Membrane Autopsy

Scanning Electron Microscopy

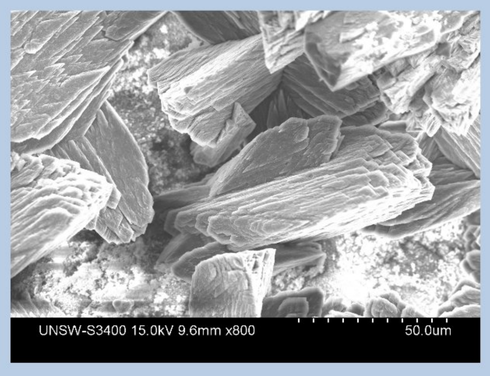
Scanning electron microscopy (SEM) is a microscopic method capable of producing very high magnification images of a membrane surface. Due to the manner in which the image is created, SEM images have a characteristic three-dimensional appearance and are useful for judging the surface structure of the sample.

SEM is useful for identifying scale and biological fouling. It can also be used for morphological identification of scaling material present.

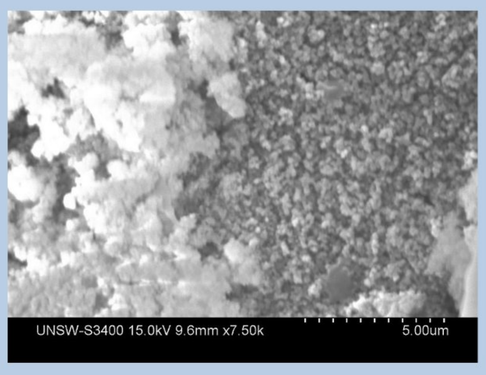
EDS (Energy dispersive x-ray spectroscopy) uses the x-rays emitted by the sample as it is exposed to the electron beam in the SEM to get the elemental information of the material being analysed.

The polymeric membrane materials will appear in the EDS spectra to a varying extent depending on the density of the sample. The EDS has a nominal penetration of 10 µm. Reverse osmosis membranes typically consist of a 0.2 μm layer of a polyamide over a microporous 40 μm polysulfone support whose molecular formula is $C_{27}H_{22}O_4S$.

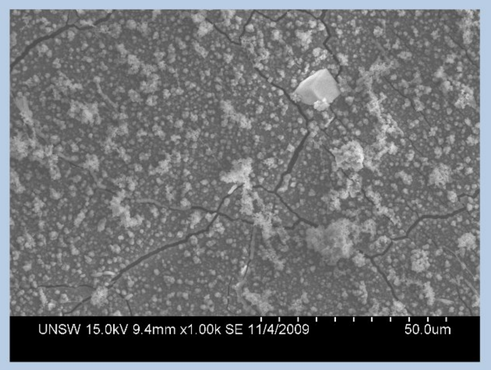
The images below illustrate different types of fouling observed using SEM.



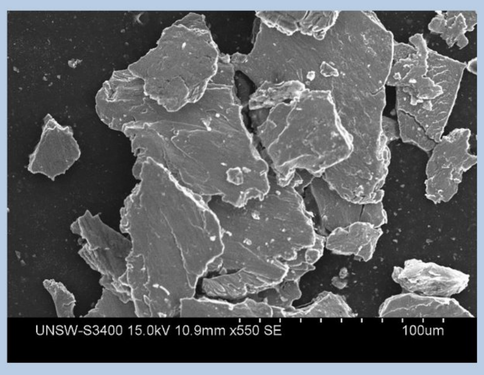
Rhomboidal calcium carbonate crystals with underlying silica fouling.



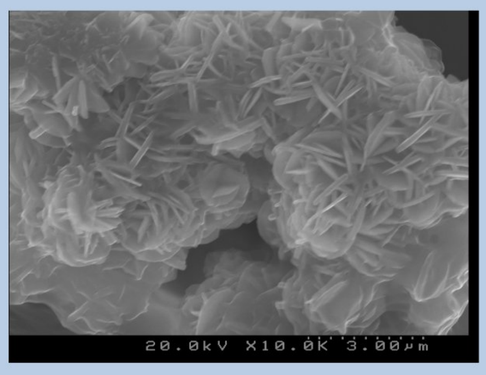
Silica fouling.



Amorphous iron cake. Large crystal is sodium chloride.

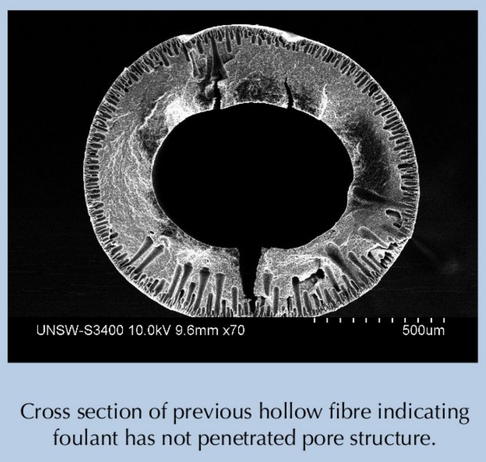


Amorphous inorganic scale most likely calcium based.

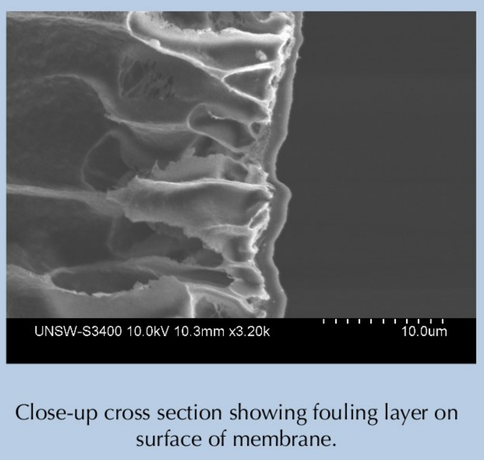


Ferric oxide crystals.

Severely fouled microfiltration hollow fibre from a MBR.

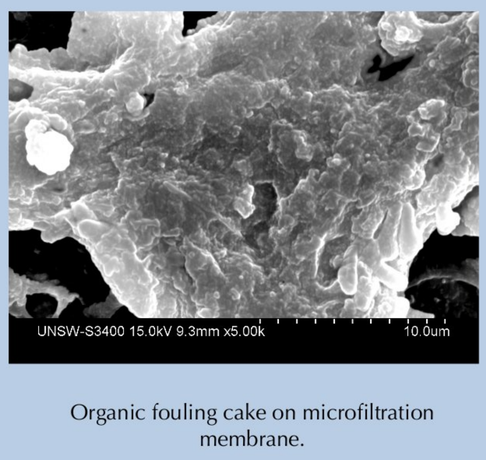


Cross section of previous hollow fibre indicating foulant has not penetrated pore structure.



Close-up cross section showing fouling layer on surface of membrane.

Unidentified microorganism found on surface of membrane.



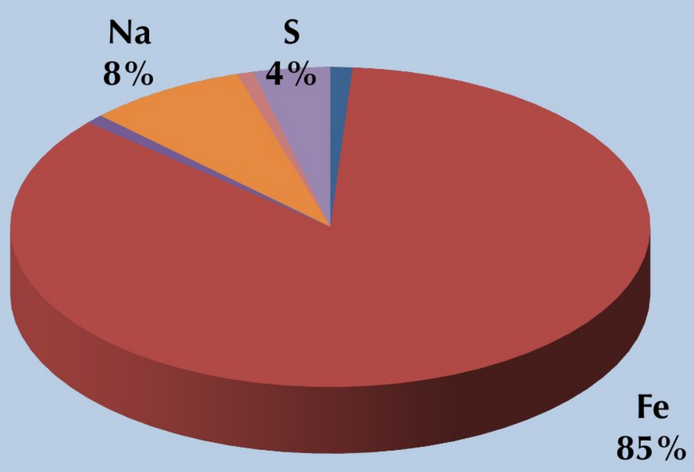
Organic fouling cake on microfiltration membrane.

Inductively Coupled Plasma

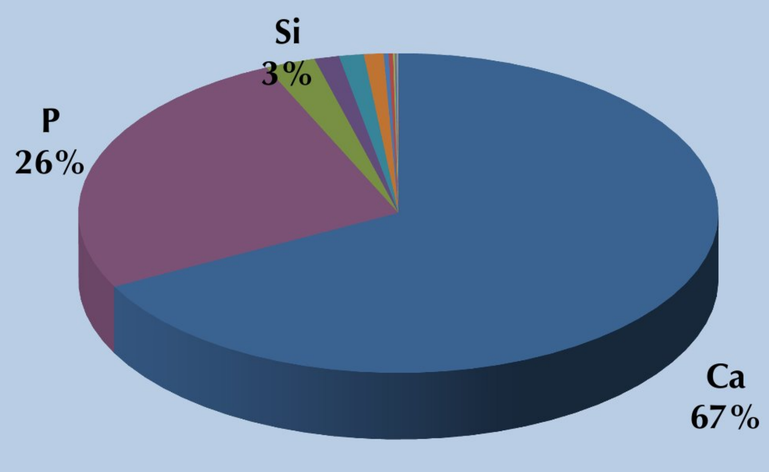
Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP-OES) uses plasma to excite atoms which emit electromagnetic radiation at a wavelength characteristic of a particular element. The intensity of this emission is indicative of the concentration of the element within the sample.

The metals that are identifiable using this technique are sodium, magnesium, aluminium, phosphorus, sulphur, potassium, calcium, chromium, iron, nickel, boron, barium cadmium, copper, manganese, lead, silicon and zinc.

Problematic material for ICP analysis includes minerals which are insoluble such as silica and chlorine.



ICP analysis of removed fouling material indicative of high iron material. Total mass 2367mg/m^2



ICP analysis of removed fouling material indicative of calcium and silica scale. Total mass 1842mg/m^2

Loss on Ignition

Loss on ignition analysis is an approximate technique used to determine whether the fouling material found on the surface of an RO or NF membrane is predominantly inorganic scale or organic/biofouling.

This test is not able to characterise the type of fouling present beyond these broad classes. This technique is useful for quantifying the amount of volatile and fixed solids in the foulant material.

LOI analysis is useful for determining the additional techniques required to characterise the fouling material.