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Techniques for UF and MF autopsies

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ABSTRACT

Autopsies on reverse osmosis (RO) elements have been carried out for many years. Although new techniques are regularly developed, the overall procedure is well established. Autopsies on ultrafiltration (UF) and micro filtration (MF) membranes are less common, but are increasingly being requested. While some of the techniques used on RO membranes can be carried over to UF, new techniques are required to fully understand the cause of the foulant and/or damage to the membranes. The paper outlines how an autopsy on a UF or MF membrane may differ from an RO membrane, and several case studies will be given to show the range of foulants that may be expected.

1. Introduction

Autopsies on reverse osmosis (RO) elements have been carried out for many years. Although new techniques are regularly developed, the overall procedure is well established. Some analytical procedures can carried out on both RO and UF/MF membranes, but a new procedure for performance testing is required, along with the development of certain test protocols on fibres harvested from a module.

2. Autopsy purposes

For the majority of autopsies, the need is set by a change in the product water quality or quantity. Providing a system is producing the required flow and quality, no intervention is seen as being required. Therefore, the majority of membrane autopsies are required to identify a decline in product quality or a decline in product flow.

The other reason for an autopsy is to assess the condition of a membrane following a pilot study. A typical study would involve operating the membrane under design conditions, backwashing (with or without chemicals) and carrying out standard chemical cleans. Examining the membrane after a pilot study identifies any foulants that have not been removed during the regular backwashing and cleaning, and assists in the prediction of membrane life.

3. Techniques—difference between RO and UF/MF

3.1. Performance test

The most significant difference between an RO membrane autopsy and that of a UF/MF module is the non-standardisation of UF/MF modules. The RO

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industry has standardized on 2.5´´, 4´´, 8´´ for the majority of applications, and more recently the development of 16´´ and 18´´ diameter. Membrane lengths are invariably 40´´ for anything other than very small systems. Test conditions for a membrane have standardized to a few different pressures and salinities with all standard tests taking place at 25˚C. Sodium chloride is the standard choice for salt, though nanofiltration membranes use either calcium chloride or magnesium sulphate. A normalized permeate flow rate is obtained using the standard test conditions.

MF/UF pressurized modules are invariably fed from the bottom. Product is taken from the top in the case of "in to out" systems or from a side port in the case of "out to in". Obtaining standard test conditions can be difficult, as the flux is often set by the fouling rate of the water, and the pressure varied to suit. Membrane manufacturers rarely put standard conditions on the specification sheets.

For that reason, each time a "new" membrane is received for autopsy, it is necessary to make modifications to the existing plant. Careful design has allowed the reuse of a set of valves and instrumentation, with only the local pipework between the valve array termination point and the membrane connections. A range of flux rates is measured at different pressures to determine flux rates, using a feed of ultrafiltered water, and the flux is normalized for temperature variations.

For an RO membrane, rejection is measured either using conductivity (where sodium chloride is used) or by analyzing a specific ion for divalent salts. Experience has shown that simply using conductivity to test an used membrane for rejection does not provide an accurate rejection figure, as there is often sodium and chloride held in the membrane. Extensive flushing can overcome this, but a more accurate figure will always be obtained by measuring feed and concentrate calcium (for calcium chloride) or sulphate (for magnesium sulphate).

Rejection is much harder to establish on an UF/MF membrane, as—particularly where further analysis is required—it is imperative that the membrane is not contaminated. Therefore, in most applications, a simple integrity test is performed to establish if the membrane has ruptured. This is performed by applying a pressurized air supply to the feed side of the membrane, isolating the supply and measuring any decline in air pressure. An intact membrane will hold pressure perfectly, any broken fibres will cause a drop in pressure.

Dow UF module under test, with Pall module in background

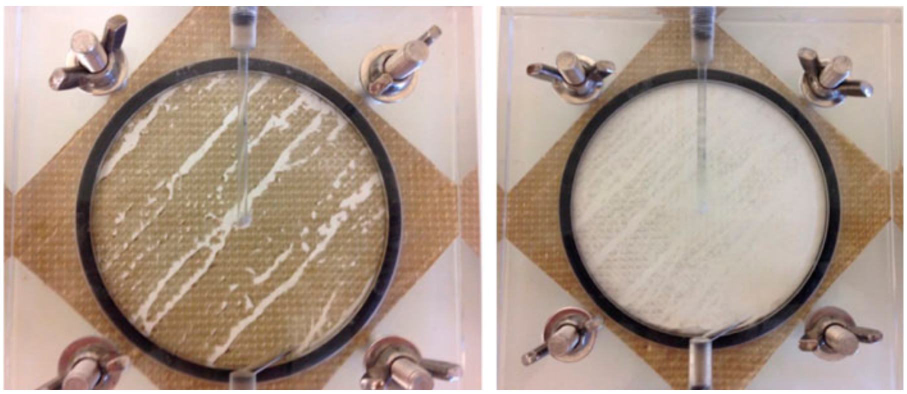
3.2. Analysis

Membrane surface analysis can be carried out on both UF/MF membranes and RO membranes. In general, the analytical techniques are the same. Because of the nature of the process, RO membranes are much more likely to have suffered inorganic scaling than that of an UF/MF membrane, though scale crystals have been seen to foul UF/MF, generally due to their presence in the feed water. Occasionally the presence of crystals has been traced to the use of alkaline cleaning chemicals coming into contact with hard water, precipitating calcium salts on the membrane surface.

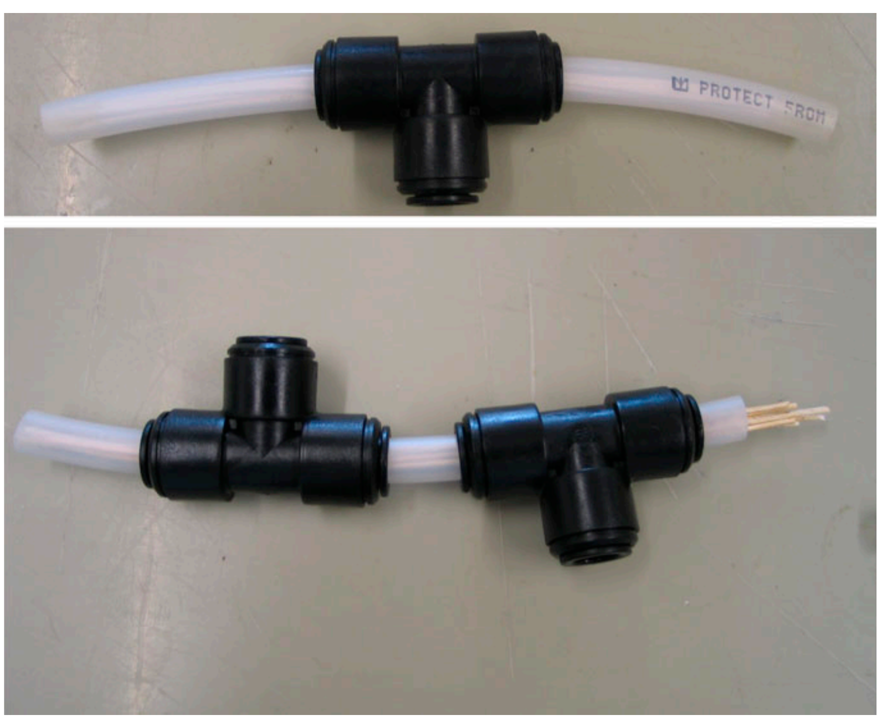
The most common techniques used are SEM/EDX to detect inorganic material, FTIR for organic breakdown and bacteriological analysis such as dip slides, ATP or gram staining. More recently, Avista Technologies have introduced chromatic elemental imaging (CEI), which gives a 3D effect to images allowing the presence of layers of foulants to be seen.

A common feature of an RO autopsy is the use of a cell tester. In a cell test, a sample of the membrane is removed from a leaf of the RO element and placed in a pressure device, such that feed water may be applied under pressure to the membrane sample. Permeate is collected from the underside of the membrane. In this way, it is possible to test the performance of a sample of membrane discreet from the whole element, and the performance of the membrane at different points along the leaf. Cell tests are also used to carry out cleaning studies, identifying the most appropriate cleaner for a particular foulant, and to dye test areas of a membrane to look for damage to the membrane surface.

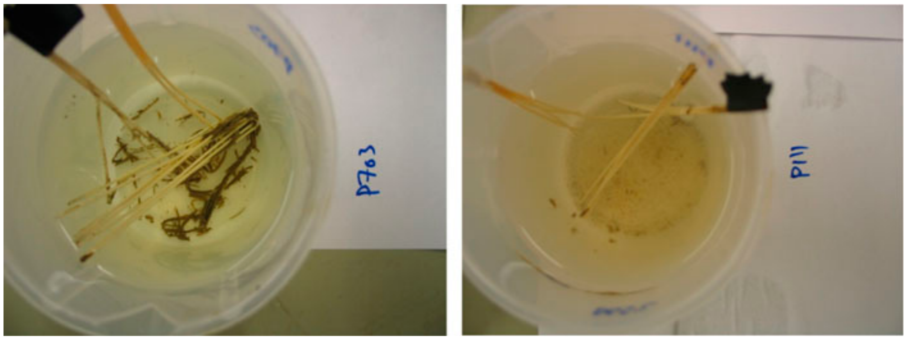
Similar results for hollow fibre membranes can be obtained by harvesting fibres from a fouled UF/MF



Membrane samples in a cell tester



Micromodules for UF fibre testing. Top picture shows an "in to out" module, bottom picture shows an "out to in" module



Soak tests with two of the cleaners studied

module, and building micro-modules with them. Fibres of 15–20 cm length are potted in plastic tubes, with pressure connectors fitted. Fibres may be taken from different positions along the module to identify any flux imbalance, which would represent different rates of fouling. The micro-modules may be used to carry out cleaning studies, enabling a range of different cleaners to be trialled on one module. Flux tests are performed before and after the clean, and the difference establishes the most effective cleaner.

4. Case studies

4.1. Cleaning study on fibres only

Fibres were received from a membrane manufacturer. The manufacturer had received the module back from a site, and the standard cleaning recommended had not successfully restored the flow of the module.

Initially, samples of the fibres were placed in beakers of cleaning solution and held at 35˚C for three hours. A visual inspection was made to identify which cleaners were most likely to remove the foulant.

Following the soak tests, micro-modules were produced to carry out flux tests. Each module was tested at a fixed pressure to obtain a flux rate. A cleaning solution was then passed through the module for one hour at 35˚C. Following this, the modules were rinsed and the flux rate was measured again.

	Cleaner A	
Parameter	Pre clean	Post clean
Perm. flow, ml/min	0.102	0.216

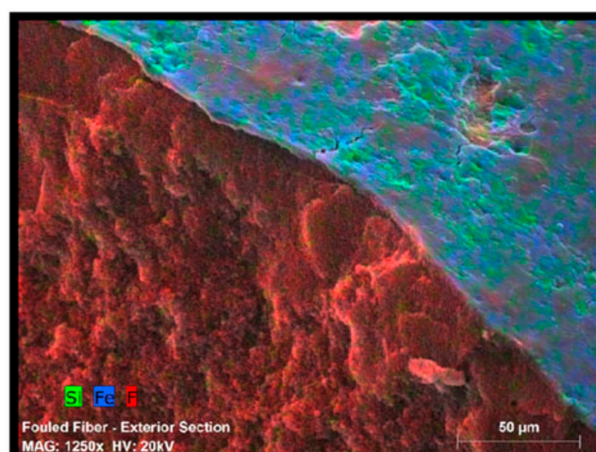
As can be seen above, the flux rate was increased by over 100%. The cleaner was then recommended for a site application, and similar results were achieved.

4.2. Module from UK power station

A UF module from a UK power station was sent for analysis. The module had been suffering from a decline in flux which was not recovered using the standard cleaning products recommended by the manufacturer.

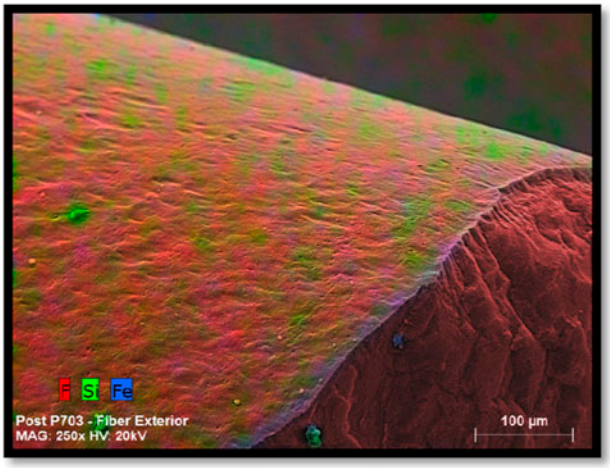
Upon opening the module, it was immediately apparent that there was a large amount of foulant present. The orange colour gave cause to assume the presence of iron.

CEI™ analysis was carried out on the fibres to confirm the foulant.



Using CEI, the foulant was confirmed to be mainly iron with some silicon also present. The element distribution clearly shows the presence of fluorine in the PVDF membrane, with the iron "coating" the outside of the fibres.

Soak tests on samples of the fibres showed RoClean P703 to be the most appropriate cleaner. Following the soak tests, a further CEI was carried out on the cleaned fibres to demonstrate foulant removal.



Some silicon remained, but the iron was almost entirely removed demonstrating the efficacy of the cleaner in this instance.

5. Summary

Many of the existing tests and procedures used on RO membranes can be used or adapted to assist in the troubleshooting of UF/MF membranes.