) with distinct absence of any foul smell. Moreover, these foulants could be easily scraped off from the membrane surface (Fig. 13) and are very loosely bound. Additionally, the feed spacer looks very clean with no adherence of foulants.

Similar to BCIP, ACIP also had uniform coating of foulant with absence of reddish brown color and having more water content (Fig. 14). Deposits on ACIP also could easily scraped off the surface (Fig. 15). In addition, no foul smell was detected on either membranes. The distinct color differences of deposits removed from membrane are quite apparent in Fig. 16, where the higher water content on ACIP membrane deposits is clearly evident.

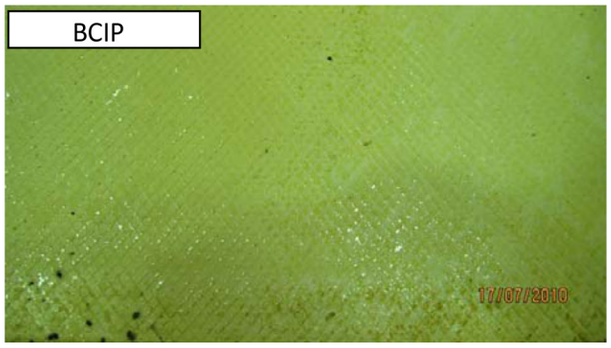

Fig. 12. BCIP membrane surface showing uniform coating of foulant.

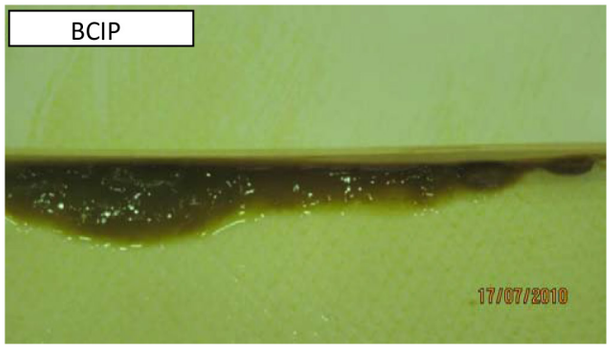

Fig. 13. BCIP membrane element showing foulants removal.

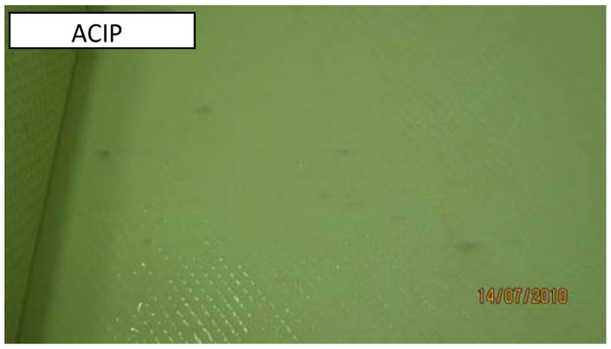

Fig. 14. ACIP membrane showing uniform deposit of foulant on the surface.

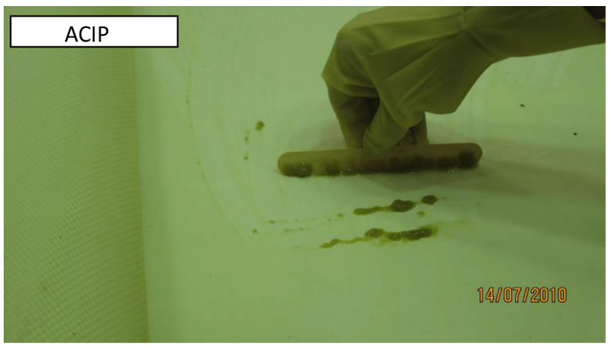

Fig. 15. Foulant being scraped off from the ACIP membrane element.

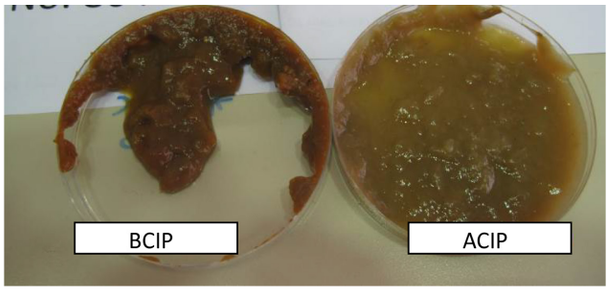

Fig. 16. View of foulant collected from both membranes.

The acid test on membrane surface showed that BCIP membranes contains significant amount of iron whereas the ACIP showed distinct absence of iron as seen in Fig. 17 where yellow color due to chemical reaction is absent on ACIP membrane.

Results of chemical analyses of foulant scraped off both membrane elements are shown in Table 2. The amount of foulant deposited on the BCIP membrane was 346 μg/cm^2 which reduced by 87% to 45 μg/cm^2 in ACIP membrane indicating significant removal of foulants with the chemical cleaning. The BCIP foulant consists of primary organic matter of about 51.8% and iron forms the major inorganic component amounting to 12.43%. On the ACIP membrane, most of the inorganic components were removed thus making the primary organic matter as the major fouling component with 72.5%.

SEM and its corresponding EDX spectra of BCIP are shown in Fig. 18. The spectra reveal the presence of inorganic foulant deposits.

SEM and its corresponding EDX spectra of ACIP membrane are shown in Fig. 19. The SEM image reveal the presence of strands like structure which is mainly organic in nature. On higher magnification, the strands are blue green algae (cyanobacteria) (Fig. 20). Moreover, a few bacteria could also be seen on the same membrane surface (Fig. 21).

The autopsy reveals that membrane element was mainly fouled by organic matter as well as iron in addition to corro-

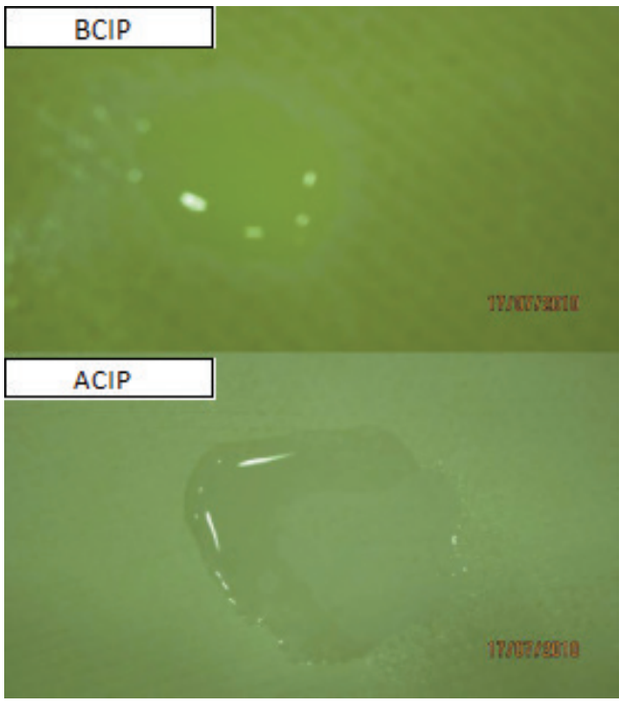

Fig. 17. View of acid test performed on the membrane.

Table 2
Chemical analyses of Foulant deposit on SW TFC SWRO membrane

S. No.	Parameter	BCIP		ACIP	
		µg/cm²	%	µg/cm²	%
	Foulant (dry)	346	–	45	–
1	Primary organic matter	179.21	51.8	32.34	72.5
2	Iron	43.00	12.43	1.36	3.05
3	Magnesium	11.97	3.46	0.79	1.78
4	Sodium	10.69	3.09	0.00	0
5	Calcium	8.75	2.53	0.66	1.48
6	Potassium	7.85	2.27	0.11	0.24
7	Phosphorus	6.12	1.77	1.82	4.09
8	Chromium	5.47	1.58	1.29	2.89
9	Aluminum	1.38	0.4	0.33	0.73
10	Copper	0.90	0.26	0.00	0
11	Nickel	0.38	0.11	0.10	0.23
12	Strontium	0.24	0.07	0.00	0
13	Zinc	0.00	0	0.01	0.02

sion product. Although cleaning was efficient in removing most (87%) of the foulant, especially the inorganic portions, some of the organic matter still remain on the membrane. Moreover, presence of blue green algae could have enhanced the organic matter content on the membrane after cleaning.

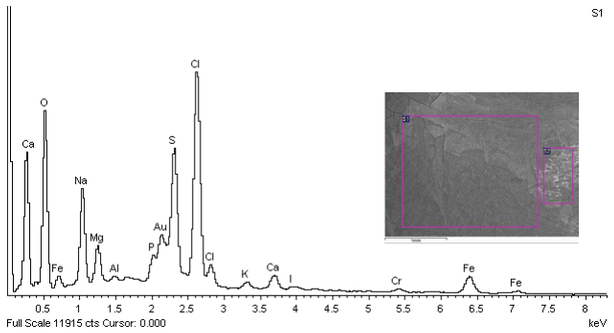

Fig. 18. SEM and EDX Spectrum of BCIP membrane foulant deposits.

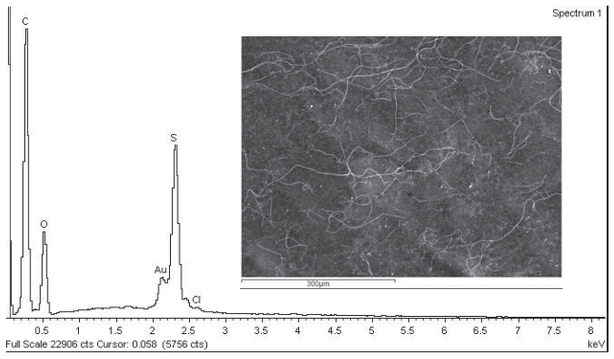

Fig. 19. SEM and EDX Spectrum of ACIP membrane showing strand like structure.

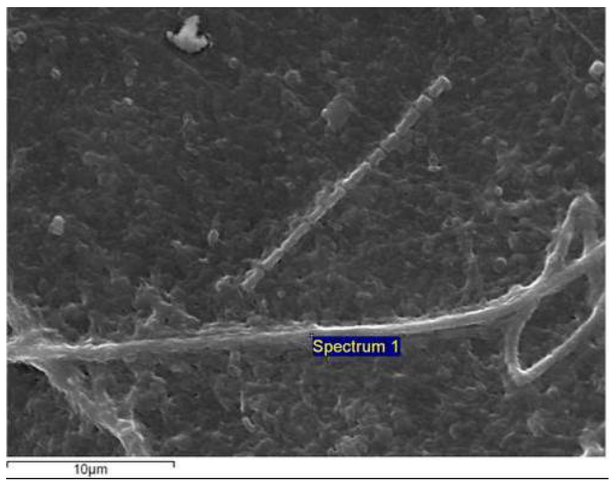

Fig. 20. SEM micrograph of ACIP membrane at higher magnifications showing presence of blue green algae.

4. Conclusion

By performing membrane autopsy of two different types of SWRO membranes operated on Red Sea coast, it was concluded that the major foulants on both HFF CTA membrane and SW TFC membranes are primary organic matter followed by iron. Moreover, HFF CTA membrane

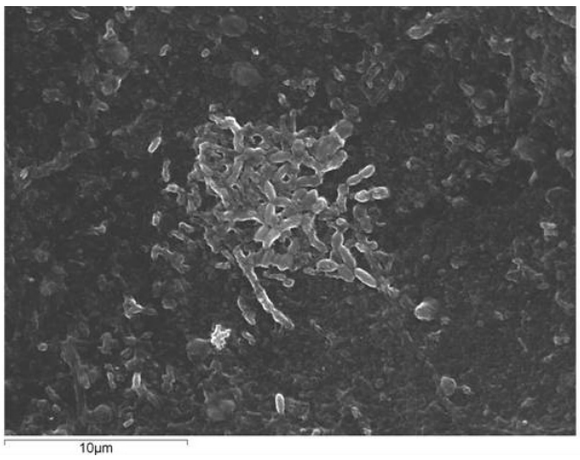

Fig. 21. SEM micrograph of ACIP membrane at higher magnifications showing presence of bacteria.

had lower percentage of organic matter, which could be mainly attributed to the exposure of the membrane to residual chlorine. The iron fouling could be mainly due to the use of $FeCl_3$ as coagulant. Although, chemical cleaning of SW TFC membrane could remove majority (87%) of foulants, it failed to completely remove the foulants from the membrane surface. These findings helped the respective SWRO plants to make proper corrective measures for the smooth operation of the plants. Thus confirming that autopsy is an important tool in resolving operation problems faced by SWRO desalination plants.

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