

## **RESULTS FROM 99 SEAWATER RO MEMBRANE AUTOPSIES**

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### **Abstract**

This paper presents results from 99 sea water reverse osmosis (SWRO) membrane autopsies carried out by the Genesys laboratory in Madrid from 2002 and outlines the technical issues surrounding fouling and scaling of sea water membranes. The authors discuss historical results of membrane autopsies and link recent trends and changes in plant design to potential fouling and scaling issues that may result in the future.

Forty percent of autopsies showed the presence of iron, 33% clay and 21% calcium and magnesium. The principle reason for membrane failure showed 27% was due to microbiological fouling, 19% were abraded, 18% oxidised, 12% of the samples showed metal oxide present as iron, manganese and aluminium. Scale formation from calcium carbonate, calcium sulphate and silica scale accounted for 8% of the membrane failures. Only 10% of the membranes were described as clean.

There has been a trend over the last decade to increase recovery rates from 30% to 40 - 50%. This results in a greater concentration of cations and anions and likelihood of scale formation in last position elements. The presence of suspended solids and micro-organisms causes fouling in lead elements. To counter this there is a common practice of chlorinating, acidifying and coagulating the feed water. This is done principally with the commodity chemicals, sodium hypochlorite, sulphuric acid and iron chloride coagulant. The authors conclude from the results of ninety-nine SWRO membrane autopsies that the use of these commodity chemicals can be beneficial in the pre-treatment system but also can have some serious side effects for the membranes. Suggestions for alternative strategies are made.



## I. INTRODUCTION

In the 1980's most seawater reverse osmosis (SWRO) plant operated at very low recoveries of 30%. Scaling and fouling was minimised using sodium hexametaphosphate (SHMP) combined with sulphuric acid dosing. This approach was not very successful as phosphate acted as a nutrient source for bacteria and was prone to hydrolysing resulting in calcium phosphate precipitation. Acidifying the feed water increased the corrosive tendency of the water and higher sulphate levels resulted in calcium sulphate scale formation. More complex polymeric antiscalants were introduced to replace SHMP but still required acid dosing and were found to contribute a nutrient which increased microbial activity. In the 1990's phosphonate based antiscalants were successfully introduced and acid use could be significantly reduced or replaced. With the building of "mega" plant +100,000 m<sup>3</sup>/day operating at higher recoveries of 40-50% there is a much higher tendency for scaling and fouling.

In 1936, Wilfred Langelier developed a method for predicting the pH at which water is saturated in calcium carbonate. [1] Ryznar, Puckorius and others made later modifications to the LSI calculation. [2] In 1952 a paper on the Stiff and Davis saturation index was published which takes into account the "common ion" effects of sea water on the driving force for scale formation. [3] The accuracy of predicting scale formation in sea water has been questioned more recently in work by R. Sheikholeslami who proposes an alternative saturation index. [4] There are some in the desalination industry that contest that SWRO membranes do not scale up at all.

The Dow FilmTec manual states "In RO plants operating on sea water with TDS in the range of 35,000 mg/l, scaling is not such a problem as in brackish water plants, because the recovery of sea water plants is limited by the osmotic pressure of the concentrate stream to 30-45%. However, for safety reasons we recommend using a scale inhibitor when operating above a recovery of 35%." [5] Accurate scaling prediction in first pass SWRO plant is difficult and the accepted industry view has been that it is safer and more efficient to operate plant with an antiscalant.

The concept of membrane autopsy is as old as membranes themselves, with the idea that the only way of knowing what is taking place inside the membrane is to cut it open and have a look. [6] In recent years new and improved technologies such as Fourier Transmission by Infrared (FTIR) and X-ray Photoelectron Spectroscopy (XPS) and affordability of techniques such as EDAX and SEM have enabled the autopsying chemist to be more accurate in their assessment. At the same time membrane prices have halved making it more acceptable to the user to provide elements for the destruction technique known as 'Membrane Autopsy'.

The author's present data from ninety-nine SWRO membrane autopsies carried out since 2002 detailing the frequency with which the most common foulants and scalants are found. The reasons for and mechanisms of deposition are explained. Conclusions are drawn on various chemical operational strategies and alternative strategies suggested.

## II. MEMBRANE AUTOPSY

In most cases, membranes are sent for autopsy because they have failed or are underperforming significantly. That is salt passage, flow, flux and pressure differentials are outside of the design specifications. The first stage of membrane autopsy is to record the element model, serial number, position in the plant along with the chemical treatment regimes being applied. External examinations and photographs are taken to record the physical condition. A vacuum test is conducted to assess physical membrane damage. Samples can be taken from the feed inlet and outlet. The outer casing is removed and membrane leaves unrolled in readiness for an internal examination of the membrane sheets, glue lines, vexar and permeate carrier. Representative deposit samples are taken from the membrane surface and photographs are taken. Membrane samples are cut and prepared for characterisation and cleaning tests in a specially



**Fig 1 Flat sheet test rig**

designed flat sheet test rig. [Fig 1] Flow rate and salt rejection are determined and compared to design specifications. Physical damage to the membrane samples is conducted using the methylene blue dye test and oxidation by halogens is tested using the Fujiwara test. The validity of the Fujiwara test has been questioned in some quarters so additional tests using attenuated total reflectance infrared spectroscopy (ATR/FTIR) is carried out. This can provide valuable information related to the chemical structure of membrane and deposits. In the mid-infrared, absorption of radiation is related to fundamental vibrations of the chemical bonds. Internal reflection spectrometry provides information related to the presence of specific functional groups which can determine the presence of halogens. Deposits are identified using elemental determination with the SEM-EDXA system. This is based on analysis of X-rays produced via electron beam excitation of a sample area. This technique allows analysis of a sample in selective areas. The limited depth of analysis (typically a few microns), and the possibility to select the area of interest under the electron beam, allows for local analysis to reveal differences in composition. The identification and measurement of individual peak intensities in the X-ray spectrum can be done with a computerized multichannel analyzer. In addition X-Ray diffraction is carried out to identify crystalline solids by measuring the characteristic spaces between layers of atoms or molecules in a crystal. Each crystalline solid has its unique characteristic X-ray powder pattern which may be used as a "fingerprint" for its identification. X-ray photoelectron spectroscopy analysis (XPS) or electron spectroscopy for chemical analysis (ESCA) is a quantitative spectroscopic technique that measures the empirical formula, chemical state and electronic state of the elements that exist within a material. XPS spectra are obtained by irradiating a material with a beam of X-rays while simultaneously measuring the kinetic energy and number of electrons that escape from the top 1 to 10 nm of the material being analyzed. When the nature of the deposits on the membrane surface is understood then several membrane samples are cleaned with a variety of cleaning solutions at different pH and temperature to assess the best method for cleaning the plant. A full cleaning protocol can then be recommended. The autopsy is written up as a complete report and forwarded to the customer.

### III. RESULTS OF 99 SWRO MEMBRANE AUTOPSIES

From 2002 to 2010 the Genesys Membrane Products Laboratory in Madrid has completed over 400 membrane autopsies. The results of ninety-nine sea water membrane autopsies are presented here. The membranes were sent to our laboratory from thirteen different countries with over half coming from Spain, 12% from UAE and 9% from Chile. Of the ninety-nine autopsies in this study thirty-six came from “mega plant” with over 50,000 m<sup>3</sup>/day of permeate production. These results are important to understand the operational issues with large SWRO plant, a sector which has shown rapid recent growth. Figure 2 and Table 1 show the principle findings of 99 autopsies which are discussed below along with techniques commonly adopted to prevent them.

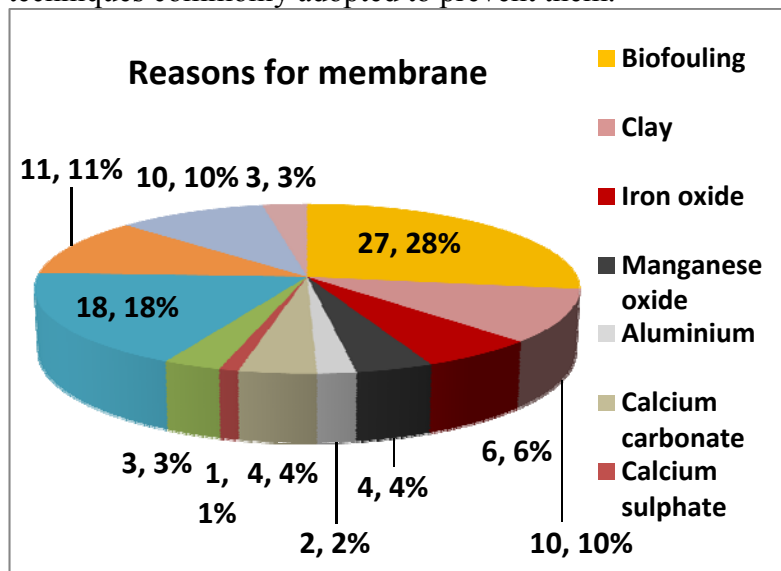


Fig 2 Common reasons for membrane failure

Principle Issues	Number
<b>Biofouling</b>	<b>27</b>
<b>Clay</b>	<b>10</b>
Iron oxide	6
Manganese oxide	4
Aluminium	2
<b>Metal oxides</b>	<b>12</b>
Calcium carbonate	4
Calcium sulphate	1
Silica	3
<b>Scales</b>	<b>8</b>
<b>Oxidation</b>	<b>18</b>
<b>Abrasion</b>	<b>11</b>
<b>Clean</b>	<b>10</b>
Undefined	3
<b>TOTAL</b>	<b>99</b>

Table 1 Autopsy findings

#### 3.1 Biofouling

The most common problem identified is biofouling which includes principally bacteria and associated biofilm but can include fungi, algae and even nematode worms. All membranes can support a viable biofilm. As a general rule as long as the biofilm is not exuding a protective polysaccharide film then it will be stable and will not adversely affect system performance.[7] The introduction of GRP piping has resulted in increased algal growth which is only stopped when the GRP is painted to prevent light. Good practice dictates that the nutrient supplies should be restricted as far as is practicable. This is generally done through a combination of filtration and biocide addition. Chlorination is the most common practice and although chlorine is effective in killing bacteria in free flowing water (planktonic bacteria), it is not effective against established biofilm in pipes and equipment surfaces upstream of the RO membranes. It is reported that the biofilm killing dose can be between 1000 - 1500 times as high as the planktonic killing dose due to protection from extracellular polymeric structures excreted by the bacteria. Bacteria grow back very quickly when free chlorine is lost [8]. This is attributed to the surviving bacteria feeding on the new nutrients from the chlorine degraded organic material.[9] The biocidal efficiency of free residual chlorine is directly related to the concentration of undissociated hypochlorous acid (HOCl), this is 100 x more effective than the hypochlorite ion OCl<sup>-</sup>.

Eg. At pH <5 HOCl = 100%  
 At pH 6.5 HOCl = 90%  
 At pH 7.5 HOCl = 50%  
 At pH >10 HOCl = 0 %

Dissociation of HOCl to OCl<sup>-</sup> increases with increasing pH. As most SWRO waters will have a pH of 7.5 to 8.3 for chlorination to be effective the feed pH needs to be reduced to 7.5 or below. There has been a trend towards intermittent chlorination which can vary from site to site from a daily or weekly cycle. More recently RO plant are operating successfully without any chlorination thus reducing the nutrient content of the feedwater reaching the membranes.

### 3.2 Oxidation

It is not surprising that the second most common problem identified by membrane autopsy is oxidation given the very common use of chlorination in SWRO plant. Free chlorine coming into contact with polyamide membranes will cause oxidation damage resulting in higher salt passage. For Dow FILMTEC™ RO polyamide membranes, degradation can occur after roughly 200-1,000 hours of exposure to 1 mg/L of free chlorine. Chlorine is easily removed by the addition of sodium metabisulphite (SMBS) to the feed water prior to contact with the membranes. Membrane oxidation occurs usually due to dosing pump failure of the SMBS, operator error or simply incorrect measurement of total and free chlorine reserve. Cellulose and tri-cellulose acetate (CA, TCA) membranes are resistant to chlorine but very susceptible to bacteria attack which use the membrane as a nutrient source. TCA membranes have been included in recent large plant designs like Shuqaiq, Rabigh and Yanbu in Saudi Arabia. This is to allow higher chlorine dosing in an attempt to cope with red and green algal blooms in the Red Sea. They tend to have continuous chlorination with neutralisation of free chlorine using SMBS prior to the membranes. Every eight hours the SMBS dosage is stopped to allow chlorinated water with 0.2 - 0.5mg/l of free chlorine to reach the membrane surface to help control bacteria growth.

Oxidation can occasionally occur following disinfection of pipe work with other oxidising agents such as peracetic acid and hydrogen peroxide or potassium permanganate. If the disinfecting solution reaches the membrane oxidation damage will result. The major difference between the chlorination chemistry of seawater and that of brackish water is the presence of bromide in seawater in concentrations of typically 65 mg/L. Bromide reacts rapidly with hypochlorous acid to form hypobromous acid.  $\text{Br}^- + \text{HOCl} \rightarrow \text{HOBr} + \text{Cl}^-$

Thus, in chlorinated seawater the biocide is predominantly HOBr rather than HOCl. Hypobromous acid then dissociates to hypobromite ion as follows:  $\text{HOBr} \leftrightarrow \text{OBr}^- + \text{H}^+$

HOBr dissociation is less than HOCl dissociation. At pH 8, where 72% of HOCl is dissociated, about 17% of HOBr is dissociated. In other words, effective treatment can be performed at a higher pH than in brackish water, where no bromide is present. The rate of membrane oxidation is catalysed by the presence of aluminium and transition metal ions.

### 3.3 Metal Oxides

Twelve percent of membranes autopsied show the principle reason for failure was the presence of metal oxides. These consist principally of iron oxide, manganese oxide and aluminium. Furthermore iron was detected (by SEM-EDX analysis) in 40% of the membranes tested. Pre-treatment inorganic coagulants are usually based on multivalent cations such as iron (ferric or ferrous) and aluminium salts. When in solution they form iron hydroxide which coagulates suspended solids into flocs which can be removed by the filter media[13]. These positively charged molecules interact with negatively charged particles to assist in charge aggregation. Many of these chemicals, under appropriate pH, temperature and salinity conditions, react with water to form insoluble hydroxides which, on precipitation, link together to form long chains or meshes, physically trapping small particles into the larger floc. Van der Waals attraction and/or the use of polymeric flocculants complete the process, forming larger aggregates which can be further flocculated or separated from the waste stream. Commonly used coagulants are Ferric chloride [FeCl<sub>3</sub>], Ferrous chloride [FeCl<sub>2</sub>], Ferric sulphate [Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>], Ferrous sulphate [FeSO<sub>4</sub>], Aluminium



chloride [ $\text{AlCl}_3$ ], Aluminium sulphate [alum,  $\text{Al}_2(\text{SO}_4)_3$ ], Polyaluminium chloride [PAC]. The Israeli Ministry of the Environment has voiced concerns that 3,000 tons of iron per year will be discharged into the sea as a result of coagulant use on desalination plants. Iron fouling has a damaging effect on both cartridge filters and membranes as shown in Fig 3 & Fig 4.



**Fig 3 Iron fouled cartridge filters**



**Fig 4 Failed membranes**

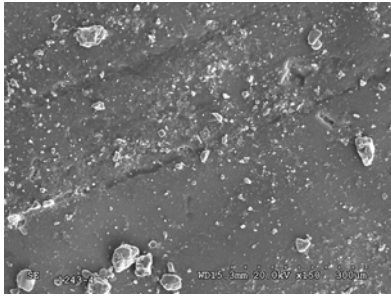


**Fig 5 Iron acrylate fouling**

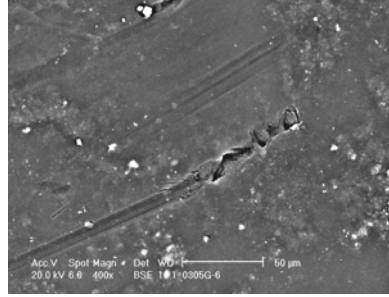
The use of phosphonate based antiscalants is common place in SWRO plant as they have the distinct advantage of sequestering iron and preventing deposition on the membrane surface. The choice of antiscalant is critical as polymer based products react with iron to form iron acrylate which blinds and irreparably damages the membrane as seen in Fig 5. The autopsy deposit analysis frequently showed small quantities of transition metals such as titanium, vanadium, chromium, manganese, cobalt, nickel, copper and molybdenum. It was reported by Murphy [10] that deacetylation (of CA membranes) was more effective in presence of cobalt and ruthenium, moderately in the presence of iron, nickel, rhodium, palladium and silver, and slightly in presence of copper and gold salts. The source of these metals is thought to be as a result of plant corrosion but may also be due to impurities found in iron salt coagulants. Work conducted by Dr. Nabil Nada of SWCC demonstrated an increased likelihood of membrane oxidation when using poor grade ferric chloride coagulant due to the presence of transition metals which catalysed the oxidation reaction.

### **3.4 Abrasion**

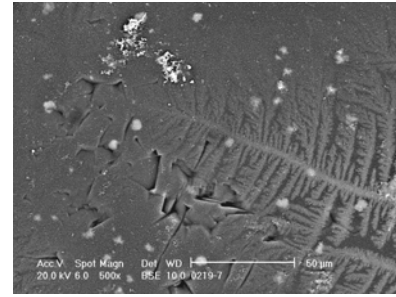
Due to the high operating pressures of sea water RO plant (50-70 bar) there is a high incidence of abrasion damage on the membrane surface. Damage can occur due to the feed spacer being pressed into the membrane due to excess pressure often as a result of fouling and/or scaling within the membrane element. Using scanning electron microscopy the marks left by the feed spacer can clearly be seen Fig 6. This results in increased salt passage, lower permeate quality and a higher than expected flow rate. Abrasion can also occur when particles are drawn across the membrane surface. The particles are often from the filter media which has been inadvertently washed on to the membranes during commissioning. Also metallic particles (corrosion by-products or welding particles) from pumping and pipes have been identified. Sand and anthracite particles are particularly damaging as shown in Fig 7. Poorly designed cleaning rigs and practices can contribute to membrane abrasion. Inadequate filtration of the cleaning solution results in the circulation of particulates during cleaning resulting in damage to the lead elements. Some algae can be less than 5 micron in size and pass through the cartridge filters. Their exoskeletons are made from calcite or silicon and can also cause abrasion of the membrane surface.



**Fig 6 Spacer indentation**



**Fig 7 Sand particle abrasion**



**Fig 8 Scale indentation**

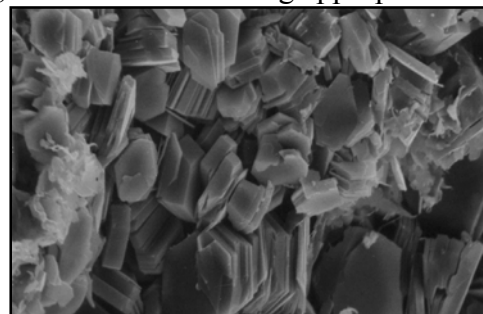
Figure 8 shows the indentations into the membrane surface left behind when calcium carbonate scale has been removed by an acidic clean. This demonstrates that even when cleaned successfully membranes may not work to their original design specification due to physical damage.

### 3.5 Clay (Alumino-silicates)

Alumino-silicate fouling was found in one third of the sea water membranes autopsied. Ten percent of membranes were failing primarily due to clay deposition [Fig 9]. Clay is the by-product of chemically weathered rocks and is ubiquitous in all waters around the world. Clay minerals are part of the Phyllosilicate (sheet structures) subclass of minerals. Structurally they are composed of tetrahedron rings linked by shared oxygen atoms to other rings in a two dimensional plane forming a sheet-like structure [Fig 10]. These sheets are connected to each other by weakly bonded layers of cations which often have water and other neutral atoms or molecules trapped between the sheets giving clay their soft compressible nature. The resultant crystal structure is flat and plate like. According to Swaddle [11] we refer to them as aluminosilicates when some of the  $\text{Si}^{4+}$  ions in the silicates structure are replaced by  $\text{Al}^{3+}$  ions. For each  $\text{Si}^{4+}$  ion replaced by an  $\text{Al}^{3+}$ , the charge must be balanced by having other positive ion such as iron, magnesium, alkali metals, alkaline earths and other cations. Armstrong [12] concluded that “The structural characteristics of clay minerals mean that operational increases in pressure can compress the deposit resulting in further loss of membrane flux. The test data supports the fact that clay deposits on a membrane surface can produce irreversible damage in terms of salt rejection due to abrasion processes. Improvements in pre-treatment can be considered although practical experience shows that small particulate/colloidal matter ( $< 2 \mu\text{m}$ ) cannot be completely removed from feed streams.” Clay is very difficult to remove completely from the feed stream through filtration. A build up is almost inevitable therefore regular cleaning with specialised clay cleaners is recommended. Clay fouling can be minimized by improving media filters, microfilters and using appropriate flocculants.



**Fig 9 Clay fouling on a membrane**

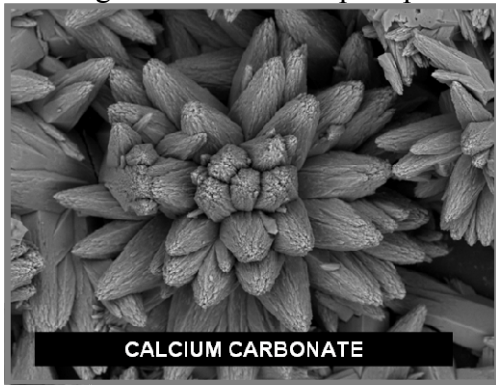


**Fig 10 Tetrahedron clay rings**

### 3.6 Mineral Scale

Mineral scale was identified as the major cause of membrane failure in eight percent of the membranes autopsied. However calcium and magnesium deposits were found in 21% of the membranes studied. The scaling species identified included most commonly calcium carbonate, then calcium sulphate and finally

silica. The lack of scale formation may be a result of the common ion effect in sea water significantly reducing the likelihood of precipitation of calcium salts.



**Fig 11 Calcium Carbonate**



**Fig 12 Calcium Sulphate scale**

More likely is the fact that almost all SWRO plant operate with the use of an antiscalant and acid dosing. The authors conclude that this is the real reason scale is not often found. Some desalters claim that SWRO membrane plant do not require the use of antiscalant, but this seems to contradict the advise of the membrane manufacturers and the best practice on the vast majority of plant in operation. The authors in a literature search and contact with plant operators failed to identify any significant plant that were operating without either acid or an antiscalant. The daily quantities in kg of 95% sulphuric acid required to reduce a sea water pH of 8 on very large plants is shown in Table 2. This is based on a standard sea water chemistry and plant details as shown in Table 3. This data correlates well with actual operational use quantities.

Plant Size MLD	pH 6.5	pH 7	pH 7.5
100	11,537kg	4,725kg	1,762kg
200	23,075kg	9,450kg	3,525kg
300	34,612kg	14,175kg	5,288kg

**Table 2: Daily Sulphuric acid consumption to reduce pH**

Ions	mg/l	Plant details	
Calcium	500	1st Pass	SWRO
Magnesium	1,500	Pressure	60-75 bar
Sodium	10,000	Temperature	30°C
Sulphate	2,500	pH Sea Water	8.0
Chloride	20,000	Recovery Rate	40%
Bicarbonate	200		

**Table 3: Sea water chemistry and plant details**

At the Larnaca SWRO plant Koutsakos demonstrated that the first pass SWRO plant could operate at the natural sea water pH of 8.2 and an S&DSI of 0.9 to 1.3. Acid dosing was removed and a phosphonate based antiscalant dosed. It was commented that “The trial was initiated in March 2007 and successfully completed 12 months later with no evidence of scaling based on the historical process data of the plant.” The authors added further that the plant “has lead the way in operating for a number of years now at normal sea water feed pH (pH 8.2) by suppressing the acid injection.”[14] The removal of acid dosing did not have an adverse effect on the pre treatment efficiency and the higher operating pH had the positive impact of increased boron rejection which meant the second pass elevated pH brackish water



plant was no longer required for half the year. A study by Hassan et al concluded that “Dosing of acid in seawater ahead of the filtration step has no major effect on filtrate SDI when compared to the SDI of non-acidified filtrates.” [15] There seems to be a body of evidence building that acidification of the feed water could be significantly reduced or replaced without impacting the effectiveness of iron coagulants and the antiscalant performance.

#### **IV CONCLUSIONS**

The results of SWRO membrane autopsies presented here specifically identifies the most common reasons for membrane failure. Regardless of membrane type it has been common practice on SWRO plant to chlorinate the feed water, dose sulphuric acid and an iron salt coagulant. These common practices are now being questioned in an attempt to further reduce membrane fouling, cleaning frequency and extend the lifespan of the membranes.

The authors have shown the most common problems in the membranes autopsied were in decreasing order: biofouling, oxidation, metal oxide fouling, abrasion, clay, mineral scale.

In trying to combat these problems with the use of commodity chemicals it would appear that additional problems or side effects can result. The important findings are:

- Chlorination can result in additional nutrient for bacteria and biofilm growth on the membrane surface.
- Chlorination increases the likelihood of polyamide membrane oxidation.
- Iron and aluminium salt coagulants foul cartridge filters and membranes.
- Sulphuric acid and iron chloride can contain transition metal impurities that at low levels on the membrane help catalyse oxidation.
- Reducing the pH with sulphuric acid aids the coagulation process for aluminium salts but has little effect on iron salts.
- Sulphuric acid may not be required in addition to a phosphonate antiscalant.
- Sulphuric acid dosing increase the product water TDS and boron levels.
- A polyelectrolyte flocculant could be dosed in order to reduce or replace an iron salt coagulant.

Further studies are required to look at SWRO plants that operate successfully with reduced or without commodity chemicals. The reliance on mass dosage of chlorine, acid and iron salts is being questioned by plant operators and designers alike. The result of autopsies on SWRO membranes gives a useful insight into areas of future study to reduce the use commodity chemicals.

#### **ACKNOWLEDGEMENT**

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