8-6. Analytical Methods for Heavily Fouled Elements

8-6-1. Visual Inspection

To a certain degree by a visual inspection of the autopsied element, the appearance of the foulant will provide clues to its nature and to the difficulty in its removal. Large filter media particles like activated carbon will be evident to the sight.

A biological foulant will have a different appearance than an inorganic scale, and smell differently. Other mechanical problems with the element such as the broken glue line and, the damaged feed and product water channel materials can be spotted visually.

8-6-2. Dissolution in Acid

If the deposits on the membrane appear to be crystalline and dissolve in an acidic solution (HCl) of pH 3 to 4 with some gas evolution (carbon dioxide), then it is likely that the deposits are consisted of carbonates such as $CaCO_{3.}$

Sulfates or silica will only dissolve with difficulty in very low pH(e.g. pH 1). If the scale is soluble in 0.1M hydrofluoric acid (HF) solution, it is possibly silica.

8-6-3. Dye Test

A dye such as methylene blue or rhodamine B can be added in a concentration of 0.001 to 0.005% to the feed water of the test element on an individual element test stand. The dye will permeate the membrane through areas of degradation and mechanical leaks in the element. The dye in the permeate water can be detected visually or measured using a spectrophotometer.

After the dye test, the element can be autopsied to visually inspect the specific location of the dye passage. Damaged areas may pick up more dye than unaffected areas. Chemical attack by chlorine or high dosage acid induced hydrolysis of the membrane will tend to result in uniform absorption of the dye.

8-6-4. Optical Microscopy

When the visual inspection of the autopsied element can not reveal enough information about the nature of foulants, a high power light microscope can tell if a foulant is biological or inorganic scale. It can also provide information about the crystalline structure of a scale formation. Using polarized light, the microscope can tell the difference between calcium sulfate and calcium carbonate scales, since calcium sulfate crystal has more than one refractive index to give a unique appearance.

Sometimes it can be recognized under the microscope that a layer of scale sitting on the membrane surface is covered by another layer of organic and biological foulants. In this case, it may be a most effective cleaning to remove the organic and biological foulants first with an alkaline cleaning solution, prior to attempting to remove the scale with an acidic solution.

8-6-5. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR can provide additional information about foulants on the membrane surface. When there is a thick layer of foulants on the membrane, it is preferred to scrape off a sample of foulants from the membrane surface. The sample is dried and FTIR of the dried sample is run. If a suitable size of sample cannot be collected from the membrane surface, an FTIR analysis may be performed directly on the fouled membrane using a technique called attenuated total reflection (ATR). ATR will give FTIR spectrum corresponding to the foulants after it subtracts FTIR spectrum of a fresh membrane from that of the fouled membrane. Sometimes, the subtraction process does not work well to result in a mixture of peaks corresponding to both the foulants and the membrane.

Organic and inorganic compounds have their own specific FTIR peaks which can in turn be used to identify the compounds in a mixture. From the peak intensity, semiquantitative analysis is also possible. If the FTIR spectrum shows peaks due to Si-O-Si, CO_3, and SO_4, then it indicates there are silica, calcium carbonate, and sulfates, respectively in the foulants. If the spectrum exhibits peaks corresponding to C-H, -CO-, C-C, and C-N, then it indicates the foulant is organic or biological. Peaks due to C-H, -CO-, C-C, and phenol groups strongly suggest that the foulant is consisted of humic acid.

8-6-6. Scanning Electron Microscopy (SEM)

SEM can distinguish much smaller objects than the optical microscope. So SEM gives clear photographs of particles as small as 0.1 μm to identify small crystalline and amorphous inorganic scaling matter, and also the cell structure of microorganisms. SEM could be very helpful in analyzing the foulants in more detail.

8-6-7. Energy Dispersive X-ray (EDX)

During the process of SEM, X-ray radiation is emitted from the sample due to the electron bombardment. The X-ray is low energy and characteristic of the elements in the sample. Thus EDX can identify elements in the sample and even offer a semi-quantitative analysis of the sample. It can detect very small amount of inorganic elements in the sample and also identify carbon, nitrogen and oxygen, though less sensitively. It works the best for analyzing an inorganic scale, but also is useful for organic sample analysis.

The method can also furnish evidence of halogen damage to the membrane due to chlorine oxidation by detecting the presence of chlorine attached chemically to the polyamide.