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Autopsy of an old reverse osmosis membrane from Cap Djenet seawater desalination plant: case study of Algeria

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ABSTRACT/RESUME

Abstract: *In Algeria, as many other southern Mediterranean countries, the water's resources remain limited. Today there is an increasing demand of drinking water and water requirements for irrigation and industry. Therefore, if we consider the saline water potential along the 1600Km coast, using reverse osomosis R.O process, the seawater desalination has been promoted such as an efficient alternative to provide security for drinking water. However, scaling and fouling are the most serious problems in the efficient operation of reverse osmosis systems and may leads to a loss of membrane performance then the necessary to replace the membranes. Therefore, in order to prolong its life and reduce the costs of producing drinking water, it is necessary to study the phenomenon of wear of the membrane using chemical analysis and membrane autopsy to analyze reverse osmosis fouling elements. Our work investigates phenomenon of wear of the membrane of the Cap Djenet seawater desalination plant. We focus on reverse osmosis membrane autopsy including an exhaustive study of the surface of the membrane and of the food-rejection spacer. We carried out several techniques including a chemical and microbiological analysis of the deposit which covered the surface of the membrane, tests to determine chemical oxidation (Fujiwara test), Tests with Scanning Electron Microscopy (SEM) with elemental analysis by X-ray Dispersive Energy (EDX) to study the atomic composition of fouling and an analysis of Fourier Transform Infrared (FTIR-ATR). The results obtained from the different analyses carried out on the autopsied elements indicate that the membrane from 1st position shows a high presence of fouling. The fouling on membranes show mainly characteristics: a mixture of protein derivatives related to biofilm, sodium chloride, iron, aluminosilicates and other elements commonly detected on sea water membrane fouling were identified. Due to the presence of fouling, membrane from first position showed a lower permeate flux than reference values. Considering these results, a review of plant pre-treatment and disinfection should be carried out in order to avoid the presence of the fouling components identified during the autopsy.*

I. Introduction

Since two decades, as many other southern Mediterranean countries, the hydrologic situation in Algeria faced with a real problem: provide drinking water to inhabitants.

In Algeria, the water's resources remain limited and the country is threatened by scarcity by early 2020. Two of the important factors exacerbating this issue is climatic change and the concentration of industrial and agricultural activities. The irregular rainfall oscillates only between 100 and 600 mm / year and the long period of droughts let to over exploitation of the water reserves until a large part of them has been exhausted[1].

Nevertheless, today there is an increasing demand of drinking water and water requirements for irrigation and industry. Therefore, if we consider the saline water potential along the 1600Km coast, the seawater desalination has been promoted such as an efficient alternative to provide security for drinking water in the main cities of northern Algeria[2,3].

Among processes for seawater desalination, the two most common process are: i) distillation and ii) membrane separation[4,5]. Among the membrane separation processes, reverse osmosis (RO) is a frequently used process in desalination and progressive water treatment which meets the cost and productivity constraints of desalination plants [4]. However, scaling and fouling are the most serious problems in the efficient operation of reverse osmosis systems. For instance, fouling leads to a loss of membrane performance such as increase of pressure drop to a point where it may become necessary to replace the membranes[6].

Under the operating conditions of the seawater desalination plants, in the absence of good pre-treatment, the membranes are subjected to an accelerated clopping. Even if the pre-treatment exist, it is often very perfunctory and induces a lifetime of membranes which does not exceed five years.

Therefore, in order to prolong its life and reduce the costs of producing drinking water, it is necessary to study the phenomenon of wear of the membrane using chemical analysis and membrane autopsy to analyze R.O fouling elements.

The objective of the autopsy was to know the state and degree of fouling of the membrane to determine the need for chemical cleaning. It is common for the surface of the membranes and the spacer materials to become dirty, causing a decrease in the rejection of salts, a change in the characteristics of the production of permeated water and an increase in the differential pressure. There may also be the situation of increased product flow and an increase in the passage of salts, caused by oxidation or abrasion of the surface of the membrane. The most common soiling is caused by biofouling, organic matter, inorganic incrustations, material of colloidal origin or physical or chemical degradation of the surface of the membrane.

In our study, according the methodology described byPontie et al.[7,8], we performed the autopsy of the used membrane of the Cap Djenet seawater desalination plant in order to consider a second life of the used reverse osmosis membrane [9,10].

Our work focus on reverse osmosis membrane autopsy including an exhaustive study of the surface of the membrane and of the food-rejection spacer.

We carried out several techniques including a chemical and microbiological analysis of the deposit which covered the surface of the membrane, tests to determine chemical oxidation (Fujiwara test), Tests with Scanning Electron Microscopy (SEM) with elemental analysis by X-ray Dispersive Energy (EDX) to study the atomic composition of fouling and an analysis of Fourier Transform Infrared (FTIR-ATR).

II. Materials and methods

II.1. Seawater desalination unit

The seawater desalination plant is located on the Boumerdes city in Northern east of Algeria to provide the drinking water for the inhabitants. This unit started to run in 2012 operating continuously with nominal capacity of 100.000m^3/day, a conversion rate of 45% and a feed water salinity between 35 and 40 g/L. In our study, the membranes located in first position (DOW FILMTEC SW30 HRLE-400i, serial number F4810019) and was analyzed.

As shown the figure 1, the desalination plant consists of four major components: *i) high pressure pump; ii) RO membranes; iii) pump booster; iv) pressure exchanger*.

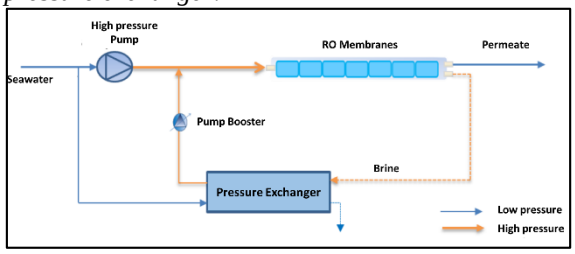


Figure 1. *Process schemes of the CAP djenet desalination plant.*

II.2. Seawater analysis

The Table 1 described the analysis results of the seawater used as feed to the reverse osmosis (RO) plant. For instance, the analysis revealed seawater consists of about 35 g/L - 39 g/L total dissolved solids. It presents a variable concentration of Sodium (Na) and Clorides (Cl) reaching

respectively 12051 mg/L and 22115 mg/L. The feed water temperature varied between 14°C and 27 °C acording season.

Table 1. *Analysis of seawater of Cap Djenet plant*

Parameters	Result	Units	Method
Turbidity	0,3	UNF	Nephelometry
pH/T^a	8,0/23,2	u.pH/ºC	Electrometry
Conductivity	56.400	µS/cm	Electrometry
TDS	39.654	mg/L	Calculated
Total Hardness	7.261	mg/L $CaCO_3$	Calculated
Calcium	450,2	mg/L Ca^{2+}	Volumetry
Magnesium	1.491	mg/L Mg^{2+}	Volumetry
Sodium	12.051	mg/L Na^{+}	I.Chromatography
Potassium	333,5	mg/L K	I.Chromatography
Total Iron	0,13	mg/L Fe^{3+}	Colorimetry
Aluminium	<0,008	mg/L Al^{3+}	Colorimetry
Total Manganese	0,094	µg/L Mn	Colorimetry
Sulphates	3.035	mg/L SO_4^{-2}	I.Chromatography
Chlorides	22.115	mg/L Cl^{-}	I.Chromatography
Fluorides	0,22	mg/L F^{-}	Electrometry
TA	0	ºF	Volumetry
TAC	13,2	ºF	Volumetry
Bicarbonates	161	mg/L HCO_3^{-}	Volumetry
Carbonates	0	mg/L CO_3^{-2}	Volumetry
Hydroxides	0	mg/L OH^{-}	Volumetry
Nitrates	15,3	mg/L NO_3^{-}	I.Chromatography
Sílica	1,4	mg/L SiO_2	Colorimetry
o-Phosphates	<0,01	mg/L PO_4^{-3}	Colorimetry

II.2.1. Membrane cleaning

In Cap djenet plant, among many methods of cleaning (e.g. physical and chemical), the cleaning process is mainly chemical.
At this time, our periodic membrane cleaning used only alkaline cleaners to remove organic fouling including biological matter.
Our cleaning process follow 6 steps: 1) Preparing the cleaning solution; 2) Pumping the mixed cleaning solution to the vessel in the condition of low rate and low pressure to displace the process water; 3) During cleaning to follow the solution behavior and to adjust some parameters (Temperature and pH); 4) Turning the pump off and allowing the elements to soak; 5)High-flow pumping: Feed the cleaning solution for 30–60 min. Cleaning is started when the pressure drop increases by 15%; 6) Flushing out the cleaning solution using the RO permeate.

II.3. Membrane autopsy

In this work, we perfomed membrane autopsy, the only accurate method used to identify the different causes of poor membrane performance.
We removed a membrane element from desalination plant for destructive analyses[11].
In order to determine the nature of membrane foulant present on the membrane surface, we caried out several analytical techniques.
Reverse osmosis membrane autopsy includes an exhaustive study of the surface of the membrane and of the food-rejection spacer, a chemical and microbiological analysis of the deposit that covered the surface of the membrane.

II.3.1. Visual Inspection for Autopsied Membrane

In order to evaluate external and internal condition of the membrane, we perform an inspection of element for physical damage to the casing, deposits on outer casing, telescoping, scale on ends of element and dimensions. We inspected also the internal condition including extent of fouling, damage to the membrane surface, glue lines condition, spacer material condition and carrier material condition.
Analysis of fouling removed from membrane surface is very useful in order to distinguish membrane components from fouling components and because it is a way to concentrate fouling components and to have a more accurate identification of it.

II.3.2.Analysis of organic compounds

Organic content of foulant was estimated by the loss on ignition (LOI) method. Loss on Ignition-Loss on ignition (LOI) is a common and widely used method to estimate the organic content of foulant. Organic matter is oxidised at 500–550 °C to carbon dioxide and ash. The weight loss during this reaction is easily measured by weighing the dry sample (LOI 105 ºC) before and after heating and is closely correlated to the organic matter.

II.3.3. Chemical analysis

Scanning Electron Microscopy – Energy Dispersive X-ray analysis (SEM-EDX): it is the most powerful analytical tool to get an of fouling composition. Energy Dispersive X-ray Analysis (SEM-EDX) 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 theability to select a very specific area of interest, allows for local analysis to reveal differences in composition. The identification and measurement of individual peak intensities in the

X-ray spectrum is done with a computerized multichannel analyser.
SEM-EDXis used in our study to*i)*get a general view and in detail of membrane surface (extension of fouling, abrasion marks, etc.) and *ii)* get an elementaldetermination of fouling composition.
In addition, we used Infrarred spectroscopy (ATR/FTIR) which identifying characteristic functional groups in order to identify of both organic and inorganic components of fouling can be achieved.

II.3.4. Microbiological analysis
Due to continuous operation of membranes with water, presence of certain microorganisms on membranes surface is expected (aerobic bacteria for example). Four characteristic microbiological parameters are quantified in order to get an approach to microbiological charge on membrane surface: i) Aerobic Bacteria; Sulphite-Reducing Bacteria; Pseudomonas Sp.; Moulds and yeasts

II.3.5. Membrane integrity
A number of tests were carried out in order to check for physical and chemical damage to the membrane. Methylene Blue test were performed to detect if the membrane is damaged, then Fujiwara Test were used to detect significant levels of polyhalogen compounds.
In addition, we carried out Infrarred spectroscopy (ATR/FTIR) analyze to study polyamide bands conditions on a clean membrane surface to detect structural damages/oxidation. Internal reflection spectrometry provides information related to the presence or absence of specific functional groups. Attenuated Total Reflectance Infrared (ATR/IR) Spectrometry can provide valuable information for the chemical structure of the membrane, so it is very useful in order to detect possible structural damages on membrane composition.
Commonly, the identification of polyamide layer damage by IR, must consider the following bands:
- Amide II N-H bend: 1540 cm-1
- C=C ring vibrations: near 1610 and 1448 cm-1
Intensity of these bands on membrane sample must be compared to a membrane blank in order to check if there is any significant change on membrane sample.
In order to check polyamide layer integrity of the autopsied membrane, a mechanical cleaning of membrane surface was carried out. IR spectra obtained from first position clean surface are compared to a membrane blank spectrum at figures 2 to 3.

III. Results and discussion

III.1. Visual Inspection of the membrane
III.1.1. Externel inspection

For autopsy, one membrane in the first position was removed: one fouled membrane from seawater plant used for five years. A second new membrane was analyzed as a reference.
The table 2 describe both external and internal inspection of the fouled membrane and reference membrane.
This visual external inspection of fouled membrane shows:
- some fissures were detected on external housing (feed end).
- presence of some particles which was observed on element feed end (photographs 3 and 5).
- no telescoping or significant spacer protrusion was detected.

Table 2. *External and internal inspection of the fouled membrane and reference membrane*

Reference Position and serial number s/n	***GA16035 first Position s/n F4810019***	***Memrane model sw30 HRLE-400i***
External inspection		***Reference values***
External housing integrity	*fissure on feed side*	*OK*
weight	*17,8 kg*	*13kg*
presence of particules / deposits	*A few on feed side*	*NO*
ATD condition	*OK*	*OK*
PWT condition	*OK*	*OK*
ATD central positioning membrane feed side	*OK*	*OK*
ATD central positioning membrane Rejection side	*OK*	*OK*
PWT protrusion test	*OK*	*OK*
Internal inspection		***Internal inspection***
Odour	*Absence*	*Absence*
Presence of fouling/ particules	*Presence of fouling*	*Absence*
failures on membrane surface	*Absence*	*Absence*
Spacer prints	*Absence*	*Absence*
Spacer material condition	*Presence of fouling on feed side*	*good absence of fouling*
membrane colour	*white*	*white*
ATD: Antitelescoping devices *PWT: Product water tube*		

III.1.2. Internal inspection

This visual internal inspection of fouled membrane shows:

- Presence of brown fouling was detected all over the surface. Same kind of fouling was observed on spacer material, mainly on feed side.
- Presence of a black mass that at first glance appeared like a bacterial colony. A similar results were observed by previous studies [12,13].Some studies suggest that this phenomen was ascribed to the foulants which have been deposited on the membrane surface rather than formed by a precipitation mechanism [14].
- No additional details were observed on membrane surface. No significant failures were detected.

III.2. Investigation of the membrane surface and foulant

III.2.1. Organic compounds

The Figure 3 describes LOI data obtained for GA160355 (1st position) fouling data. Considering these results, fouling from GA160355 membrane surface shows similar organic and inorganic content.

(a)

(b)

Figure 2. *Fouled membrane of cap Djenet desalination plant:* visual external inspection (a); visual internal inspection (b).

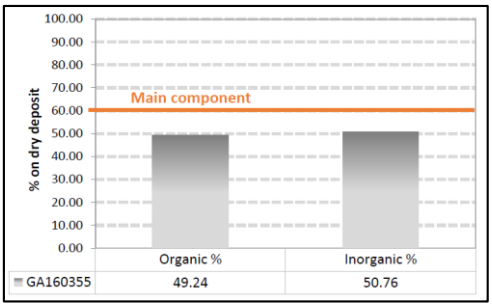


Figure 3.*Loi results obtained from GA160355 membrane fouling*

III.2.2. Investigation of the membrane surface and foulant by SEM/EDS- Chemical results

III.2.2.1. SEM results

A) Foulant (The deposit)

Analysis of fouling removed from membrane surface is very useful in order to distinguish membrane components from fouling components and because it is a way to concentrate fouling components and to have a more accurate identification of it.

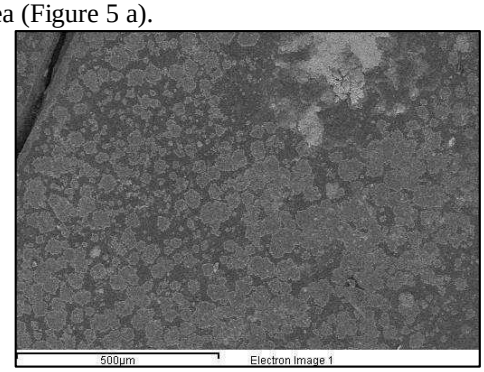
Thus, a sample of fouling from GA160355 membrane surface was taken for analysis by this technique. Also particles from GA160355 feed end membrane was analyzed.

TheFigure 4describes the different microphotographs and spectra obtained during these analyses and the observations achieved for each sample.

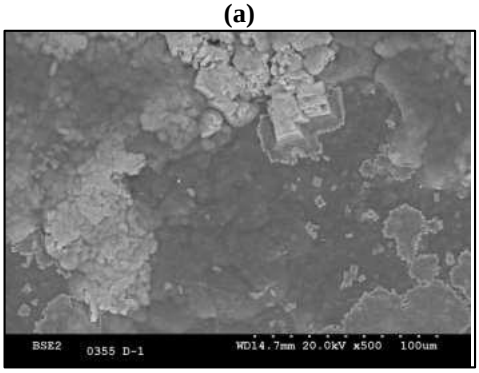
The SEM micrograph of fouling from membrane surface shows an organic fouling mixed with sodium chlorine from sea water.
There is also presence of aluminosilicates and common elements on sea water membranes (Figure 4 (a) and (b)).
The SEM Microphotograph 2F of membrane fouling shows that fouling looks organic with presence of inorganic crystalline structures (Figure 4 (b)).
The SEM micrograph of particules from feed end showed grains with presence of organic matter, sodium chloride, silica and metallic components.
They are probably fouled silica grains (Figure 4 (c))

B) Membrane Surface (Fouled membrane)

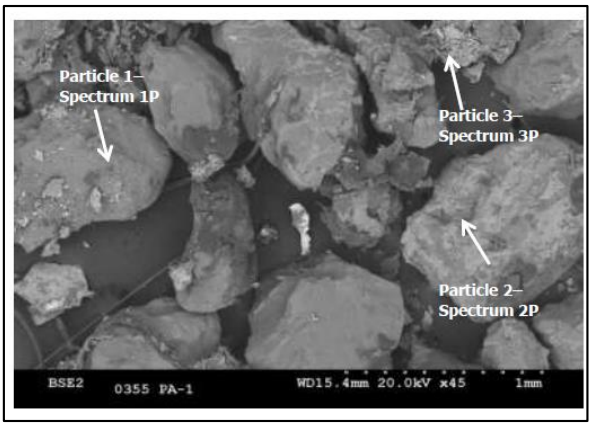
Figure 5 describres the different microphotographs and spectra obtained during thefouled membrane analyses and the observations achieved for each sample.
As the images shown, membrane surface is covered by thin organic fouling that didn't allow distinguishing membrane structure. On this thin organic covering, there is presence of sodium chloride dendritic growing characteristic of seawater membranes surface and some particles of different composition were detected also.
General analysis of this membrane surface sample indicates that there is a main presence of sodium chloride, aluminosilicates and small percentages of elements that are common also on sea water fouled membranes: magnesium, calcium, etc.
The general view of GA160355 membrane surface showed that there is a significant covering of membrane surface which shows inorganic component more concentrated on spacer support area (Figure 5 a).



(a)



(b)



(c)

Figure 4. *SEM Microphotograph of membrane fouling; Microphotograph 1F- General view of GA160355 (1st position) fouling from membrane surface– General analysis: Spectrum 1F (a); Microphotograph 2F. Detail of GA160355 (1st position) membrane fouling: Spectrum 2F (b); Microphotograph 1 General view of Particules (c).*

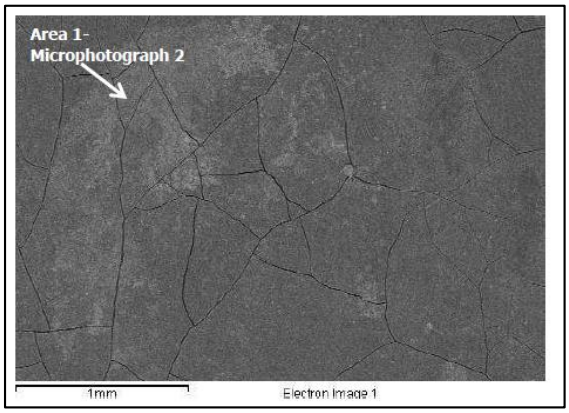
The second SEM micrograph of the surface of the fouled membrane (Figure 5 b) showed the detail of fouling on area 1 – spacer support area Inorganic component of fouling is concentrated on spacer support area.
The third SEM microphotograph of the surface of the fouled membrane (Figure 5 c) showed the detail of fouling on spacer support area. The figure 5c shows that fouling is composed by a mixture of an organic component with very small particles and presence of microstructures.
The last SEM microphotograph of the surface of the fouled membrane (Figure 5 d) showed the detail of membrane surface at high magnifications on area with thin organic covering. It's revealed that on none of the areas, membrane structure couldn't be distinguished. There is also presence of microstructures.

III.2.2.2. EDS results

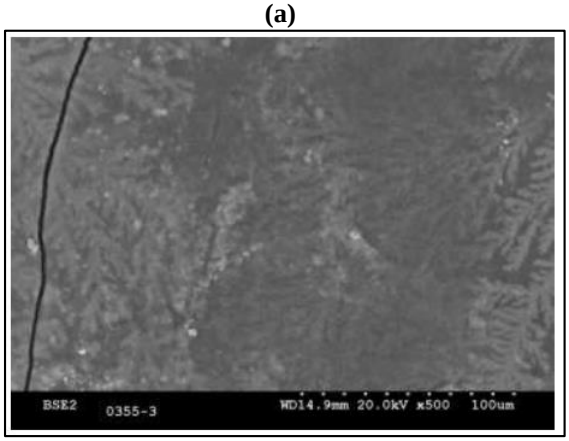
A) **Foulant (The deposit)**

The EDS analysis of the deposit are presented in the figure 6 and figure 7.
The general analysis of GA160355 membrane fouling indicates that fouling is composed by an organic component, sodium chloride, aluminosilicates, calcium, and common elements on sea water membranes fouling as sulphur, magnesium, phosphorous and potassium (Figure 6).
While the analysis of area 1 on GA160355 membrane fouling indicates that fouling is composed by an organic component with chlorine, sodium chloride small percentages of the rest of elements detected on the general analysis carried out on membrane surface sample (Figure 7).
The EDS analysis of the particlue in the deposit are presented in the figure 8, 9 and 10.

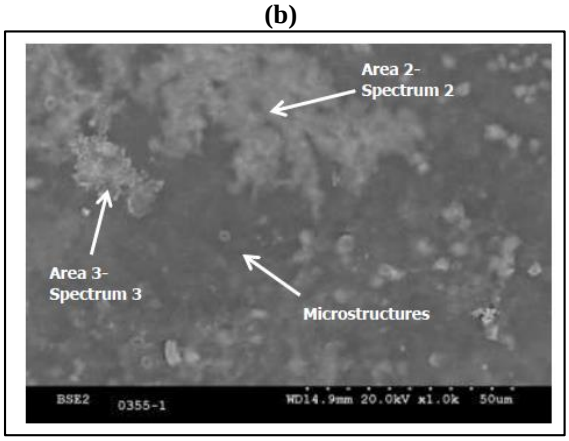
The analysis of particle 1 from GA 160355 showed the major constituents to be sodium chloride and silica (Figure 8).



(a)



(b)



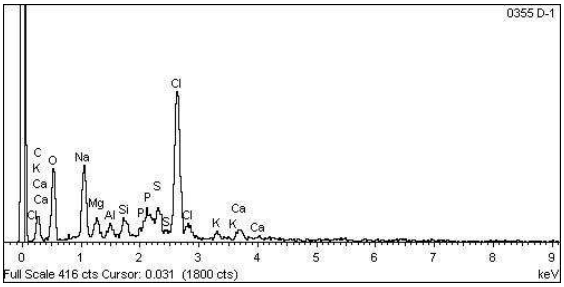
(c)

(d)

Figure 5. *SEM Microphotograph of Membrane Surface; Microphotograph. 1 General view of GA160355 membrane surface (a); Microphotograph. 2 Detail of fouling on area 1 (b); Microphotograph. 3 Detail of fouling on spacer support area (c); Microphotograph 4.- Detail of membrane surface at high magnifications on area with thin organic covering (d)*

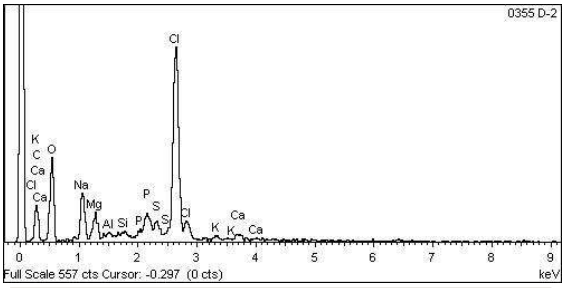
The analysis of particle 2 from GA 160355 is mainly composedof silica, with small percentages of sodium chloride and iron and aluminosilicates (Figure 9).

While the last particule 3, metallic particle was composed of titanium with small percentages of chromium and iron. We found also presence of sodium chloride, calcium phosphorous, magnesium (Figure 10).



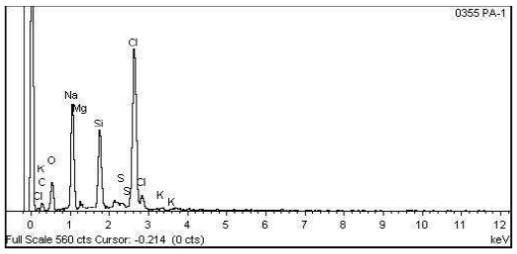
Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	3.30	0.1310	31.59	7.24	42.85
O K	10.53	0.3180	41.47	4.60	42.23
Na K	4.14	0.6705	7.75	0.94	5.49
Mg K	0.73	0.5845	1.57	0.29	1.05
Al K	0.40	0.6968	0.71	0.18	0.43
Si K	0.72	0.7995	1.14	0.20	0.66
P K	0.28	1.1961	0.29	0.24	0.15
S K	1.22	0.7989	1.92	0.34	0.98
Cl K	6.84	0.7157	11.98	1.35	5.51
K K	0.35	0.9006	0.49	0.16	0.20
Ca K	0.77	0.8745	1.10	0.21	0.45

Figure 6*-General analysis of GA160355 membrane fouling (Spectrum 1F)*



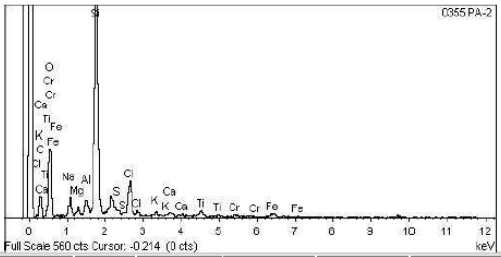
Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	6.74	0.1388	38.47	5.10	50.09
O K	14.64	0.2908	39.81	3.48	38.92
Na K	3.55	0.6612	4.26	0.44	2.89
Mg K	1.16	0.6140	1.49	0.20	0.96
Al K	0.17	0.7232	0.19	0.09	0.11
Si K	0.17	0.8270	0.16	0.09	0.09
P K	0.88	1.2392	0.56	0.16	0.28
S K	1.18	0.8175	1.14	0.19	0.56
Cl K	12.05	0.7295	13.07	1.14	5.77
K K	0.37	0.9002	0.33	0.09	0.13
Ca K	0.57	0.8736	0.51	0.11	0.20

Figure 7*. Analysis of area 1 on GA160355 membrane fouling (Spectrum 2F)*



Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	5.02	0.0880	30.37	5.78	44.55
O K	13.61	0.2776	26.07	2.74	28.71
Na K	21.82	0.7900	14.69	1.34	11.26
Mg K	0.88	0.5852	0.80	0.20	0.58
Si K	11.05	0.8193	7.18	0.68	4.50
S K	0.86	0.7780	0.59	0.20	0.32
Cl K	26.64	0.7102	19.96	1.76	9.92
K K	0.57	0.8668	0.35	0.12	0.16

Figure 8. *Analysis of particle 1 from GA160355 membrane feed end*



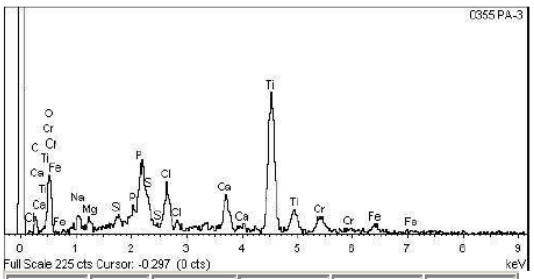
Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	5.16	0.1502	29.69	7.11	40.42
O K	17.93	0.3498	44.28	4.66	45.25
Na K	1.50	0.6498	2.00	0.32	1.42
Mg K	0.42	0.6319	0.57	0.15	0.38
Al K	0.88	0.7526	1.01	0.18	0.61
Si K	15.15	0.8365	15.65	1.65	9.11
S K	0.41	0.7141	0.49	0.18	0.25
Cl K	2.73	0.6705	3.52	0.42	1.63
K K	0.35	0.9085	0.33	0.11	0.14
Ca K	0.31	0.8827	0.30	0.12	0.12
Ti K	0.63	0.7647	0.71	0.18	0.24
Cr K	0.33	0.7819	0.37	0.16	0.12
Fe K	0.98	0.7860	1.08	0.25	0.32

Spectrum 2P.- Analysis of particle 2 on GA 160355 membrane feed end. Mainly composed of silica, with small percentages of sodium chloride and iron and aluminosilicates

Figure 9. *Analysis of particle 2 from GA160355 membrane feed end*

B) **Membrane Surface (Fouled membrane)**

The EDS analysis of fouled membrane presented in the figure 11, 12 and 13.



Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	3.52	0.2269	18.21	3.05	29.13
O K	9.38	0.2490	44.28	3.16	53.19
Na K	1.04	0.5251	2.33	0.53	1.95
Mg K	0.45	0.5338	1.00	0.30	0.79
Si K	0.56	0.7841	0.84	0.25	0.58
P K	1.40	1.1947	1.37	0.46	0.85
S K	0.51	0.7979	0.75	0.41	0.45
Cl K	2.12	0.7401	3.37	0.40	1.83
Ca K	2.54	0.9800	3.05	0.37	1.46
Ti K	14.04	0.8063	20.47	1.41	8.21
Cr K	1.80	0.7807	2.71	0.49	1.00
Fe K	1.10	0.7968	1.62	0.46	0.56

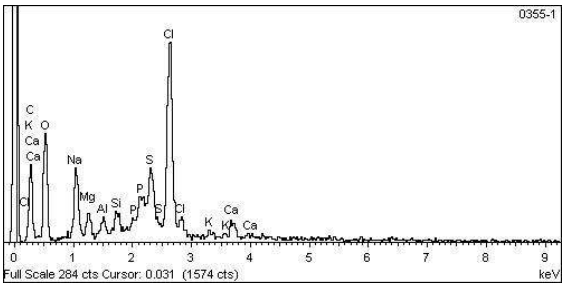
Figure 10. *Analysis of particle 3 from GA160355 membrane feed end*

The EDS analysis of membrane surface shows that the fouled membrane surface consisted of

membrane components (carbon, oxygen and sulphur), sodium chloride, chlorine, aluminosilicates and common elements on sea water membranes fouling (magnesium, calcium and potassium)(Figure 11).
Whereas, the EDS analysis of area 1 indicates that fouled membrane surface consisted of membrane components (carbon, oxygen and sulphur) and presence of sodium chloride and chlorine (Figure 12). The EDS Analysis of area 2shows that fouled membrane surface consisted of Membrane components (carbon, oxygen and sulphur), sodium chloride and chlorine and common elements on sea water membranes fouling (Figure 13).

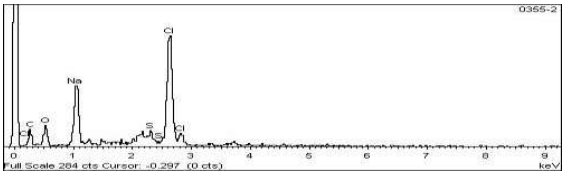
III.2.3. Microbiological count results

Due to continuous operation of membranes with water, presence of certain microorganisms on membranes surface is expected (aerobic bacteria for example). When presence of these microorganisms is very high, or when some specific microorganismsare detected on membrane surface, a direct relation to biofilm could be established.



Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	9.28	0.1832	46.64	3.81	57.71
O K	10.88	0.2711	36.92	2.87	34.30
Na K	2.33	0.6654	3.22	0.36	2.08
Mg K	0.58	0.6271	0.86	0.15	0.52
Al K	0.33	0.7405	0.41	0.11	0.23
Si K	0.58	0.8357	0.64	0.12	0.34
P K	0.54	1.2356	0.40	0.17	0.19
S K	1.91	0.8110	2.17	0.28	1.01
Cl K	6.04	0.7193	7.72	0.62	3.24
K K	0.25	0.9151	0.25	0.10	0.10
Ca K	0.75	0.8819	0.79	0.14	0.29

Figure 11. EDS General analysis of GA 160355 membrane surface.

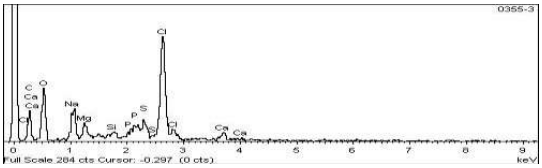


Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	6.56	0.1213	46.88	3.94	60.70
O K	7.12	0.2442	25.26	2.82	24.56
Na K	8.94	0.7484	10.35	0.96	7.00
S K	1.25	0.8312	1.31	0.30	0.63
Cl K	13.75	0.7351	16.21	1.32	7.11

Figure 12. *EDS Analysis of area 1 on GA 160355 membrane surface.*

The Table 3 describes some parameters and some reference values that should be considered in order to identify presence of biofilm on membrane surface. Reference values included at this table are indicative according. For more accurate biofilm identification, additional parameters like presence of protein derivatives/polysaccharides should be considered.

Results obtained from GA160355 (1st position) membrane surface microbiological counts are included at the Table 3. Considering these results, there is just slightly presence of aerobic bacteria on both membranes, so no presence high enough to be related to membranes performance failures.



Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	9.26	0.1651	43.42	2.79	54.71
O K	13.88	0.2799	38.35	2.37	36.28
Na K	3.38	0.6620	3.95	0.41	2.60
Mg K	0.96	0.6169	1.21	0.21	0.75
Si K	0.27	0.8313	0.25	0.13	0.14
P K	0.72	1.2390	0.45	0.21	0.22
S K	1.53	0.8149	1.45	0.24	0.69
Cl K	9.38	0.7260	10.00	0.63	4.27
Ca K	1.05	0.8805	0.92	0.16	0.35

Figure 13. *EDS Analysis of area 2 on GA 160355 membrane surface.*

Table 3.*Microbiological counts obtained from autopsied membrane surface*

Parameter	GA160355-1st position (CFU/cm²)	GA160356-7th position (CFU/cm²)	Reference values for biofilms*
Aerobic Bacteria	94	4	10^5
Sulphite-Reducing Bacteria	< 1	< 1	Presence
Pseudomonae Sp.	< 1	< 1	Presence
Moulds and yeasts	< 1	< 1	Presence

* (CFU/ cm² = Colony Forming Units per cm²)

III.2.4. Integrity of fouled membrane

III.2.4.1.Methylene Blue Test

Methylene Blue test detects if the membrane is damaged. The test will be positive if dye presence (blue colour) is detected on the permeate side.

As shown in figure 14, the passage of dye was detected on GA160355 permeate side, which verifies that membrane has suffered some damage.

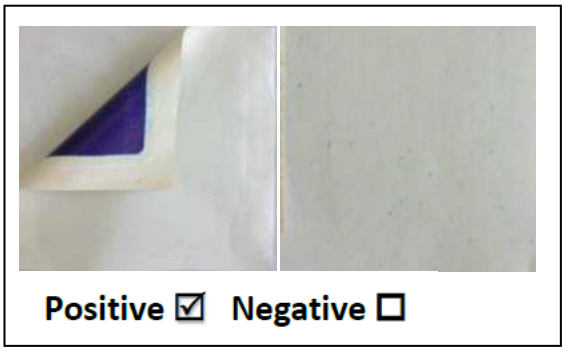


Figure 14. *Methylene Blue Test of used fouled membrane*

III.2.4.3. Fujiwara Test

Fujiwara Test (FJ) detects significant levels of polyhalogen compounds. This is a colorimetric test in which a pink colour in the analytical solution indicates organically bound halogens.

FJ test is always carried out on RO water rinsed membrane samples and without deposit (physically **removed).**

The result showed no pink color was observed on the analytical solution, so contact of fouled membrane GA160355 with halogens was not detected.

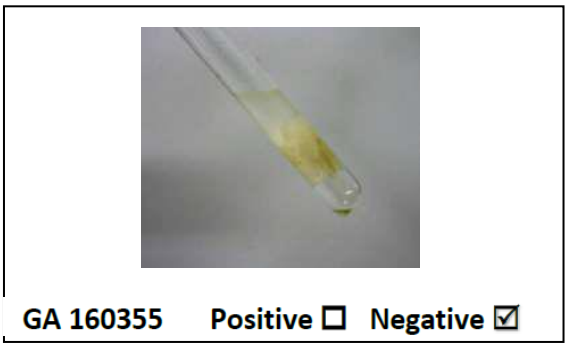


Figure 15. *Fujiwara Test of used fouled membrane*

III.2.4.2. ATR/FTIR Analyses results

As shown in Figure 16, surface spectrum still shows some fouling residues, which don't allow checking polyamide bands conditions.

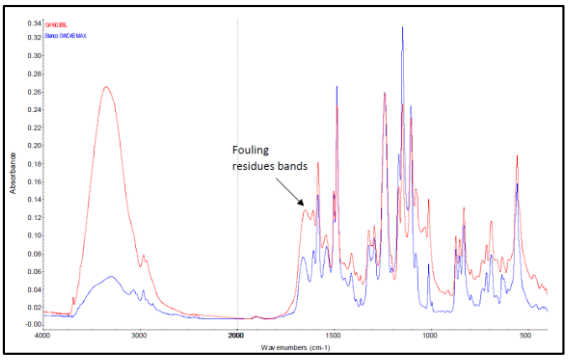


Figure 16.*Comparison of ga 160355 membrane ir spectrum after a mechanical cleaning (red line) compared to membrane blank (blue line).*

IV. Conclusion

Our work investigates phenomenon of wear of the membrane of the Cap Djenet seawater desalination plant. We focus on reverse osmosis membrane autopsy including an exhaustive study of the surface of the membrane and of the food-rejection spacer using several techniques.

In this study, we performed several technique to analyze the deposit and fouled membrane to provide a characterization of the RO fouled memebrane in fisrt position in seawater desalination plant after 5 years of operation.

The results obtained from the different analyses carried out on the autopsied elements indicate that the membrane from 1st position shows a high presence of fouling.

The fouling on membranes show mainly characteristics: a mixture of protein derivatives related to biofilm, sodium chloride from sea water, aluminosilicates and other elementscommonly detected on sea water membrane fouling were identified. Besides, presence of seawater microstructures was observed.

Due to the presence of fouling, membrane from 1st position showed a lower permeate flux than reference values.

Although some damage was detected on the membrane, this is not significant enough and salt rejection capabilities of the autopsied membranes are close to minimum value established by manufacturer.

Considering these results, a review of plant pre-treatment and disinfection should be carried out in order to avoid the presence of the fouling components identified during the autopsy.

Besides, cleaning procedures should be scheduled when membrane performance parameters change more than 10-15% from reference values in order to avoid membrane surface damage and fouling ageing or compaction, which can make much more difficult its removal.

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