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Calculating particle log reduction values based on pressure decay tests on Multibore® hollow fibre membranes

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ABSTRACT

The Multibore® hollow fibre membrane has a distinct advantage in comparison to other hollow fibre membranes due to its extreme durability, which prevents fibre breaks to a very high degree. The integrity of a system nevertheless will need to be checked in order to make sure no membrane damage has occurred. The integrity of the membrane system is normally tested by means of a pressure decay test (PDT). The results of this test have to be interpreted so that a plant operator knows the actual log reduction value (LRV) of the plant. This paper deals with this interpretation and a formula is derived from which the LRV can be calculated. The method is partially based on the ASTM D6908-6906 method in which case, the air flow in an integrity test is compared to the air flow through a complete membrane breakage. It is shown that the PDT is sensitive enough to be able to detect a single broken membrane in even the largest membrane racks and that a 4-LRV can be guaranteed as long as the pressure decay rate is within the specified range.

Keywords: Membrane; Integrity test; Pressure decay; Multibore®; Log reduction value

1. Introduction

One of the great advantages of membrane-based water treatment lies in the fact that the filtrate quality in terms of particle removal is completely independent of the feed water characteristics. Membrane plants are, therefore, often used to eliminate potential micro-organisms from drinking water sources. In most of these plants, the membranes perform a very good job at providing safe drinking water. A potential issue for membrane plants, however, is the occurrence of fibre breaks which happens regularly with many single bore membrane plants [1,2]. In such cases, the membrane systems need to be shut down and the affected membranes have to be repaired or the modules have to be exchanged. The Multibore (see Fig. 1) that inge supplies is characterized not only by extremely good overall performance like virus rejection, permeability and low-fouling propensity, it is also extremely stable to mechanical problems that would lead to fibre breakage in single bore membranes. Based on this, as well as inge's 10 years experience with membrane production, inge guarantees that the customer will not have any costs related to membrane repairs, as inge takes over these costs. Despite the fact that the Multibore membrane is extremely stable, a membrane plant operator needs to know whether the plant is always delivering a constant quality of

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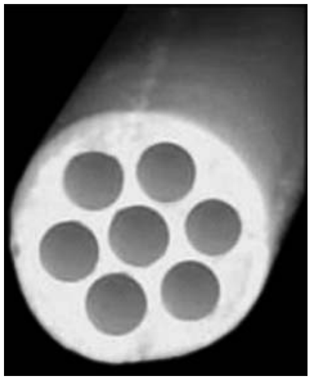

Fig. 1. Photo of a multibore membrane.

produced water. Integrity checks are often carried out based on a pressure decay test (PDT). These integrity tests themselves can be stressful to the membranes, but with the Multibore membrane, this is not the case, and there is no restriction on the number of integrity tests performed on the membranes. This paper deals with the interpretation of the value of the PDT so that a good estimation can be made of the particle rejection level, expressed as the log reduction value (LRV), of the plant. The dependencies of the PDT results on the position of the damage, as well as the trans-membrane pressure (TMP), are very important factors which are taken into account in the discussions.

2. Methods

The Multibore® membranes are produced in a diffusion-induced phase separation (DIPS) process [3,4]. This means that the base materials of the membrane together with additives are dissolved in a solvent, which diffuses out of the nascent membrane by means of a non-solvent. The main material for this membrane is polyethersulphone (PES).

Measurement of water flow: a Multibore® membrane was cut cleanly with a sharp knife. One side of the membrane was connected to a pressurized water supply, while the other side was left free. The water exiting from the open end of the Multibore® was collected in a measuring cylinder over a certain time. The pressure at the membrane inlet was measured with a pressure transducer.

Measurement of air flow: the same membrane from which the water flow was measured was used to determine the air flow. The membrane was connected to a PMI porometer, which is normally used to determine poresize distributions in microfiltration membranes. This machine measures the gas flow accurately at a given pressure.

3. Discussion

3.1. Theoretical background

The pressure drop in a PDT is caused by air that flows through pores or defects which have opened under the pressure applied during the PDT. This pressure is normally set at a much lower value than the bubble point of the membrane. The bubble point for UF membranes is in the range of 100 bar, which is well beyond the pressure that this polymeric membrane can withstand. The pressure chosen is usually 1 bar, sometimes this is the requirement of the membrane producer, but in some countries, this pressure is actually required by regulations. The 1-bar pressure ensures that all defects above 3 micron are checked, which ensures that no larger organisms like Cryptosporidium or Giardia can pass the membrane.

In theory, this means that by measuring the pressure drop in a PDT, it is not possible to conclude that viruses are also retained. However, in our experience with single bore membranes, whenever there is a breach in integrity, this is usually caused either by a larger defect which was physically damaged by a large sharp particle that entered the membrane, or by pressure shocks. The damage to a membrane could then be in the form of a deep scratch over the surface of the membrane, a large chunk ripped out of the surface of the membrane or a fissure in the surface. In addition, most of the damages will, over time, lead to (virtually) complete membrane breakage.

If we calculate the amount of small damages that would lead to substantial loss of integrity and, therefore, a drop in the rejection of viruses in the water to be treated, we will come to a very high amount of defects. For example, when we take 1 industrial scale module from inge with 60 surface area (dizzer XL 0.9 MB 60) and operate this at a flux of $100 l/(m^2 h)$ at a high TMP of 1 bar, we need almost a million large pores or defects of 1 micron to drop below 4-log removal of virus (calculation based on the well known Hagen–Poiseuille equation). Clearly, this is very unlikely to happen without a substantial amount of large defects to occur simultaneously.

In order to estimate the LRV at any point in the systems life, a starting value of the membrane and module in terms of virus rejection has to be known. Several UF suppliers have data for the rejection of particles with their membranes when there were no known defects in the module. Some have these values accredited by independent institutes. This is also the case for inge whose membranes have been tested many times at well-known independent institutes. They have consistently measured more than 4-log reduction on inge's production membranes while testing with ultrapure water, and for the above-mentioned industrial scale, modules with 60 m^2 have tested more than 5.6 log, with no MS2 phages detected in the filtrate. With real feed water, the measured LRV has always been measured at a level above 5 log. MS2 phages are mostly used instead of viruses, as they have a similar size to small viruses. The above means that with the measurements of the PDT, it is possible to gather information not only on larger micro-organism rejections, but we can also get at least a good estimate of virus rejection.

Several papers have been written on the interpretation of the PDT data in regards to micro-organism rejection values. An ASTM method has been published [5], which gives an approach for estimating the LRV of a membrane system. This ASTM method is based on the Hagen–Poiseuille equation, which is only valid for laminar flows. The method states that it can be used to estimate an equivalent number of fibre breaks, but that it is only valid for smaller capillaries, below 0,4 mm. Several membrane manufacturers have used a similar approach to quantify the LRV with their membrane systems [6,7]. In larger fibres, this theoretical method would largely overestimate the calculated remaining LRV. The ASTM suggests an experimental approach for these cases; and with the 0,9 mm capillaries that inge uses in its Multibore membranes, this approach is outlined here. In a PDT, the measured pressure decay can be translated into an airflow going through the potential defects in a membrane system. This air flow is compared to the experimentally obtained air flow through a completely cut membrane. An equivalent number of broken fibres is now obtained by dividing the measured air flow in the system by the measured air flow through a cut fibre. In the same way, the experimental water flow through a cut membrane is measured. This water flow is multiplied by the equivalent number of broken membranes and from this, a total bypass water flow is obtained. It is now possible to calculate the theoretical LRV by comparing the bypass flow to the total filtrate flow.

One of the main criteria when estimating LRV from PDTs is the TMP at which the system is running. In times of higher fouling, or when plants are allowed to operate at higher TMPs, the bypass flow going through the defects quickly becomes larger, while the total flow through the membrane plant remains constant. In case of a defect, this leads to a reduction in LRV with increasing TMP.

3.2. *Air flow calculation from PDT*

When a gas (normally air) pressure is applied to one side of a membrane, the gas flows through the defects in the membrane; which means that, when no gas inlet or other gas outlet is allowed, a pressure drop can be measured. When the volume and starting pressure are known, and the pressure decay measured, the flow of gas Q_{gas} can be derived from

$$\Delta P_{itest} = \frac{Q_{gas} \times t}{V_{total}} \times P_a \tag{1}$$

where ΔP_{itest} equals the result of the PDT, i.e. the pressure drop during the test t equals the time during which the pressure loss is measured in an integrity test. P_a is atmospheric pressure or the pressure at the non-pressurized side of the membrane and V_{total} equals the volume on the pressurized side of the membrane.

One should also bear in mind the fact that there will be air diffusion in an integrity test as well [8]. Air will dissolve in the pores under the higher pressure and diffuse through the pores to the lower (atmospheric) pressure side. The estimation and calculations for the flow of air diffusion ($Q_{diffusion}$) in the Multibore membrane will be presented in a further follow-up paper.

$$Q_{gas} = Q_{defect,gas} + Q_{diffusion} \tag{2}$$

where $Q_{defect,gas}$ is the flow of gas through the defects in the membrane.

The test can also be adapted so that the air flow is measured instead of the pressure decay, but in practice, this is seldom done [9].

When we define $Q_{MB,gas}$ as the flow of gas that goes through both ends of a complete Multibore membrane which is cut at the same place as where the damage occurred in the module, and under the same circumstances (pressure, temperature, etc.), then we can calculate the equivalent number of broken fibres (n) with Eq. (3).

$$n = \frac{Q_{defect,gas}}{Q_{MB,gas}} \tag{3}$$

Combining the last 2 Eqs. (2 + 3), we obtain:

$$n = \frac{(\Delta P_{itest} - \Delta P_{diffusion}) \times V_{total}}{Q_{MB,gas} \times t \times P_a} \tag{4}$$

whereby, the part of the pressure drop attributable to gas diffusion equals $\Delta P_{diffusion}$.

It is off course possible to calculate the air flow through capillaries of 0,9 mm and with varying lengths. We have done this through an online pore-flow calculator [10]. We have also measured these air flows experimentally to check the validity of the theoretical values. In these experiments, a single Multibore was used while the airflow was measured at various pressures and length of the membrane. The results of these tests, together with theoretical data, are summarized in Fig. 2.

From the graph, we can see that the experimental data compare very well with the theoretical values obtained for the longer pieces of membrane. However, for the shortest piece, at 6-cm length, the experimental value lies significantly under the theoretical value. Values with Hagen–Poiseuille lead to even higher flows compared to the experimental values due to the turbulent flows that are reached with the shorter membrane pieces. The theoretical values (Hagen–Poiseuille or the web-based calculator) would over-predict the airflow through the membrane which would lead to a lower calculated number of broken membranes. For this reason, we will only consider the experimental values for the calculations of LRV.

We can now compare the value obtained for the airflow from the PDT and compare this with the obtained experimental data on one fibre break. If we would know the position of the membrane damage, this then gives us a value for the number of broken membranes in the system. Note that this value can be lower than one, in which case, only a fraction of a membrane is completely broken.

3.3. Water flow through defects

Now, as we know the amount of damage in the membrane system (by comparing the PDT results with the airflow through a complete Multibore break), we need to translate this information into an estimate of the remaining particle-rejection capability of the membrane system or membrane rack. Again, the simple method used here uses a single Multibore membrane, whereby the water flow is measured through the capillaries of the membrane at various lengths of the MB and various pressures. The results can be seen in Fig. 3. The values here are for water at a temperature of 20°C. At lower temperatures, less water will flow through the capillaries due to the increase in viscosity.

Just like Eq. (3), where the number of completely broken membranes equals the flow of air through the membrane defects in the membrane system divided by the flow of air through a completely broken membrane, the number of broken membranes can also be calculated by comparing the water flow through a membrane defect ($Q_{\mathrm{defect,water}}$) with the flow through a completely cut Multibore ($Q_{\mathrm{MB,water}}$).

$$n = \frac{Q_{\mathrm{defect,water}}}{Q_{\mathrm{MB,water}}} \tag{5}$$

3.4. Determining the worst position for membrane damage to occur

As previously explained, when a membrane breaks, this will leave two lengths of membrane, and through both parts, there will be a flow of bypass water during filtration or air during an integrity test. When we now add the amounts of water coming through both ends of the broken membrane, and do

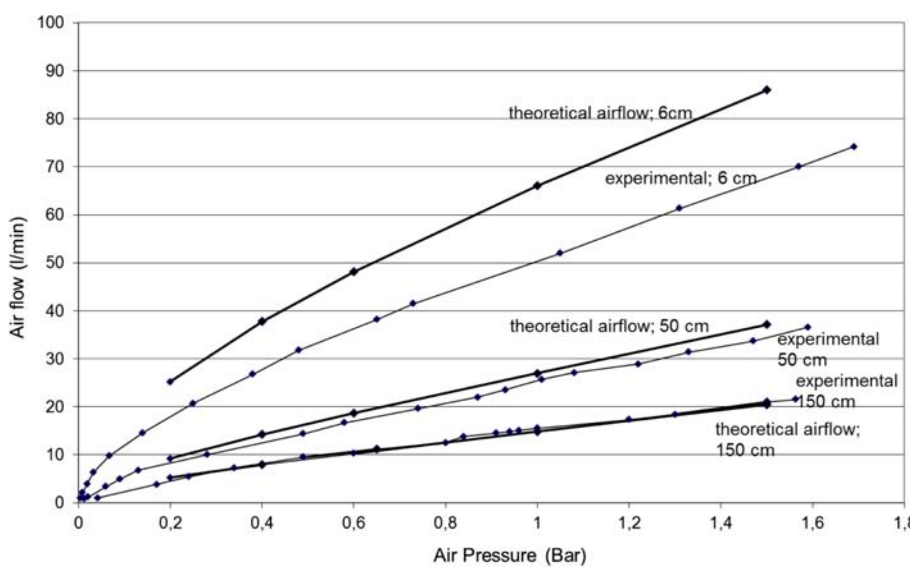

Fig. 2. Comparison between theoretical and experimental air flows through a cut-through multibore membrane.

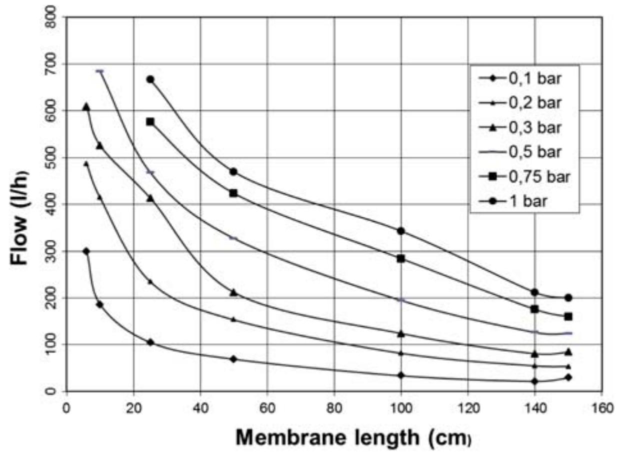

Fig. 3. Water flow through a single cut-through Multibore.

the same for the air flows in integrity testing, we obtain values depicted in Fig. 4 (where, a TMP on filtration of 0,3 bar is used as an example).

From this figure, we can see two possible problem areas for the fibre damage to occur. When the damage is situated in the middle of the module, less air will flow through the damaged membrane due to frictional losses. The corresponding pressure loss will, therefore, be lower when the damage occurs near the middle of the module. On the other hand, the bypass feed water flow through the defect is even more affected by the position of the membrane damage. This means that, although the PDT becomes less sensitive to determining how much damage there is to the membranes, the actual LRV is less affected. In short, this means that the main problem area for membrane damage is at the entrance side of the module, which could be bottom or top depending on the way the module is operated. The rest of this paper will assume that the damage occurs at the feed entrance, so can be considered as an absolute worst case scenario.

3.5. Calculations on a large membrane rack

Let us consider the implications for large membrane systems. The largest rack recommended by inge, through its T-Rack system, uses 80 Dizzer MB 0.9 XL 60 modules of 60 m^2, giving a total of 4,800 m^2 of membrane surface area. Per module, we have approximately 30 litres (20 in the capillaries and end caps plus 10 litres per module in the headers) of volume on the feed side of the rack. This gives a total of 2,400 litre. Here, we will assume a relatively low diffusion rate through integer membranes of 2 mbar/min, but please note that this is dependent on air and water temperatures.

A single completely broken Multibore will lead to a pressure drop in the integrity test of 120 mbar in 5 min, or 24 mbar per minute. This type of membrane failure would mean an immediate shut down of the plant as the predicted LRV would drop to 3,8 at medium TMP's around 0,5 bar. Membrane manufacturers often recommend a threshold value of 50-mbar pressure drop for a 5-min test. The equivalent number of completely broken fibres when this level is reached is 0,37. This is a save level, as the predicted LRV will always stay above 4 log, as we will later see.

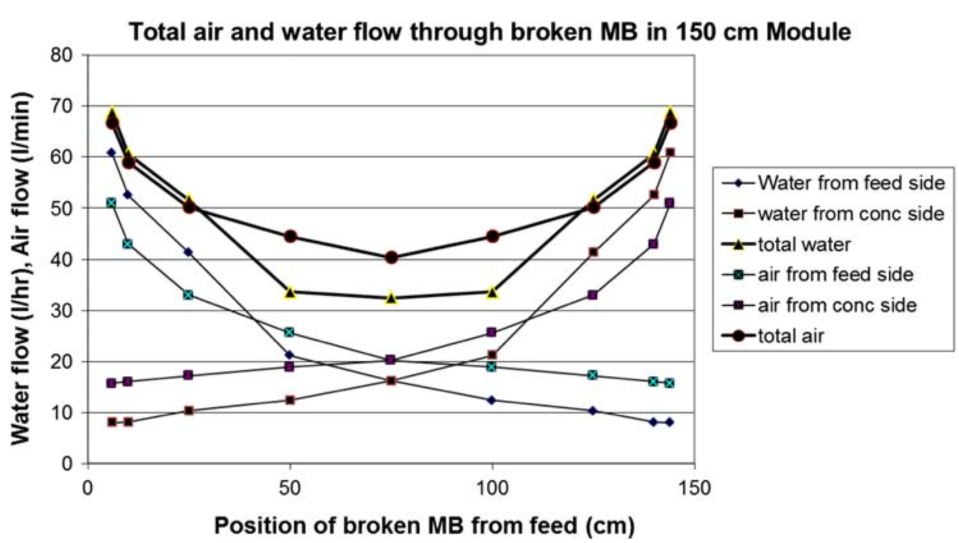

Fig. 4. Total flow of water or air through a cut multibore.

Since with a PDT we can easily detect these 0,37 broken Multibores, we will now focus on systems with less than a complete membrane breakage. This leads to the possibility of not having to look at the second end of the damaged membrane: if a membrane is only damaged, not broken, the total flow through the damage will be dependent only on the inlet pressure through the shortest length.

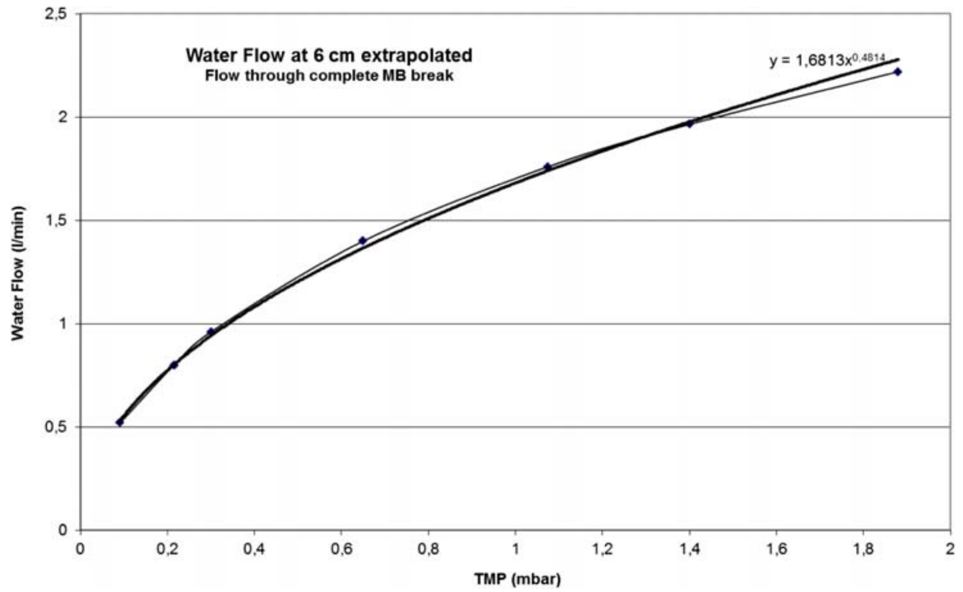

Fig. 5. Experimental water flow and power function to fit against TMP.

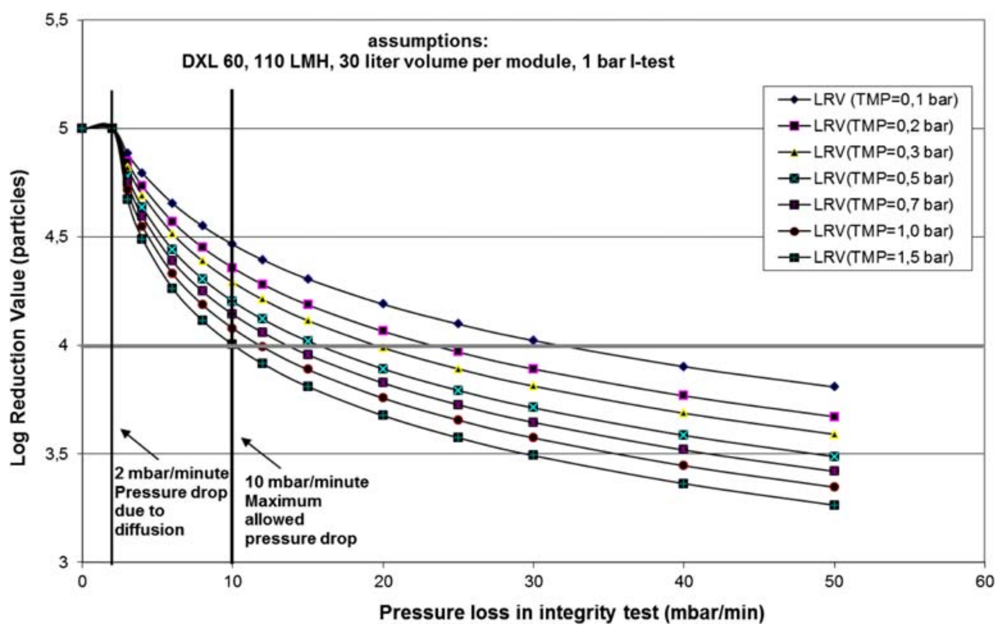

Fig. 6. LRV against pressure drop in an integrity test.

3.6. Derivation of a common formula for inge Multibore systems

No membrane, module or system is a 100% barrier to viruses or bacteria. However, most good UF membranes will deliver more than 4-log virus rejection and all membranes UF or MF should get greater than 4-log removal of bacteria. The inge membrane has often been tested and a consistent log reduction of viruses of more than 5 log has been obtained. A very high rejection rate of 9,7 log for Pseudomonas Diminuta has even been measured by an independent institute based on the ASTM method, ASTM F838-05.

In order to be able to estimate the virus rejection rate of a Multibore system, a base removal of 5 log is assumed.

$$Q_{5\log} = 10^{-5} \times Q_{\text{filt}} \tag{6}$$

where, $Q_{5\log}$ equals to equivalent flow necessary to reach a 5-log removal and Q_{filt} equals the total flow of water filtered by the membrane rack.

The LRV of a membrane system can be calculated by

$$\text{LRV} = \log_{10}\left(\frac{Q_{\text{filt}}}{Q_{\text{bypass}}}\right) = \log_{10}\left(\frac{Q_{\text{filt}}}{Q_{\text{defect,water}} + Q_{5\log}}\right) \tag{7}$$

$Q_{\text{defect,water}}$ is the defect stream through the defects identified by the PDT and follows from Eqs. (4) and (5).

$$Q_{\text{defect,water}} = \frac{\Delta P_{\text{itest}} - \Delta P_{\text{diffusion}} \times V_{\text{total}} \times Q_{\text{MB,water}}}{Q_{\text{MB,gas}} \times t \times P_{\text{a}}} \tag{8}$$

And, by combining the above Eq. (7) with Eq. (8), we obtain

$$\text{LRV} = \log_{10}\left(\frac{Q_{\text{filt}} \times Q_{\text{MB,gas}} \times t \times P_{\text{a}}}{V_{\text{total}} \times Q_{\text{MB,water}} \times (\Delta P_{\text{itest}} - \Delta P_{\text{diffusion}}) + t \times P_{\text{a}} \times Q_{\text{filt}} \times Q_{\text{MB,gas}} \times 10^{-5}}\right) \tag{9}$$

Now, we only have to know $Q_{\text{MB,water}}$ and $Q_{\text{MB,gas}}$ in order to be able to directly quantify the rejection capability of the membrane system.

$Q_{\text{MB,gas}}$ is obtained directly from the experimental gas flow in Fig. 2, and equals 50 l/min (at a PDT at 1 bar).

The value for $Q_{\text{MB,water}}$ as a function of the employed TMP can be obtained from a simple power formula which gives a good fit to the curve for the experimental values of water flow through a Multibore membrane (again at 20°C) (Fig. 5).

From this curve, the fitted power equation is:

$$Q_{\text{MB,water}} = 1.68 \times \text{TMP}^{0.48} \tag{10}$$

This now gives us the final

$$\text{LRV} = \log_{10}\left(\frac{Q_{\text{filt}} \times Q_{\text{MB,gas}} \times t \times P_{\text{a}}}{1.68 \times V_{\text{total}} \times \text{TMP}^{0.48} \times (\Delta P_{\text{itest}} - \Delta P_{\text{diffusion}}) + t \times P_{\text{a}} \times Q_{\text{filt}} \times Q_{\text{MB,gas}} \times 10^{-5}}\right) \tag{11}$$

This formula can be used to estimate the remaining rejection rate of a membrane system for viruses. If the rejection rate of bacteria is needed, one can start with a 100% rejection, as the rejection rate of an integer inge membrane system is over 9 log. This then leads to

$$\text{LRV} = \log_{10}\left(\frac{Q_{\text{filt}} \times Q_{\text{MB,gas}} \times t \times P_{\text{a}}}{1.68 \times V_{\text{total}} \times \text{TMP}^{0.48} \times (\Delta P_{\text{itest}} - \Delta P_{\text{diffusion}})}\right) \tag{12}$$

3.7. Example of estimated LRV for particles against TMP

As an example of how this works out in real membrane systems, we can look at the following figure (Fig. 6) which was compiled from data obtained with Eq. (11).

We see that even at a very high TMP of 1,5 bar, the virus LRV will still stay above 4 log, when the PDT gives a pressure drop of 10 mbar/min or less.

This clearly demonstrates the validity of the PDT and its 10 mbar/min pressure drop limit.

Please note that the actual values are dependent on the volume of the pressurized side (during integrity testing), so the actual accuracy could be lower when unusually large headers are used. They are also dependent on the actual water temperature and viscosity (the above system is calibrated at 20^{o}C). Lastly, this analysis is only valid for relatively large defects, whereby the flow through the damaged membrane is largely turbulent. When the defects are small (but numerous), the experimental values are not valid anymore. These situations will also be looked at in further publications.

4. Conclusions

The PDT gives information that can be translated into an estimate for the LRV of particles in a membrane rack. Although the PDT is performed at a pressure that can only detect defects larger than 3 micron, it gives a very good indication about virus rejection as well. The PDT has been shown to be sensitive enough to detect even less than a single broken Multibore in a large rack with 4,800 m^2 of membrane surface area. Furthermore, the test is so sensitive that the calculated resulting LRV can always be maintained above a 4 log level, independent of the TMP.

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