

Magnetic nanoparticles for UF membrane integrity: Industrial scale

H. Guo¹, Y. Wyart¹, J. Perot², F. Nauleau² and P. Moulin^{1*}

¹Université Paul Cézanne Aix Marseille, Laboratoire de Mécanique, Modélisation et Procédés Propres (M2P2 – UMR-CNRS 6181), Europôle de l'Arbois, BP. 80, Bâtiment Laennec, Hall C
13545 Aix en Provence Cedex 04, France

²SAUR, 1 avenue Eugène Freyssinet, 78064 Saint Quentin en Yvelines Cedex, France

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Abstract. An alternative ultrafiltration membrane integrity test was already developed in laboratory scale. It is based on the use of magnetic nanoparticles (Fe_3O_4) and measurement of magnetic susceptibility. The mean size of nanoparticles used is around 35 nm and they show a good disparity between 20 and 100 nm. In this paper, validation of this membrane integrity monitoring method was achieved by industrial-scale tests. Two holes with 0.6 mm internal diameter in a module containing 10 000 fibers (35 m² surface area) was efficiently detected by injecting 750 mL of 1.7 g.L⁻¹ nanoparticle solution during 2s when the test was operated at low TMP (0.096 bar, corresponding to a flux of 2.2 m³.h⁻¹). In addition, it has been demonstrated that within the detectable range, this membrane integrity test with magnetic nanoparticles has a very rapid response time. The response time depends on the permeate flux and the dead-volume of the pilot. This membrane integrity test, with the advantages of on-line operation, high detection sensitivity, detection specificity and very low influence on membrane fouling, seems to be suitable for large scale drinking water plants.

Keywords: drinking water; integrity test; magnetic nanoparticles; magnetic susceptibility; industrial scale.

1. Introduction

The most widely used membrane configuration in drinking water treatment is hollow fiber membrane filtration. In a membrane drinking water treatment plant, the detection of membrane system failures requires an effective membrane integrity test system with an acceptable frequency. As the most widely used methods, the pressure decay test (PDT) and diffusive air flow (DAF) test are considered to be reliable for monitoring membrane system integrity. The principle of the PDT is based on the measurement of pressure drop in the feed side after draining and pressurizing (Adham *et al.* 1995, Farahbakhsh and Smith 2004, Giglia and Krishnan 2008, Johnson 1998, Trimboli *et al.* 2001). DAF test is fundamentally similar to the PDT. However, rather than measuring pressure decay rate, DAF test measures the diffused gas filtrate flow or displaced water flow through the fully-wetted membrane pores when applying a constant feed side gas pressure below the bubble point of the selected whole size (Cheryan 1998, Jacangelo *et al.* 1997). The PDT and DAF test are generally proposed to be conducted according to the same sequences: 1) detecting compromised rack(s) in the plant, 2) detecting compromised module(s) in the rack and 3) detecting compromised

* Corresponding author, Professor, E-mail: philippe.moulin@univ-cezanne.fr

fiber(s) in the module. Current PDT or DAF test for hollow fiber membranes are typically set to alarm against parameters based on an absolute size removal (4 μm and greater) and a particle log removal value (4-5 LRV). Some companies set alarms against other measured variables, *e.g.*, feed turbidity, high pH in treated water. These criteria reflect the international position but do not satisfy the regulatory requirements for an absolute removal of more than 1 μm . Higher starting pressure for the test would enable smaller defects (*e.g.*, 1 μm) to be detected thereby satisfying the current requirements. However, higher start pressures may cause some membranes to experience higher defect rates than those are currently assumed. For different commercially available membranes, an annual failure rate of the membrane has been found between 1 to 10 fibers per million fibers.

The frequency of the membrane integrity test is important for maintaining specific LRV requirement. There are no common guidelines for the frequency of membrane integrity tests. Membrane integrity test frequencies need to be related to a risk assessment as the first priority and operational feasibility as the second priority. Given an expected number of fiber breaks per year and a LRV target, the frequency of membrane integrity tests can be calculated based on a simple mode or a statistical model (Bennett 2005). Membrane integrity tests can be implemented once a day, once every 72 hrs or longer, or after cleaning. It was summarized that, in most cases, a weekly test is required to ensure LRV more than 4.5. For Bristol Water, a PDT frequency of once every week to once every other week is recommended to provide a margin of safety for the target of 4 LRV (Bennett 2005). Generally, a daily test frequency would be beneficial when ascertaining system integrity, which is consistent with the continuous sampling and analysis practice. In general, the data from membrane integrity tests during operation should be kept between 2-5 years (Jackson 2001).

A perfect method for low-pressure membrane integrity monitoring should be cheap, simple, on-line and continuous. In addition, it should be reliable and highly sensitive to detect membrane breaches, even for nanoscale breaches. A summary of current different membrane integrity tests is given by Guo *et al.* (2010).

However, PDT and DAF are conducted off-line. In contrast, indirect integrity tests, such as particle counting and turbidity monitoring, are performed simply and on-line but have lower sensitivity. They are not able to detect water quality changes at the levels required to ensure pathogen removal. To realize reliable and efficient on-line membrane integrity monitoring, some other methods are proposed, including acoustic sensor method, liquid porosimetry, surrogate challenge tests, etc. All these methods have their own advantages and disadvantages. Membrane integrity tests are specific for the type of membrane used and are dependent of membrane manufacturer and membrane system supplier. In general, membrane suppliers have their own membrane integrity test procedures not more than 4-5 LRV. During practical operation, the frequency of membrane integrity tests is very important to achieve the required LRV.

So, to better satisfy the regulatory requirements of the drinking water industry, it remains urgent to develop an alternative on-line monitoring technique for quick, accurate, simple, continuous and relatively inexpensive evaluation of the low-pressure membrane (UF and MF) integrity. Based on the above information, surrogate challenge tests with nanometric material are interesting and promising because it can ensure the disinfection efficiency of the UF membrane and make it possible to realize the required objectives with the development of more accurate and advanced measurement apparatus. For this purpose, an alternative on-line UF membrane integrity test by using magnetic nanoparticles as surrogates reveals that this challenge test is suitable for large-scale drinking water applications. This new integrity test was developed (Moulin 2008). To investigate the feasibility and efficiency of this test for UF membrane integrity monitoring, a lot of experimental work have been done,

including preparation of magnetic nanoparticles, characterization of magnetic nanoparticles, lab-scale membrane integrity tests (Guo *et al.* 2010). This membrane integrity test, with the advantages of on-line operation, high detection sensitivity, detection specificity and very low influence on membrane fouling, seems to be suitable for large scale drinking water plants. Using the surrogates with nanometric size, this method makes it possible for UF membrane system to better satisfy the regulatory requirements of the drinking water industry.

In this paper, the validation of this test is investigated at an industrial scale.

2. Material and methods

2.1 Membrane and pilot plant

To further validate the relevance of this new membrane integrity test for large-scale application, industrial-scale tests were carried out in the Jaunay drinking water treatment plant of the SAUR group in France. As shown in the experimental set-up (Fig. 1), two membrane modules are connected in series. The left one is an impaired module and the right one is an intact module. The filtration process with one or both of these two modules can be controlled by the valves in the system. Here, all the filtration tests were operated with dead-end mode under very low industrial transmembrane pressures (0.08-0.13 bar). The permeate flux of one module is about $2.1\text{--}3.5\text{ m}^3\cdot\text{h}^{-1}$. Norit X-flow membranes are used for industrial-scale tests. The characteristics of the membrane used here are shown in Table 1. While for industrial-scale tests, a membrane module with 10 000 fibers and 1.5 m length (membrane area = 35 m^2) is used. The membrane modules should be well rinsed and then be characterized by water permeability measurement prior to membrane integrity tests. The permeability of this membrane is around $1\text{ 000 L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}\cdot\text{bar}^{-1}$. For the impaired membrane module containing 10 000 fibers, two defects with 0.6 mm internal diameter in the module have been intentionally made.

2.2 Suspensions and apparatus

The membrane integrity tests are operated by injecting a given amount of challenging materials (Fe_3O_4 nanoparticles or else) as shown in Fig. 1. Firstly, these two membrane modules are challenged with high concentration of powder activated carbon (PAC) solution simultaneously to investigate whether the PAC can efficiently detect the presence of the impaired membrane. The PAC used here has a size range from 2 to $125\text{ }\mu\text{m}$ and 75% of these particles are in the range from 2 to $4\text{ }\mu\text{m}$, resulting in a quantity of $69\text{ 000--}70\text{ 000 particles}\cdot\text{mg}^{-1}$. Permeate of these two modules are then analyzed with a water particle counter WPC 2000 (ART instruments, Inc. USA), which counts the particles greater than $2\text{ }\mu\text{m}$. Then, the impaired membrane module is challenged by different amount of Fe_3O_4 nanoparticles with different injection modes. And the permeate is analyzed with the two magnetic susceptibility meters to investigate the efficiency of this new membrane integrity test to detect the presence of these two holes out of 10 000 fibers (35 m^2 surface area).

After synthesis and stabilization (Guo *et al.* 2010), the prepared Fe_3O_4 nanoparticles are characterized in terms of particle size, particle number, concentration and magnetic susceptibility because these nanoparticle characteristics are closely related to the application for membrane integrity test. Here, different apparatus are used to characterize the nanoparticles, including granulometer, spectrophotometer and magnetic susceptibility meter. In electromagnetism, the magnetic susceptibility is the degree of

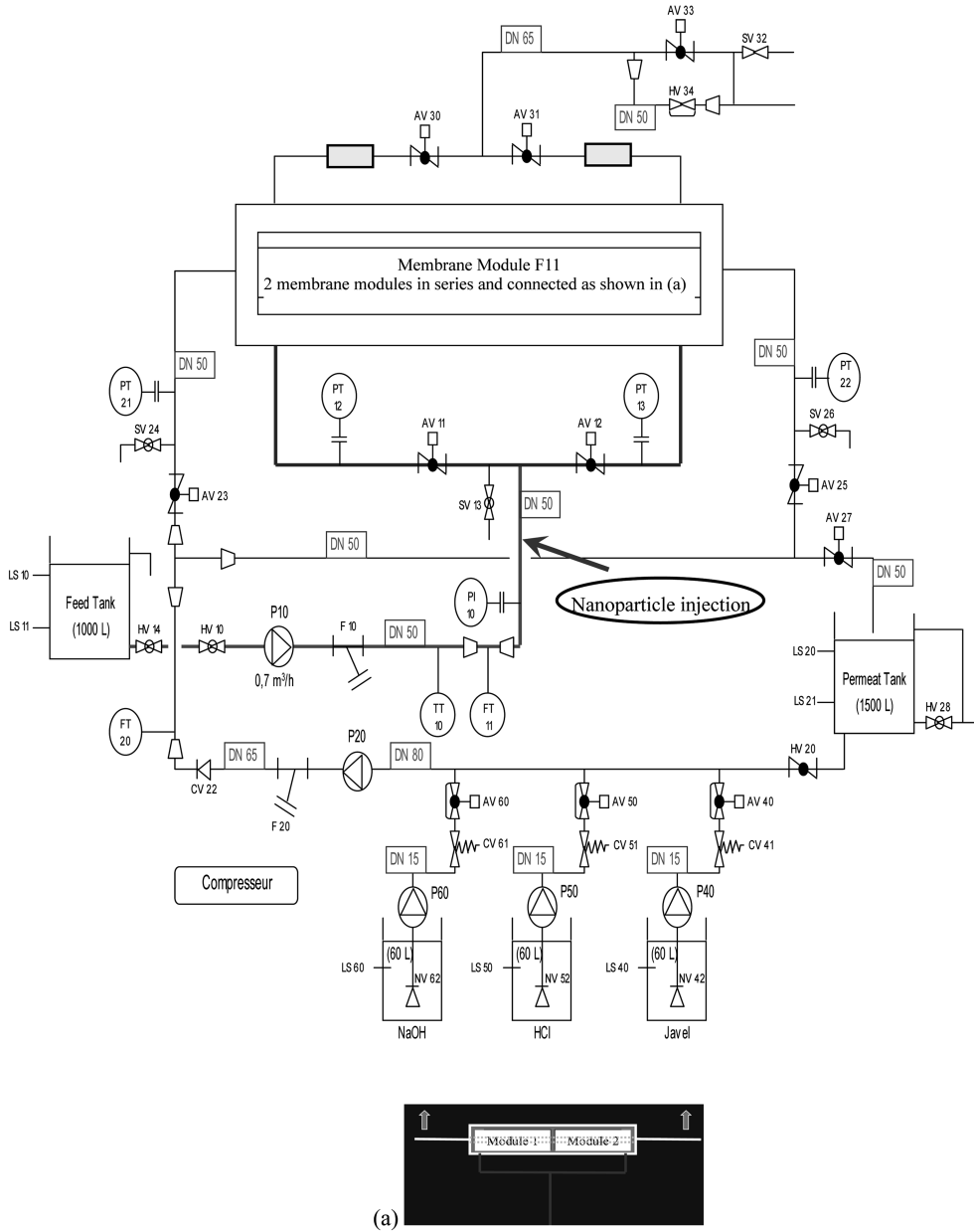


Fig. 1 Industrial-scale experimental set-up for UF membrane integrity tests in Jaunay

magnetization of a material in response to an applied magnetic field. The magnetic susceptibility can be expressed as volume magnetic susceptibility, mass magnetic susceptibility and molar magnetic susceptibility. The volume magnetic susceptibility χ_v is defined by the relationship (1)

$$M = \chi_v H \quad (1)$$

where χ_v is in SI unit, M is the material magnetization (the magnetic dipole moment per unit

Table 1 Characteristics of the Norit X-flow membrane used

Membrane type	Hollow fiber membrane
Internal diameter of fiber	0.8 mm
Material	Polyethersulfone (PES) + Polyvinylpyrrolidone (PVP)
Membrane cut-off	150 kDa
Nominal pore size	25 nm

volume, A.m^{-1}) and H is the magnetic field strength (A.m^{-1}). If χ_v is positive, the material can be paramagnetic, ferromagnetic, ferrimagnetic or antiferromagnetic. In this case, the magnetic field is strengthened by the presence of the material. Alternatively, if the χ_v is negative, the material is diamagnetic (*e.g.*, water) and the magnetic field is weakened in the presence of the material. Here, the magnetic susceptibility of Fe_3O_4 nanoparticles is measured by means of magnetic susceptibility meters. Two apparatus are used, including a Bartington magnetic susceptibility meter equipped with a MS2B sensor (Bartington Instrument, UK) and an AGICO Kappabridge magnetic susceptibility meter (MFK1-FA, AGICO Instrument, Czech Republic). For this membrane integrity monitoring technique, the application of Fe_3O_4 nanoparticles is always based on the water-based diluted solution. As a result, the volume magnetic susceptibility is preferred here since it is only related to the sample volume, independent of the density. Moreover, it is simpler to analyze and compare the samples with the same volume (10 mL) and to know whether nanoparticles can be detected. The volume magnetic susceptibility of Fe_3O_4 nanoparticles is obtained by subtracting magnetic susceptibility of water from that of water and nanoparticles. As the volume magnetic susceptibility of water is negative ($\approx -0.9 \times 10^{-5}$ SI unit), calculated positive values indicate the presence of Fe_3O_4 nanoparticles in the solution.

Of these two magnetic susceptibility meters, the Bartington instrument is a portable apparatus. It can display values from 0.1 to 9 999 (*e.g.*, $(0.1 \sim 9\,999) \times 10^{-5}$ SI unit for volume magnetic susceptibility), providing a very wide analytical range to detect magnetic material. Generally, the response time for measuring a sample is 2 s or 12 s, depending on the selected measurement range. In contrast, the AGICO Kappabridge magnetic susceptibility meter has the same advantages of a very wide analytical range and rapid detection. Moreover, it offers more detection sensitivity (the minimum detectable value is 20×10^{-8} SI unit). However, the Kappabridge magnetic susceptibility meter is very susceptible to the changes of operational conditions, such as temperature, humidity and the presence of iron in the room (pump, phone, etc.). As a result, it is ideal that the temperature and humidity in the room where the Kappabridge is installed are stable.

3. Results

3.1 PAC injection

In this part, industrial-scale membrane integrity tests with high concentration of PAC suspension and stabilized magnetic nanoparticles have been carried out to investigate the efficiency of this new integrity test. In addition, for the membrane integrity test with PAC particles, a high concentration of PAC suspension (20 g.L^{-1}) was injected into the membrane system using a peristaltic pump at a flux of 3.24 L.h^{-1} for 10 min. Here, the PAC suspension was injected into the impaired membrane

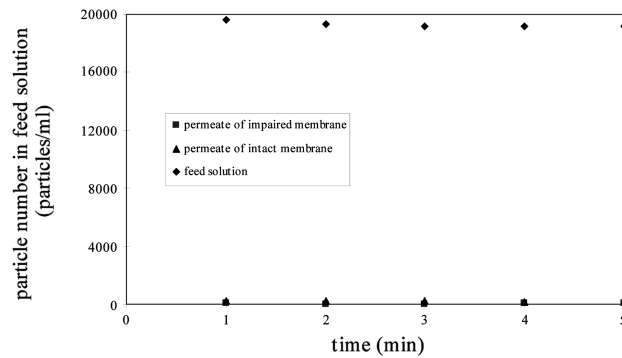


Fig. 2 Variation of particle number in feed solution and in permeate as a function of filtration time with the impaired and intact modules for PAC injection

module and the intact one simultaneously. This membrane integrity test was operated at a TMP of 0.112 bar and the flux of $1.2 \text{ m}^3 \cdot \text{h}^{-1}$ for each module. For both modules, the permeate samples were taken continuously every minute during the last 5 min of the PAC injection. As shown in Fig. 2, with the high concentration of PAC in feed solution (more than $69\,000 \text{ particles} \cdot \text{mg}^{-1}$), the PAC number in permeate of the intact module was more than that of the impaired one. The particle counting is non-specific, *i.e.*, it counts all the particles related to a certain size range. Here, the counted particles refer to those larger than $2 \mu\text{m}$. In fact, the particle number in permeate of the intact module acts as a background value, because the PAC particles with the size range of $2\text{--}125 \mu\text{m}$ can be completely retained by the intact membrane. The particle number in permeate of the impaired module less than the background value actually indicates that there is no difference between the intact and impaired modules. As a result, the impaired module with two holes with 0.6 mm internal diameter which locate in two fibers out of $10\,000$ fibers cannot be detected by high concentration of PAC challenge test combined with particle counting.

3.2 Magnetic nanoparticle suspension

Then, industrial-scale magnetic nanoparticle challenge tests for membrane integrity monitoring were carried out with instantaneous nanoparticle injection using a $1.7 \text{ g} \cdot \text{L}^{-1}$ stabilized nanoparticle suspension. No magnetic nanoparticles are detected in the permeate of the intact module. To efficiently detect the presence of the membrane compromise, different amounts of nanoparticles were injected with different injection modes. All these tests were operated on the impaired membrane module and magnetic susceptibility in permeate was analyzed. The permeate samples were taken immediately

Table 2 Operational conditions of industrial-scale membrane integrity tests with instantaneous stabilized nanoparticle injection: [initial suspension] = $1.7 \text{ g} \cdot \text{L}^{-1}$

	Test 1	Test 2	Test3	Test 4
TMP (bar)	0.121	0.096	0.084	0.096
Flux ($\text{m}^3 \cdot \text{h}^{-1}$)	2.5	2.1	2.2	2.2
Nanoparticle injection	1 min at a flux of $20 \text{ L} \cdot \text{h}^{-1}$	100 mL in 4 s	100 mL in 1.5 s	0.75 L in 2 s
Time for one sample	15 s	5 s	5 s	5 s

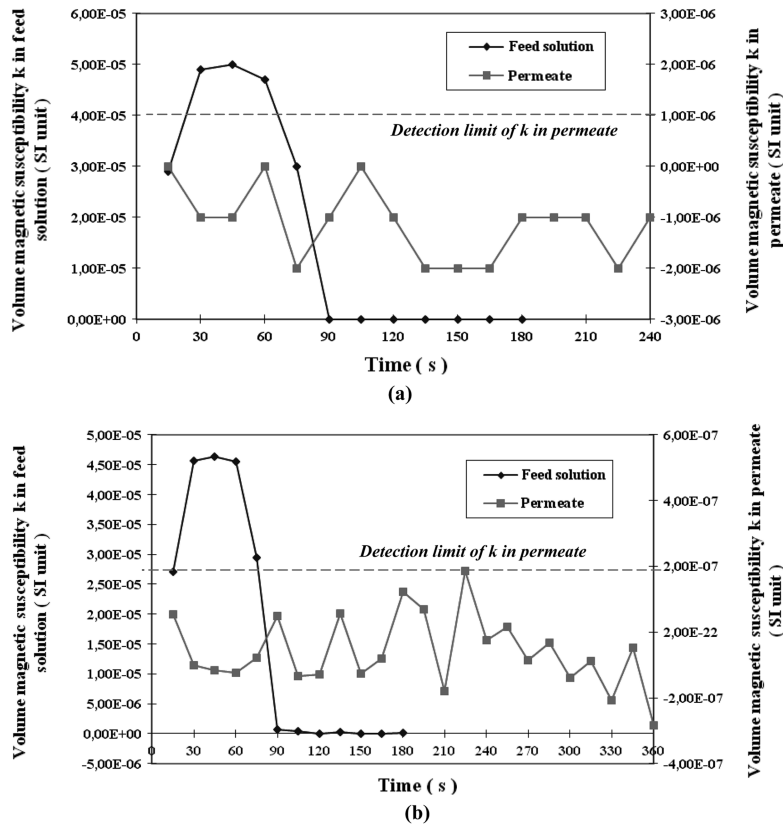


Fig. 3 Variation of volume magnetic susceptibility in feed suspension and in permeate as a function of filtration time: (a) Bartington and (b) Kappabridge apparatus [Dead-end filtration, TMP = 0.121 bar, permeate flux = $2.5 \text{ m}^3 \cdot \text{h}^{-1}$, 333 mL of nanoparticles injected in 1 min, [initial suspension] = $1.7 \text{ g} \cdot \text{L}^{-1}$]

following the nanoparticle injection. The operating conditions of each test are listed in table 2.

The results of test 1 are shown in Fig. 3. For this test, 1 min of nanoparticle injection means that 333 mL of $1.7 \text{ g} \cdot \text{L}^{-1}$ nanoparticle suspension was injected into the membrane system in 1 min. In this case, the nanoparticle concentration in feed suspension increased in the first 45 s and then decreased rapidly. The nanoparticle feed concentration was close to 0 just in 30 s after the end of nanoparticle injection (shown in both Fig. 3 (a) and (b)), which was due to the rapid dilution effect under the condition of instantaneous nanoparticle injection. This rapid change of feed concentration determines the short duration of membrane integrity test needed even if the nanoparticles in permeate can be detected due to the presence of membrane compromises. Based on the relationship between the nanoparticle concentration and magnetic susceptibility, the maximum nanoparticle feed concentration in test 1 corresponding to the maximum magnetic susceptibility in feed suspension of about 5.0×10^{-5} SI unit is about 12 ppm. This feed concentration is not enough to efficiently detect the presence of two holes with 0.6 mm internal diameter out of 10 000 fibers (35 m^2 surface area). It can be seen that no reliably positive magnetic susceptibility in permeate was detected by both Bartington (Fig. 3 (a)) and Kappabridge magnetic susceptibility apparatus (Fig. 3 (b)).

Similar results were also obtained in test 2 (Fig. 4). In this case, although a smaller amount of nanoparticles (100 mL) were injected, the feed concentration is higher than that in test 1 due to the

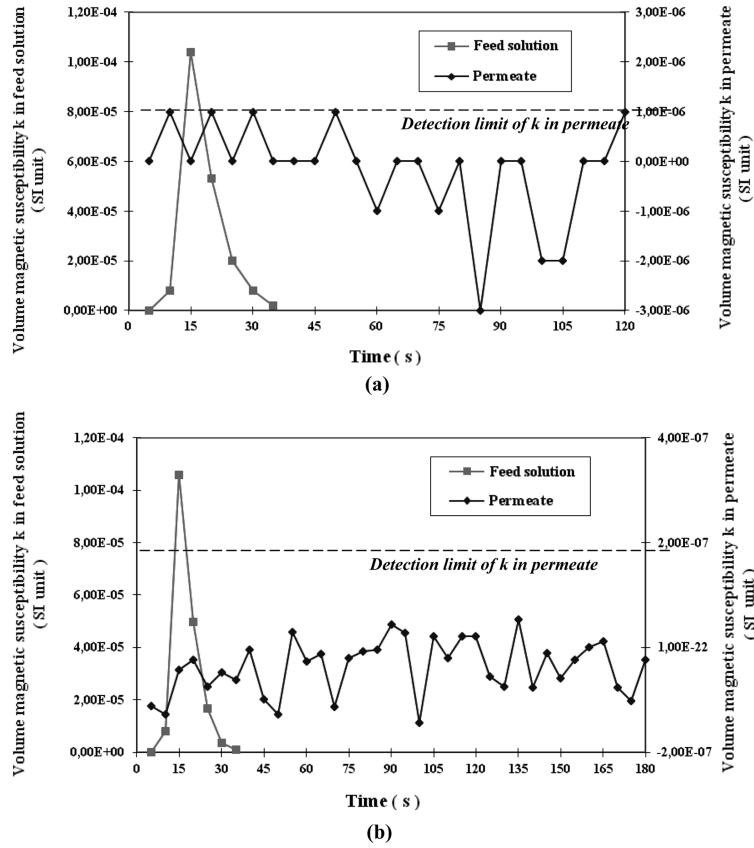


Fig. 4 Variation of volume magnetic susceptibility in feed solution and in permeate as a function of filtration time: (a) Bartington and (b) Kappabridge apparatus [Dead-end filtration, TMP = 0.096 bar, permeate flux = $2.1 \text{ m}^3 \cdot \text{h}^{-1}$, 100 mL of nanoparticles injected in 4 s [initial suspension] = $1.7 \text{ g} \cdot \text{L}^{-1}$]

quicker nanoparticle injection (100 mL in 4 s). The maximum nanoparticle feed concentration here was about 26 ppm. The rapid change of feed concentration was also observed in this case. The duration of detectable nanoparticles in feed solution was about 30 s. However, this nanoparticle injection cannot detect the presence of the impaired module neither. Magnetic susceptibility in permeate fluctuated in the range of $-0.3 \times 10^{-5} - 0.1 \times 10^{-5}$ and $-2.0 \times 10^{-7} - 1.0 \times 10^{-7}$ SI unit when using the Bartington and Kappabridge apparatus, respectively. These results showed that 100 mL of $1.7 \text{ g} \cdot \text{L}^{-1}$ nanoparticle injection in 4 s cannot efficiently detect the presence of such membrane compromises when the membrane module is operated under the low TMP (0.096 bar).

Even when 100 mL of nanoparticles was injected more quickly (in 1.5 s) as shown in Fig. 5 (test 3), magnetic susceptibility in permeate analyzed with the Bartington apparatus showed the similar results as those obtained in test 2. This indicates that the impaired module cannot be detected in this case by the use of the Bartington apparatus. In contrast, the results analyzed with the Kappabridge apparatus showed an interesting phenomenon between 30 s and 125 s. In this range all the magnetic susceptibility in permeate were positive values but mostly in the range of $0 - 2 \times 10^{-7}$ SI unit. Considering the efficient detection limit of the Kappabridge apparatus (20×10^{-8} SI unit), this nanoparticle injection with $2.2 \text{ m}^3 \cdot \text{h}^{-1}$ flux does not indicate the reliable nanoparticle detection in

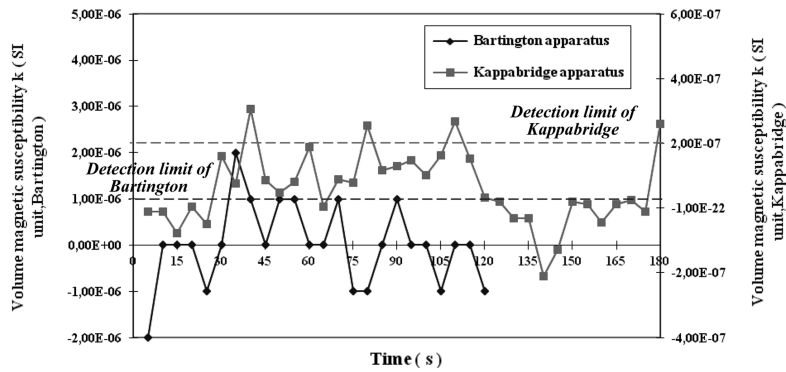


Fig. 5 Variation of volume magnetic susceptibility in permeate as a function of filtration time [Dead-end filtration, TMP = 0.084 bar, permeate flux = $2.2 \text{ m}^3 \cdot \text{h}^{-1}$, 100 mL of nanoparticles injected in 1.5 s, [initial suspension] = $1.7 \text{ g} \cdot \text{L}^{-1}$]

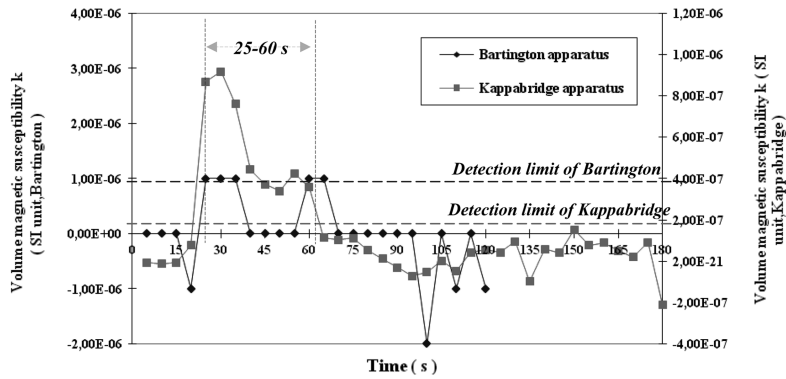


Fig. 6 Variation of volume magnetic susceptibility in permeate as a function of filtration time [Dead-end filtration, TMP = 0.096 bar, permeate flux = $2.2 \text{ m}^3 \cdot \text{h}^{-1}$, 750 mL of nanoparticles injected in 2 s, [initial suspension] = $1.7 \text{ g} \cdot \text{L}^{-1}$]

permeate due to the presence of membrane compromises. But obtained continuous positive magnetic susceptibility values indicate that this nanoparticle injection is close to the detection limit of the Kappabridge apparatus to efficiently detect the two holes out of 10 000 fibers under the flux of $2.2 \text{ m}^3 \cdot \text{h}^{-1}$. In this case, the maximum nanoparticle feed concentration was about 57 ppm (corresponding to 5 s for a sample). In test 4, more nanoparticles (750 mL) were injected into the impaired membrane in very short time (2 s). The maximum nanoparticle feed concentration with this nanoparticle injection mode was about 400 ppm (based on 5 s for one sample). As shown in Fig. 6, the impaired module could not be detected by the Bartington apparatus as former cases. When using the Kappabridge apparatus, the magnetic susceptibility in permeate was continuously positive in the range of 25-85 s. Especially between 25 s and 60 s, all the magnetic susceptibility in permeate were higher than 20×10^{-8} SI unit, indicating the efficient nanoparticle detection in permeate by the Kappabridge apparatus. In the detectable range, the magnetic susceptibility in permeate increased to the maximum value and then decreased corresponding to the change of feed concentration. This change tendency is similar to that obtained in the residual time test with the use of $200 \text{ g} \cdot \text{L}^{-1}$ NaCl solution

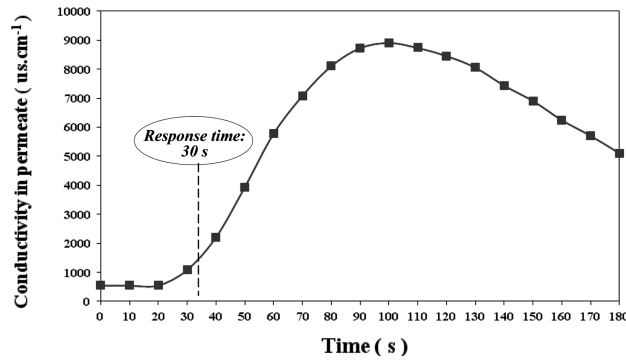


Fig. 7 Variation of conductivity in permeate as a function of filtration time [TMP = 0.109 bar, permeate flux = $2.1 \text{ m}^3 \cdot \text{h}^{-1}$, $200 \text{ g} \cdot \text{L}^{-1}$ NaCl solution ($204\,000 \text{ } \mu\text{s} \cdot \text{cm}^{-1}$)]

($204\,000 \text{ } \mu\text{s} \cdot \text{cm}^{-1}$) and conductivity measurement in permeate (Fig. 7). As a result, this membrane integrity test successfully realized the detection of two holes with 0.6 mm internal diameter out of 10 000 fibers (35 m^2 surface area) when injecting 750 mL of $1.7 \text{ g} \cdot \text{L}^{-1}$ nanoparticle suspension and using the Kappabridge apparatus.

Additionally, compared Fig. 6 with Fig. 7, it can be seen that the response time of efficient detection in permeate for nanoparticles and conductivity (NaCl) is about 25 s and 30 s, respectively, which are almost the same. It should be noted that the response time in the case of NaCl injection test depends on the dead volume between the injection point and hole location and the permeate flux. In these two cases (nanoparticle and NaCl tests), the flux are almost the same. So, it can be concluded that, within the detectable range, this membrane integrity test with magnetic nanoparticles has very rapid response time, which only depends on the dead volume of the pilot and the permeate flux. The pilot was backwashed at a pressure of 0.49 bar after the four nanoparticle integrity tests and the backwashing samples were taken continuously at the same time (every minute). The magnetic susceptibility of backwash solution showed that very weak magnetic susceptibility was detected by the Kappabridge apparatus. After a backwash, it can be concluded that the magnetic nanoparticles in the membrane module caused by the membrane integrity test can be removed by backwash.

4. Conclusions

As shown in above industrial-scale tests, efficient detection for given membrane compromises using this membrane integrity test depends on the permeate flux and nanoparticle injection mode as well as the required injected nanoparticle amount and the analytical apparatus. The preciser apparatus (Kappabridge apparatus) results in higher detection sensitivity. The injected nanoparticle amount, nanoparticle injection mode and the permeate flux determine the nanoparticle dilution factor in membrane feed suspension, thus determining the nanoparticle detection in permeate due to the presence of membrane compromises. The TMP during membrane integrity tests have two opposite effects on the detection of membrane integrity changes. On the one hand, higher TMP results in higher ratio of the permeate flux passing through the impaired fibers to that passing through the intact ones, which favors the nanoparticle detection in permeate. On the other hand, the more permeate flux due to the higher TMP contributes to more injected nanoparticle dilution, weakening

the nanoparticle detection in permeate. Therefore, the influence extent of TMP on the nanoparticle detection in permeate depends on the size of the membrane compromise. Above industrial-scale tests were operated at low TMP (0.084-0.121 bar, corresponding permeate flux of 2.1-2.5 m³.h⁻¹). Under these conditions, this integrity test is valid to detect two holes with 0.6 mm internal diameter out of 10 000 fibers (35 m² surface area) when 750 mL of 1.7 g.L⁻¹ nanoparticle suspension is injected in 2 s. These industrial-scale tests further demonstrate the validation of this new membrane integrity test for UF membrane drinking water production.

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List of symbols and acronyms

TMP	TransMembrane Pressure
PDT	Pressure Decay Test
DAF	Diffuse Air Flow
LRV	Log Removal Value
UF	Ultrafiltration
MF	Microfiltration
PAC	Powder Activated Carbon
WPC	Water Particle Counter
χ_v	Volume magnetic susceptibility (SI unit)
M	Material magnetization (the magnetic dipole moment per unit volume, A.m ⁻¹)
H	Magnetic field strength (A.m ⁻¹)