Integral diagnosis of fouling problems by analysing biomass and inorganic compounds in membrane elements used in water treatment

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Abstract Operational problems in membrane installations used in water treatment can be caused by a variety of fouling types. Therefore, a systematic approach based on the application of an autopsy of membrane elements followed by analysis has been developed, which enables an integral diagnosis of the type and extent of fouling. Analysis includes both biological parameters for biomass quantification (ATP) and biomass characterisation and chemical parameters for determining the presence of inorganic compounds (ICP-MS). Advantages of this approach include: first, complete and conclusive information about the nature and extent of fouling of the membrane filtration plant; and secondly, rapid diagnosis (within 8 hours) of biofouling. In addition to membrane element analysis, also a suite of monitoring tools (AOC test, biofilm monitor, oxygen consumption monitor and scaleguard) is available for elucidation and control of the processes responsible for the fouling problems. These tools can also be used to test chemicals for their effect on (bio)fouling. Research is continuing to substantiate relationships between test parameters and the extent of operational problems.

Keywords Autopsy; biofouling; diagnosis; drinking water; fouling; membranes; nanofiltration; reverse osmosis; scaling

Introduction
The use of membrane filtration in water treatment enables the production of high quality drinking water by removing micro-organisms, inorganic and organic compounds, respectively. However, fouling of membranes may cause operational problems (increase of pressure drop and/or decrease of flux) and can be a threat to the conditions of supply (higher costs and insufficient supply of drinking water). The fouling mechanisms of spiral wound membranes include scaling (inorganic deposits), organic fouling, particulate (colloidal) fouling and biofouling. Scaling by inorganic compounds is usually controlled by the use of a scale inhibitor (polymer, acid) and scaling by particulate material can be controlled by pre-treatment (e.g. ultrafiltration). Biological filtration processes removing biodegradable compounds as pre-treatment can reduce the risk of biofouling.

Different types of fouling can occur simultaneously, influencing each other (Amjad, 1992). Biofouling is considered to be a major (and difficult to control) problem in nanofiltration (NF) and reverse osmosis (RO) membrane filtration (Khedr, 2000). Therefore, controlling biofouling is a major challenge in operating membrane filtration installations (Flemming et al., 1997; Paul, 1996). In order to explain (bio)fouling three definitions have to be illuminated: biofilm formation, biofouling and operational problem.

- Biofilm formation is defined as the accumulation of micro-organisms including extracellular compounds on a surface due to attachment and growth. Biofilms are commonly observed on water-exposed surfaces (Costerton and Stewart (2001)).
- Biofouling is defined as the accumulation of biomass (biofilm) on a surface by growth and/or deposition to such a level that it is causing problems.
• An operational problem of a membrane installation is at hand when the increase of the normalised pressure drop (NPD) and/or the decrease of the normalised flux (MTC) exceeds 15% of the start-up values of these parameters. At values >15% corrective actions are recommended and guarantees can be restricted by the manufacturers. This definition of operational problems is arbitrary but it enables a comparison of the data of studies performed at different plants.

The diagnosis “biofouling” – which has been described extensively (Amjad, 1992; Ridgway and Flemming 1996) – is only justified when a relation is found between the encountered operational problems and biomass accumulation as determined with adequate parameters. Suites of biomass parameters and analytical tools are available (Flemming et al., 1996), but the question remains which parameter(s) at which level is conclusive for identifying biofouling as the cause of the operational problems. This report describes our efforts aiming at achieving methods and strategies for diagnosis of (bio)fouling. This study is part of the integral approach of controlling biological activity in water supply as developed at Kiwa.

**Diagnosis of biofouling**

A destructive analysis (autopsy) of one or several spiral-wound membrane elements enables the identification of the cause of an operational problem of a membrane filtration (pilot) plant. The analysis of material collected from the membrane element includes adenosinetriphosphate (ATP) concentrations (a measure for the amount of active biomass), Total direct cell counts (microscopic cell count, TDC), and Heterotropic plate counts (HPC) on R2A medium (expressed in colony forming units, CFU) and inorganic compounds by ICP-MS (inductively coupled plasma mass spectrometry) analysis. The autopsy and the assessment of biofouling with ATP analysis can be performed on site using portable equipment. The on-site study gives (preliminary) results concerning the presence, type(s) and extent of fouling within 8 hours.

We have conducted autopsies in 45 membrane elements (several brands, NF and RO) obtained from 15 (pilot) plants. The various plants were supplied with water, which varied in: (i), type of source water (aerobic, anaerobic, ground water, surface water, tap water and seawater); (ii), pre-treatment; (iii), temperature (from 1 to 30°C, stable and fluctuating) and (iv), dosage of chemicals (e.g. scale inhibitor), respectively. Ranges of concentrations of biomass parameters observed in membrane elements are shown in Table 1. The quantitative information obtained from these autopsies provides a useful database concerning the accumulation of biomass and inorganic compounds. Low biomass values in the membrane element have been observed in the absence of operational problems and high levels of biomass have been observed in plants with severe operational problems. Operational problems with certain plants included an increase of the NPD up to 300% or a decrease of the MTC value of about 25% at the time of autopsy. Autopsies of membrane elements from these plants in general demonstrated high biomass concentrations (> 1,000 pg ATP/cm²).

Assessment of the water quality (concentration of microorganisms and nutrients) prior membrane filtration can also be used for the diagnosis of biofouling. Parameters applied for

**Table 1** Range of average concentrations of biomass parameters in 45 NF and RO membrane elements from 15 (pilot) plants with and without operational problems

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Range</th>
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<tbody>
<tr>
<td>Active biomass (ATP)</td>
<td>pg ATP/cm²</td>
<td>&lt; 3–45,000</td>
</tr>
<tr>
<td>Total direct cell count (TDC)</td>
<td>cells/cm²</td>
<td>5 × 10⁶ – 2 × 10⁹</td>
</tr>
<tr>
<td>Heterotropic plate count (HPC)</td>
<td>CFU/cm²</td>
<td>1 × 10³ – 3 × 10⁷</td>
</tr>
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</table>
the determination of the concentration of microorganisms in water are identical to those applied during the autopsy (ATP, TDC and HPC-values). Parameters for assessment of the concentration of growth promoting substances in water are: AOC and the biofilm formation rate (BFR). The AOC-test is a bioassay with two well-defined pure bacterial cultures. From the maximum growth level of the two individual strains the AOC concentration is calculated and expressed as µg of acetate-C equivalents/l (Van der Kooij, 1992). The BFR-value is determined with an on-line operated biofilm monitor at a continuous flow rate of 0.2 m/s. The accumulation of active biomass (ATP) on the surface of glass rings in this monitor is determined as a function of time and the BFR value is expressed as pg ATP/cm².day (Van der Kooij et al., 1995).

Severe biofouling has been observed in cases where the feed water had BFR-values – temporarily – exceeding 120 pg ATP/cm².day and/or the AOC-value exceeded 80 µg Ac-C/l (Vrouwenvelder and van der Kooij, 2001). BFR-values lower than 1 pg ATP/cm².day, as can be achieved by slow sand filtration, enabled stable operation for periods up to two years without cleaning. Different types of fouling can occur simultaneously and even influence each other (Amjad, 1992). This indicates that controlling fouling requires an integral diagnosis of the type(s) of fouling.

**Integral diagnosis of biofouling and inorganic compounds**

Five different (pilot) plants (which varied in type of source water, pre-treatment, temperature, dosage of chemicals, respectively) showed clear differences in operation of the plant and the concentration of biomass and inorganic compounds in the RO membrane elements (Table 2). Plant 1 was characterised as a plant without an operational problem since the increase of the NPD-value was below 15% of the NPD value during start-up of the plant (study performed after 2 years of operation without cleaning). The autopsy showed low concentrations of biomass (ATP) and inorganic compounds, respectively. The plants 2, 3 and 4 showed NPD increases exceeding 15% and relatively high concentrations of biomass, indicating biofouling. However the membrane element from plant 3 also had a concentration of 20,000 mg of metal ions/m² of membrane surface, with Ca as the main component (>99%). Plant 4 had a concentration of 5,000 mg of metal ions/m² of membrane surface, with Al, Ca, Mn and Fe as the main metal ions (49, 27, 11 and 10%, respectively). These concentrations of metal ions indicate the accumulation of inorganic compounds in the membrane elements from plants 3 and 4, which probably affected plant performance. At plants 3 and 4 fouling was caused by both biomass accumulated and inorganic deposits. Plant 5 differed strongly from the other four plants (Table 2). The membrane installation of plant 5 was fed with process water at high temperatures (50–60°C), a pH of 10 and contained “aggressive” chemicals. Autopsy showed low values for biomass and inorganic compounds which indicated the absence of biofouling and inorganic deposits. The presence of uniformly shaped and sized fibres was observed at the feed side of the

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Plant 1</th>
<th>Plant 2</th>
<th>Plant 3</th>
<th>Plant 4</th>
<th>Plant 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>NPD change</td>
<td>%</td>
<td>&lt; 15</td>
<td>&gt; 15</td>
<td>&gt; 15</td>
<td>&gt; 15</td>
<td>&gt; 15</td>
</tr>
<tr>
<td>Active biomass</td>
<td>pg ATP/cm²</td>
<td>200</td>
<td>13,000</td>
<td>5,200</td>
<td>14,000</td>
<td>&lt; 3</td>
</tr>
<tr>
<td>Inorganic compounds*</td>
<td>mg/m²</td>
<td>&lt; 100</td>
<td>&lt; 100</td>
<td>20,000</td>
<td>5,000</td>
<td>&lt; 100</td>
</tr>
<tr>
<td>Microscopic observation</td>
<td></td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>fibers</td>
</tr>
<tr>
<td>Diagnosis</td>
<td></td>
<td>no fouling</td>
<td>B</td>
<td>B + I</td>
<td>B + I</td>
<td>P</td>
</tr>
</tbody>
</table>

I: inorganic deposits; B: biofouling; P: particulate fouling

* sum of concentration of metal ions
membrane. Fouling (particulate) was caused by the accumulation of these fibres (which passed pre-treatment) on the membranes in plant 5.

Autopsies of membrane elements taken from plants before and after cleaning showed that cleaning was not (always) sufficient to remove most of the inorganic compounds from the membrane elements [data not shown]. An autopsy will result in the diagnosis whether or not (bio)fouling is present and includes a risk evaluation and recommendations to find the cause(s) of fouling if present. Additional tools are available for early detection of biofouling (Vrouwenvelder and van der Kooij, 2001; Kappelhof et al., 2002) and (prevention of) scaling using ScaleGuard® (Heijman et al., 2002), respectively.

**Tools for prediction, prevention and evaluation of cleaning**

Parameters for assessment of the concentration of growth promoting substances in water (AOC and BFR) can be used in order to predict and prevent biofouling. Relatively low values for these parameters have been observed in feed water of (pilot) installations with extensive pre-treatment including slow sand filtration. High values (BFR, AOC) have been observed in feed water of (pilot) plants either using surface water with only ultrafiltration as pre-treatment or treated wastewater. However, extensive pre-treatment does not guarantee absence of biofouling. The feed water quality can be influenced by (seasonal) variations of water quality prior to pre-treatment, which are not completely eliminated in the pre-treatment. Dosage of – even one batch of impure – chemicals (floculent, scale-inhibitor) to the feed water can cause serious biofouling, leading to early replacement of membranes (Vrouwenvelder et al., 1998, 2000). Testing (AOC, BFR) of 14 commercially available scale-inhibitors based on polymers and dosage to single element units demonstrated that chemicals used to prevent scaling differ greatly in their ability to promote growth of microorganisms and confirmed the experiences in practice (Vrouwenvelder et al., 2000; Van der Hoek et al., 1997). With the biofilm monitor the accumulation rates of iron and manganese of the feed water can also be determined (Van der Kooij et al., 1994). These accumulation rates might be indicative for the types of inorganic deposition in membrane elements.

Biofouling can be prevented by: (i) reducing the concentration of micro-organisms and/or reducing the concentration of nutrients by pre-treatment; and/or (ii) performing preventive/curative cleanings. Studies on membrane elements before and after cleaning of a plant showed that it is difficult to remove the biomass from the membrane (Vrouwenvelder and van der Kooij, 2001). A combination of pre-treatment and cleaning may be the most effective way to prevent biofouling.

The methods AOC, ATP and BFR are currently applied in practice for screening of the biofouling potential of water types and for monitoring (pilot) installations. An early (online) warning system for biofouling based on oxygen consumption is being developed (Kappelhof et al., 2002). The research is aiming at establishing more quantitative relationships between the concentrations of biomass and nutrients in the feed water and biofouling.

The integral approach for diagnosis of fouling is an essential step in controlling fouling (biofouling, inorganic deposition, organic fouling and particulate fouling). However, conclusions about the type(s) and extent of fouling should be based on relationships between the encountered operational problems and fouling parameters in membranes and in feed water as determined with adequate methods. The database obtained from autopsies and monitoring of feed water of (pilot) plants with and without operational problems is a start – which has proven to be valuable in controlling fouling – for an integral diagnosis of fouling.

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References


