Development of a new integrity testing system


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Abstract As membrane filtration systems are more commonly used in the water treatment industry, the call
for a reliable, fast and on-line integrity testing system becomes more urgent. Especially where membrane
filtration is used in potable water production for the removal of pathogenic microorganisms, the integrity of
such a system is of the utmost importance. Membrane integrity testing can be performed in a number of
ways, where the pressure-hold or vacuum-hold test and the diffusive airflow test are well known. Although
relatively widely applied in membrane plants worldwide, all of these methods exhibit major drawbacks like
off-line testing and the lack of a direct relationship between the measured data and the removal efficiency
(log-removal). To overcome the above mentioned drawbacks of conventional integrity testing systems, a new
kind of integrity test, the Spiked Integrity Monitoring System or SIM®-System was developed by NORIT
Membrane Technology in close co-operation with Water Supply Company North Holland and IWW
Rhenish–Westphalian Institute for Water Research, which combines the accuracy of a challenge test with
the speed of a pressure test, while keeping the system under test in operation.

Keywords Integrity testing; retention; ultrafiltration

Introduction
Membrane filtration, specifically ultrafiltration (UF), is typically used for the removal of
suspended solids and micro-organisms from a liquid stream. It is applied in a number
of fields, amongst which are the pre-treatment to reverse osmosis or the re-use of waste-
water treatment plant effluent. One of the rapidly growing applications for UF is the use as
a last unit operation in a potable water treatment scheme, specifically for the removal of
pathogenic microorganisms, like Cryptosporidium oocysts and Giardia. Both can pose a
significant health threat since they cannot be killed by means of chlorinating. Recent out-
breaks in the USA, Australia and the UK have significantly increased awareness of the risk
of microbial contamination of the potable water. This has led the British government to put
into effect new legislation, basically forcing water companies to either continuously moni-
tor the water they put into supply, or provide a “sufficient treatment plant capable of continu-
ously removing or retaining particles greater than one micron diameter”. This may be
done by means of installing a “membrane or other filtration system that has been approved
under regulation 25 of the Water Supply Regulations 1989” (DWI,1999). As a result of
these rules, a number of UK water companies are installing, or planning to install, such
systems.

Integrity testing
Obviously, membrane integrity has been an issue since the time the use of membranes
became common practice in the industry. One of the first industries to adopt membrane
filtration was the pharmaceutical industry, where proper filtration of liquid products to
establish absolute removal of any pathogenic organism was paramount. In those days, the
late 1970s and early 1980s, several membrane integrity methods were developed, all of
them initially focused on testing relatively small cartridge or plate-and frame membrane
modules. The pressure-hold or vacuum-hold test and the diffusive airflow test are well known. Since all of these methods are very simple and straightforward, they were adopted to suit larger scale membrane plants as well. As the market for large scale membrane treatment, especially for the treatment of water for potable purposes, developed world-wide in the late 1980s and the 1990s, the existing integrity testing methods were also applied for these types of plants, since their role as a bacteria barrier was recognised.

As membrane systems play a crucial role in retaining micro-organisms, a large number of investigations have been conducted to establish their removal efficiency. These studies have clearly shown ultrafiltration membranes to be able to achieve a log removal efficiency higher than 5.8 (Kamp, 1995), higher than 6 (Panglisch et al., 1998a) and well over 8 (Khow et al., 19??). An ultrafiltration system will surely provide sufficient treatment in removing or holding back 1 µm particles, but any system will have to have some means of checking and maintaining its integrity. Practically all membrane systems used for the final treatment of potable water use membrane elements filled with capillary fibres during their operational life. An integrated part of any potable water treatment plant should be a test to establish the integrity of these fibres. This does not necessarily mean that each and every fibre has to be intact. If the requirement for a plant is to provide a certain log removal for Cryptosporidium sized particles, and a method were available to actually measure this parameter, an end-user may decide to keep a plant running as long as the required removal rate is met, instead of shutting it down at the first sign of a single fibre breakage. This would enable the end-user to act upon such fibre breaks when the need arises and when taking part of the plant off-line would be convenient, while still being able to uphold the high standards in water quality and to satisfy the proper authorities.

**Integrity testing**

As discussed previously, membrane integrity testing can be performed in a number of ways, which have been in common practice since membrane filtration became an accepted technology. Most of them are based upon the same principle. The pores of the membrane are filled with the fluid that is treated (e.g. water). For any given liquid, pore size and constant wetting, the pressure required to force an air bubble through a pore is inversely proportional to the size of the pore. This pressure is called the bubble point pressure and is described by the Laplace equation for capillary tubes (Scott, 1995). If the air pressure is below the bubble point pressure, the fluid in the membrane is not removed and transport of air from one side of the membrane to the other is exclusively governed by diffusion of air through the liquid within the membrane pores. For these sorts of tests, the air pressure is always kept below 0.8 times the bubble point pressure.

**Vacuum test.** The vacuum test is probably one of the oldest tests to establish the integrity of a membrane. It has long been used to test reverse osmosis elements and can also be used to test ultrafiltration elements. When performing a vacuum test, the unit to be tested is firstly drained completely. Then, a vacuum is applied to one side of the membrane, after which this side is sealed. The other side of the membrane is kept at atmospheric pressure. The rate of pressure increase at the vacuum side of the membrane is an indication of the integrity of the membrane, as it is compared to a reference value. Even an intact membrane will show a certain pressure increase due to diffusion of air through fluid in the membrane wall.

**Pressure-hold test.** The pressure-hold test or pressure decay test is basically the opposite of a vacuum test. Again, the unit to be tested has to be drained completely. Air pressure is then applied to one side of the membrane. After sealing this side, the rate of decline of the
pressure is monitored and compared to a reference value. Again, any element will show a certain, low decay rate, due to diffusion phenomena (Adham et al., 1995).

**Airflow test.** The airflow test or diffusive airflow test is a variation on the pressure-hold test. However, it does not measure the rate of pressure decline. When executing the airflow test, the operation of the membrane unit is stopped. One side of the unit is drained and then pressurised with air, while the other side remains filled with water. Air will be transported to the other side of the membrane, either by diffusion (in case of an intact system) or by a leaking fibre. The water that is displaced by air entering the other side of the membrane is measured and the flow is an indication of the integrity of the tested membrane (Millipore, 1989).

**Challenge test.** The ultimate integrity test is the challenge test with tracer micro-organisms, which is the only one that actually measures the log removal performance of any system in a direct manner. It is however a relatively expensive test, very labour intensive and not preferred as a test that is to be carried out on a production facility on a regular basis. On top of that, it takes a relatively long time for the results to be known, since the test requires samples to be cultivated.

As far as the integrity systems go that are mentioned above, most of these exhibit major drawbacks:

1. Every method, that relies on measuring a change in pressure, e.g. the pressure decay test or the vacuum test, faces a decrease in sensitivity, as the unit to be tested increases in size; this is due to the fact that if the system size increases, the volume of the side of the membrane where the pressure change is measured increases; this results in a decreasing change rate of the pressure and thus a decreased sensitivity to broken fibres; this in effect means that the amount of membrane modules that can be tested simultaneously, is limited.

2. This may be compensated partially by increasing the testing time necessary to accurately measure the integrity of a system; obviously, this increases the down-time of the system significantly.

3. Testing always has to be performed off line; this means the plant needs to be stopped, drained, tested and re-started, the downtime of the plant will increase as the testing frequency increases and thus the net capacity will decrease; this can only be compensated by installing more membrane area.

4. There is no direct relationship between the measured data and the removal efficiency (log-removal); at best an empirical relation has been established on the basis of a number of integrity tests and microbiological analyses (Panglisch et al., 1998b).

To overcome the above mentioned drawbacks of conventional integrity testing systems, NORIT Membrane Technology has developed a new integrity testing system, which overcomes all drawbacks mentioned above, the Spiked Integrity Monitoring System or SIM\textsuperscript{-}System. This very sophisticated integrity test was developed in close co-operation with PWN Water Supply Company North Holland and IWW Rhenish–Westphalian Institute for Water Research.

**Spiked integrity monitoring system (SIM\textsuperscript{-}System)**

The newly developed method is based upon a common challenge test. It establishes the number of particles on the feed side as well as the permeate side of the membrane. The log removal can be directly calculated from the measurements. Therefore, there is a direct relationship between the removal efficiency and the number of particles at the feed and filtrate side of the membrane.

In order to be able to measure the removal efficiency adequately, the number of particles...
in the feed cannot be too low. In relatively clean feed water, i.e. from potable water production, this will pose a problem since the number of particles in the feed will be too low to demonstrate a high log removal. Filtrate counts from intact membrane modules in pilot trials are typically in the order of 0.03 to 0.08 particles per ml (for particles > 1 \( \mu m \)). Feed counts are typically in the order of 300 to 800 particles per ml (> 1 \( \mu m \)). Therefore, without further enhancement of the measuring technique, a log reduction of 3.6 to 4.4 can be measured at best.

This problem has been solved by developing a dedicated system for dosing a limited amount of inert powdered activated carbon (PAC) particles for a short period of time at the feed side of the membrane plant during filtration. This renders a dramatic increase in the measured log-removal potential to well above 6. Moreover, PAC-particle spiking increases the drop in log-removal caused by a compromised fibre. One compromised fibre in a pilot system caused the log removal to drop 0.8 with PAC spiking. Without spiking the drop was only 0.2–0.3, proving the sensitivity of the new system. The particle size of the PAC is in the same order of magnitude or smaller than the micro-organisms to be retained by the membrane barrier, Cryptosporidium (typically 2–7 \( \mu m \)) and Giardia (typically 4–12 \( \mu m \)).

**Pilot study.** To increase the range of log removal measurement a dedicated system for on line dosing of a limited amount of inert particles to the feed of the UF system during a short period of time has been developed. A dedicated type of NORIT PAC is used. This carbon type has no interaction with the membrane and is approved for use in water treatment (DWI, Kiwa ATA). The applied PAC has a particle size distribution such that almost 70% of the particles have a diameter of less than 1.7 \( \mu m \) and therefore is in the same order of magnitude or smaller than micro-organisms such as Giardia (4–12 \( \mu m \)) and Cryptosporidium (2–7 \( \mu m \)), to be rejected by the UF system.

A dosing station was constructed by means of a mixing vessel for storing the PAC suspension. The suspension contained approximately \( 1.0 \cdot 10^7 \) particles/ml (> 1 \( \mu m \)). To avoid sedimentation of PAC the vessel was stirred continuously. A dosing pump fed the suspension into the UF feed at a fixed period after a backwash cycle.

The UF pilot plant was equipped with two Met-One PCX 1 \( \mu m \) particle counters with a measuring range of 1–150 \( \mu m \) connected to the feed and permeate side. Particles were automatically measured in samples of 40 ml.

**Pilot results.** For interpretation of the data two measuring windows have been defined. The first window covers the data during PAC-spiking, the second window covers a reference measurement without PAC-spiking. A typical plot for a pressure vessel without compromised fibres is shown in Figure 1.

The measurement started after a backwash. The elevated number of particles in both feed and permeate are caused by the backwash. Valve action releases some particles at both sides of the membrane. Moreover the backwash pump introduces particles at the permeate side. These phenomena yield a short peak on the feed and permeate of the membrane but are not related to a compromised module. The feed and the permeate particle count stabilised at 700–800 particles/ml (> 1 \( \mu m \)) and 0–0.08 particles/ml respectively.

After PAC-spiking the UF feed signal rose to a level of around 14,000 particles/ml (> 1 \( \mu m \)), which took approximately two minutes due to the residence time in the particle counter. After the feed concentration reached its maximum value, the measuring window was started lasting six data points. The permeate measurements did not react at all to the particle increase at the feed side. The calculated log removal amounted to 5.83 log units, which was the lowest value during the measuring window. After six particle counts PAC-spiking was stopped, resulting in a decrease of the feed side particle counts.
Then a second backwash was started lasting approximately 45 seconds. Once again the backwash is visible in the particle count on both the feed and the permeate side. After the backwash both feed and permeate measurements stabilised at 700–800 particles/ml (>1 µm) and 0–0.08 particles/ml respectively. Another measuring window was started, once again lasting six data points. Without PAC-spiking the lowest measured log removal amounts to 4.65 log units. A typical plot for a pressure vessel with an element with one compromised fibre is shown in Figure 2.

Again two measuring windows were taken into account. After PAC-spiking up to a particle count of 14,000 particles/ml (>1 µm) the increase at the feed side is followed by a small increase at the permeate side as well. The calculated log removal amounted to 5.07 compared to 5.83 log units, a signal that membrane integrity has been breached.
In addition, results from the nonspiked measuring window did not show a pronounced decrease of log removal for fibre compromising: 4.65 log and 4.40 log removal respectively. The results of both tests are summarised in Table 1.

By increasing the amount of particles in the UF feed the potential log removal can be increased to even higher log levels, thereby showing the potential of PAC dosing during inline integrity testing.

**Full-scale evaluation.** The SIM®-system was first implemented at Yorkshire Water Services, one of the large private water companies in the UK, at their Keldgate Water Treatment Works. Earth Tech Engineering was awarded the contract to design and construct the ultrafiltration plant. The design and manufacturing of the membrane racks and supply of the membrane elements was subcontracted to NORIT Membrane Technology. Commissioning of the plant was finished in November 2000 and the plant was subsequently taken into supply. The plant treats water from four wells near the City of Hull, UK and has a capacity of 90 MLD and comprises 11 racks of 24 membrane housings, filled with four X-Flow S-225 UFC membrane elements each, comprising a total membrane area of 36,960 m² (Franklin et al., 2001). Since this plant is used as a last stage in the potable water production for the region, a removal of at least 4 log for Cryptosporidium sized particles was one of the specifications in the design. To check and maintain the required log removal, the SIM®-system was installed on the plant. The system is capable of monitoring each of the eleven racks individually. It consists of a mixing and dosing section and a particle counting section. The amount of carbon to be dosed is fixed and calibrated. This means that the total amount of carbon particles in the feed stream to the UF unit is variable, as the feed flow to the unit will vary with demand of the plant. Since a high log reduction was to be achieved by the measurement, it was decided to spike the feed to the membrane racks with $1.5 \cdot 10^5$ particles per ml at the maximum capacity of the rack. The particle concentration may be higher as the feed flow is lower. This is taken into account in the log removal calculation.

**Full-scale results.** Testing the SIM®-system was one of the requirements in the take-over procedure of the Keldgate plant. The tests have been conducted automatically. In the control system, SIM®-tests are scheduled on a periodic basis, typically one test per rack per day. The PAC suspension was dosed during a period of six minutes. Because of the time lag between the dosing station of the PAC suspension and the PAC actually reaching the racks, a measuring window was defined for each rack.

For one of the take-over tests, prior to the time the plant was put into operation, five SIM®-tests were conducted on each of the eleven racks. The results are displayed in Table 2.

A log 4 removal was required to pass the test, and the system showed very good performance, exceeding the pass criteria significantly on each measurement. Since the take-over test was performed relatively fast after the membrane elements were loaded, it is

<table>
<thead>
<tr>
<th>SIM®-System</th>
<th>UF system</th>
<th>Particle conc. feed (1 µm) 1/ml</th>
<th>Particle conc. permeate (1 µm) 1/ml</th>
<th>Log removal $\log_{10}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>PAC</td>
<td>Noncompromised</td>
<td>14,055</td>
<td>0.021</td>
<td>5.83</td>
</tr>
<tr>
<td></td>
<td>Compromised</td>
<td>13,689</td>
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<tr>
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<td>Noncompromised</td>
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</tr>
<tr>
<td></td>
<td>Compromised</td>
<td>733</td>
<td>0.029</td>
<td>4.40</td>
</tr>
</tbody>
</table>
expected that the particle counts at the permeate side of the membrane will decrease and the measured log removal subsequently will increase.

Data analysis
During the development of the SIM®-system, data collection and analysis have been pretty straightforward. However, IWW Rheinish–Westphalian Institute for Water Research has developed a statistical analysis method for doing so.

The whole procedure comprises subsequent steps, beginning with a detectable particle concentration in the permeate and ending with the necessary sudden increase of the particle concentration in the feed, enabling the proof of the target for the log-removal with a chosen confidence level.

At first, the particle concentration of the permeate is measured and analysed under common treatment conditions. The reason for a certain detected value or level is absolutely unknown. A fraction could be of raw water origin, passing the membrane through a compromised fibre, or it could be caused by the backwash procedure. Commonly, the backwash water contains a small amount of particles. During a backwash cycle, these particles can adhere on the large surface on the permeate side including pipes of a filtration unit, especially if these particles are retained in or on the supporting layer of the membrane. After starting a filtration cycle, adhered particles can escape into the permeate. Therefore, particle count data from the permeate during a common filtration process was handled like a background scattering.

In the second step, from this background scattering and a chosen level of significance, e.g. 95% confidence, the determination limit for particle counting in the permeate during particle spiking into the feed can be calculated. This is necessary to ensure that measured particle concentrations above this limit are caused by spiked particles and not by particles of the original raw water.

In the third step, the necessary particle dosage for the spiking procedure is calculated on the basis of this detection limit, the chosen level of confidence and the chosen target for the test, i.e. the log-removal to proof. If the measured particle concentration in the permeate during the spiking of the raw water is lower than the detection limit, the target for the log-removal of the membrane unit is assuredly reached. If the measured particle concentration in the permeate is higher than the detection limit, the target is failed and the actual lower log-removal is calculated.

Conclusions
As membrane filtration becomes increasingly more important in the production of potable water, it is evident that membrane integrity checks are desirable, if not mandatory. Most of the systems for membrane integrity testing that are supplied today are not capable of online testing. On-line test equipment that is used (turbidity measurement or particle counters) does not have the sensitivity required of such tests. None of the existing systems can directly measure the removal efficiency of a membrane system. The SIM®-system solves...
all of these drawbacks. It is, in essence, an on-line challenge test, which renders results in seconds and is fully automated. The system has been extensively tested on pilot scale and has proven to be very effective in measuring the removal efficiency and spotting a defect in the membranes. Upon designing and installing the system on a 90 MLD plant, it has shown its capability of establishing the removal efficiency of any single rack, thus providing the security needed to safely operate this plant as a final barrier to pathogenic micro-organisms.

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References


