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# Development of a new method for measuring the abrasive potential of water: risk of membrane failure in water treatment plants

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## ABSTRACT

The objectives of this study were to develop an analytical method to distinguish feed water used to produce drinking water, with varying concentrations of suspended solids, in terms of abrasiveness and to define an index that can assess the abrasive potential of the feed water coming in contact with a polymeric membrane. For such process configurations, membrane abrasion has been identified as one of the most recurring and major concerns in operation because the polymeric materials used in treatment plants are relatively sensitive to abrasion. Five different types of apparatus were benchmarked and were evaluated on their ability to be adapted to particles commonly found in most drinking water treatment plants at low concentrations. After comparing 10 criteria, the MCR302 with a tribological cell of Anton Paar was identified as the most relevant device. For the selected tool (MCR302), a statistical approach was used to provide a safe and robust ranking of the abrasive potential of the different types of water. An analysis of variance allowed the origin of the result variability to be explained. The newly developed methodology enables quantification of the abrasive potential of natural waters used for membrane filtration with a relevance of ranking higher than 90%.

**Key words** | abrasive potential, drinking water, membrane, microfiltration, ultrafiltration

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## INTRODUCTION

Hollow fibres made of resistant material are used for the production of drinking water from natural waters (river, lake) with or without pre-treatment. They are used to produce drinking water or to protect the reverse osmosis process for sea water desalination (Brehant *et al.* 2002). Nowadays, there is a tendency to decrease the number of treatment steps in order to reduce the capital costs of water production facilities, thus exposing low pressure membranes (i.e. microfiltration (MF) and ultrafiltration (UF)) to waters increasingly concentrated in particles. Membrane abrasion or erosion has been identified as one of the most recurring and major operational concerns because most of the polymeric materials used in plants are relatively sensitive to mechanical deterioration. Despite the fact that membrane chemical ageing (Arkhangelsky *et al.* 2007; Regula *et al.* 2013, 2014; Ravereau *et al.* 2016) and erosion phenomenon on polymeric (Anderson 1982; Hojo *et al.* 1986; Madsen 1989; Stack & Pungwiwat 1999; Yabuki *et al.* 2000; Barkoula & Karger-

Kocsis 2002; Samyn *et al.* 2006) or metallic dense materials (Khruschov 1974; Madsen 1989; Cicek *et al.* 1999; Stack & Pungwiwat 1999; Tian & Addie 2005; Das *et al.* 2006; Ramachandra & Radhakrishna 2006; Fowler *et al.* 2009; Abouelkaseem 2011) have been widely studied, few studies deal with membrane abrasion. The filtration backwash cycles combined with the erosion phenomenon due to particles in natural waters induce membrane mechanical ageing. Membrane end-of-life is linked to the loss of the membrane's physical integrity with a succession of mechanical deteriorations. Consequently, the life expectancy of membrane processes is reduced and the membrane's separation properties as well as integrity are no longer guaranteed.

In the reported research works on material abrasion, the mechanical and chemical characteristics of the suspended solids contained in the waters or of the material in contact with water have a significant impact on the produced wear. As the phenomenon is multifactorial, each study has

focused on specific experimental conditions without following a generic approach. Nevertheless, some trends have been reported. For suspensions, different parameters must be taken into account: type, concentration, size and form of particles, impact angle, etc. (Hutchings 1992). The surface/material degradation depends on sharpness and hardness between particles and the target materials. Moreover, the mechanisms of erosion for different impact angles or particle velocities have been studied in relation to ductile or brittle materials (Barkoula & Karger-Kocsis 2002) identifying that erosion by a suspension was twofold: erosion at normal impact angles and erosion at oblique impact angles. These were named respectively deformation wear for normal angles and cutting wear for angles near  $0^\circ$  (Bitter 1963). Structure, composition and geometry can be involved in the membrane wear. Generally, erosion results in a similar effect on the membrane, whatever the particle impact angle geometry is. But, when a particle has an impact angle near  $0^\circ$ , erosion is called abrasion (Barkoula & Karger-Kocsis 2002). Furthermore, the authors have studied this phenomenon but never the capability of a suspension to be aggressive mechanically. Some tribology studies have used erosive suspensions. One can notice different tribological assays on dense materials like polymers: sphere-on-prism, block-on-ring, block- or cylinder-on-plate and pin-on-disc (Samyn *et al.* 2006). For the characterization of the erosive potential of suspensions (slurry), different configurations were developed such as the 'slurry jet test apparatus' (Iwai & Nambu 1997; Iwai *et al.* 2006, 2009), 'Coriolis erosion tester' (Xie *et al.* 1999; Tian *et al.* 2007) or the 'slurry pot tests' (Clark & Hartwich 2001). However, the slurries used were synthetic without any representative conditions of the membrane process in terms of composition, concentrations, size and geometry. Some authors studied the phenomenon of membrane erosion from different perspectives. Siembida *et al.* (2010) considered this phenomenon good for polyethersulfone flat sheet membranes because the fouling layer was reduced significantly without chemical cleaning in wastewater treatment. For Cicek *et al.* (1999), the ceramic membrane was deteriorated by inorganic crystals in the wastewater, which generated a decrease of performance and structural changes of the membrane active filtration layer. Lai *et al.* (2014a, 2014b) suggested a methodology to evaluate and follow the mechanical degradation of PVDF/nanoclay hollow fibre membranes by suspensions. This method was used on different polyvinylidene difluoride (PVDF) membranes to follow the mechanical resistance against silicon carbide suspensions (Lai *et al.* 2014b; Ji *et al.* 2015). With this method, Lai

*et al.* (2014a, 2014b) considered several aspects which could modify the representative of the response linked to the membrane operating conditions: geometry of the system to identify the erosion or abrasion contribution (Lai *et al.* 2014b). The previous studies showed that the water erosive or abrasive potential was never considered with representative conditions. Low pressure membrane systems including MF and UF membranes are used increasingly in water treatments due to their high removal level against particles or bacteria. Capital investment related to MF/UF membranes increased by 30% worldwide between 2013 and 2015. In 2018, it will reach USD362 million, a 75% increase compared to 2013. Therefore, it is necessary to qualify the composition of water to define the appropriate pre-treatment and to estimate the membrane lifetime: the more the abrasive potential of the water increases, the shorter the membrane lifetime will be.

The aims of this study are the identification and development of an analytical method to distinguish the abrasive potential of different water resources to produce drinking water using natural and representative samples. The methodology consisted in benchmarking five different types of apparatus already commercialized for other applications such as rock excavation, manufacturing of toothpaste (measuring slurry abrasion) or tribology analysis. They were evaluated on their ability to adapt to particles generally present in most drinking water treatment plants at low concentrations (abrasive particles, powdered activated carbon, and mud). The newly developed methodology aims to quantify the abrasive potential of natural waters used for membrane filtration through an abrasiveness index.

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## MATERIALS AND METHODS

### Equipment assessed

The abrasive potential is generally studied in industry by following a pragmatic approach. The equipment allows a measurement of the abrasive potential. For each device, the surface of the movable element wears and fixed support abrasion in rubbing are most of the time submerged in the water during the assessment. Table 1 shows the different selected equipment with their applications.

### Abrasion monitored by gravimetric measurement

Gravimetric-based instruments measure the abrasive potential using the mass loss of movable elements. Taber

**Table 1** | List of equipment assessed

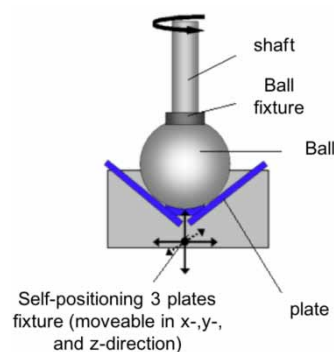
Supplier	Model	Typical sample	Activity	Measure of abrasivity (number of repetitions)	Volume slurry sample [mL]
Anton Paar S.A.S., France	MCR302 Rheometer with tribological cell	Liquids, pastes	Research	Dynamometric strength (3)	5
Brant Industries, France	Humid abrasivemeter – Ref 903	Liquids	Quality	Mass lost (3)	100
Elcometer, France	Elcometer 1720 Abrasion and Washability Tester	Liquids, materials	Quality	Mass lost (3)	50
Falex Corporation, USA	FALEX Miller Number Slurry Abrasivity Test Machine	Suspension	Quality	Mass lost (1)	300
Taber Industries, USA	5500 Multi-Media Abraser	Suspension, powder, paste	Quality	Mass lost (2)	500

Industries (United States) suggested their 5500 multimedia abraser model suitable for multiple types of samples (paints, pigments, adhesives, sealants, epoxies, pastes, detergents and industrial additives). It tests abrasion with brass pins in rotation on a stainless steel wear disc. The supplier suggested an evaluation with 2,000 rounds of a movable element at a speed of 72 rounds  $\text{min}^{-1}$ . Falex Corporation (United States) is seen as a reference in the determination of slurry abrasivity with the development of a standard test method meeting the requirements of ASTM G75 ‘Standard Test Method for Determination of Slurry Abrasivity (Miller Number) and Slurry Abrasion Response of Materials (SAR Number)’. The Miller Machine from Falex Corporation allows determination of the Miller number using specific elements abraded in a back and forth movement. For this study, Falex recommended a linear moving distance of 203.2 mm with a back and forth frequency of 48/min for 6 hours.

The Brant 930 (Brant industrie, France) and K 1720 (Elcometer, United Kingdom) devices enable the evaluation of liquid or paste abrasivity such as detergent or washing liquid. These devices are used generally to evaluate the resistance of a selected support (metal, painted substrate, plastic) with a selected liquid and a given movable element, such as brush, duster or steel wool. For a direct use, they were not adapted because the suspension volume could not be controlled, and the abraded sample could not be weighed so modification was necessary. To compare the different devices, a movable element wear was developed on the basis of the Falex wear block (see ‘Element wear’ section). For both tools, a back and forth frequency of 37/min. was set until a cumulative number of 6,000 was completed in a back and forth movement. The assay’s length was 100 mm for the Elcometer equipment and 150 mm for the one from Brant Industries.

### Abrasion followed by dynamometric measuring

The MCR302 Rheometer with a tribological cell (Anton Paar France S.A.S.) is equipment adapted for research work. The MCR302 Rheometer consists of a torquemeter with a resolution of  $0.1 \cdot 10^{-9}$  N m and can impose a maximal torque of 0.2 N m with a normal force controlled between 0.005 and 50 N on geometry adapted for the tribological cell. The side view is visible in Figure 1. Anton Paar’s apparatus allows monitoring of the assay using dynamometric measurement. At the beginning of the measurement, a normal force was applied at 10 N for 10 s. Then, the speed rotation of the ball increased progressively from 0.1 to 3,000 rounds per minute. Twenty intermediate points were measured with a log-distribution. To compare the assays, the measurements were represented as a function of the sliding speed. The abrasive potential of the suspension was assimilated to the measurement of the friction coefficient. For each analysis, steel balls and plates were changed to obtain independent data. Under the test conditions, the



**Figure 1** | Schematic side view of the tribological cell of MCR302 Rheometer.

plates used were composed of two materials: steel or Teflon (ball was always steel). The analysed suspensions acted as a lubricant between the two surfaces. Thus, the chemical composition of the liquid phase and the composition of the solid phase with the particles modulate the measurements of the friction coefficient (Khonsari *et al.* 1989; Lordanoff *et al.* 2008).

## Element wear

The whole movable element wear was used for one analysis only to avoid the effect of disturbances between analyses. Except on Anton Parr's equipment, the measurement of abrasivity was performed with the weighted measurement of the movable element wear. The abrasive potential was determined with the 27% chrome iron wear block for the Falex abrasive test machine. With the equipment from Taber Industries, the movable element wear was a brass pin. But neither Brant Industries nor Elcometer equipment could be used without modification. A movable element wear was developed to remain fixed on the equipment moving cage. It was based on the reference equipment in the determination of slurry abrasivity with Falex Corporation's equipment and the standard method G75 (GO2 committee 2007). Polymethyl methacrylate (PMMA) plates with a 40 × 30 mm side and 5 mm thick were cut (Plexiglas® GS 0F00 Incolore, ThyssenKrupp Cadillac Plastic S.A.S., France). Then four cut PMMA plates were stacked and pasted with epoxy glue and dried for 24 hours. It is necessary to avoid any fluctuation of weight related to the degassing phenomenon. The total thickness needs to be higher than the size of the movable element. The last plate of the PMMA spacer was finally pasted with glue to abrade PMMA, zinc sulfide (ZnS) or germanium (Ge). These materials were chosen according to their significantly different Knoop hardness. PMMA had the lowest Knoop hardness with very little surface

roughness. The Knoop hardness was 240 kgf mm<sup>-2</sup> (≈24.47 N mm<sup>-2</sup>) for ZnS (Lame ZnS FLIR polished 1 face, 2 bevels, shape error 3/2, surface quality S-D 40–20, Optics Concept, France) and 780 kgf mm<sup>-2</sup> (≈79.54 N mm<sup>-2</sup>) for Ge (germanium polished blade 1 face, 2 bevels, shape error 3/2, surface quality S-D 40–20, Optic Concept France) and both materials had the possibility to be mirror polished (Optics Concept, France). These materials had the same geometry as the wear block one, presented as the standard method G75 (Go2 Committee 2007). The unpasted surface (mirror polish) of ZnS and Ge could better determine the impact of surface abrasivity. The samples were rinsed with ultrapure water (Millipore Milli-Q Integral 5, Quantum and Progard cartridge) and absolute ethanol (VWR Chemicals AnalaR Normapur). They were dried completely with a lint-free cloth. The samples were then placed in an oven (Memmert Model 400, 30–250 °C) at 60 °C for 1 h to ensure correct drying. Following oven drying, the test elements were left in a desiccator to reach ambient temperature prior to being weighed. The initial weight was obtained with a precision balance (Mettler Toledo XS 204, max 220 g–0.1 mg). After abrasion tests, the samples were rinsed with ultrapure water and absolute ethanol. When there was a deposit, it was cleaned with a lint-free cloth prior to a new rinse with ultrapure water and absolute ethanol. The same protocol was used to dry the element. Then the sample was weighed, and the mass of the abraded matter was deducted with a precision of 0.1 mg.

For Anton Paar's equipment, the abrasive potential was measured with a steel ball in rotation rubbing on three steel or Teflon plates. In the tribological cell, the temperature was controlled by Peltier effect at 4 and 25 °C. A bell could come over the cell to improve the temperature stability and limit the sample evaporation during analysis. The characteristics of the different element wears are shown in Table 2.

**Table 2** | Characteristics and wear conditions of consumables

Supplier	Movable element wear	Fixed support abrasion	Load [g]	Experimental wear environment
Anton Paar S.A.S., France	Steel ball	Steel or Teflon bars	1,019	0.1 to 3,000 rpm with 20 intermediate steps
Brant Industries, France	Home-made element wear in PMMA, ZnS or Ge	PMMA	500	Length = 150 mm; speed = 37 back and forth per min; 6,000 total back and forth
Elcometer, France	Home-made element wear in PMMA, ZnS or Ge	PMMA	750	Length = 100 mm; speed = 37 back and forth per min; 6,000 total back and forth
Falex Corporation, USA	Wear block 27% chrome iron	Neoprene moulded	2,268	Length = 203.2 mm; speed = 48 back and forth per min; 17,280 total back and forth
Taber Industries, USA	Brass pins	Stainless steel wear disc	750	72 rpm, 2,000 rounds

## Abrasive suspension

Four suspensions were used for the tests: two synthetic suspensions and two natural suspensions. The synthetic suspensions were composed of powdered activated carbon in suspension in groundwater. Two synthetic suspensions were made with two activated carbons at the same concentration. The first was a Norit activated carbon, with the Norit SA UF reference (SAUF suspension). Its granulometry was centred at  $5\ \mu\text{m}$  ( $D_{50}$ ) and its apparent density (packed powder) was  $225\ \text{kg m}^{-3}$ . This activated carbon powder is particularly adapted to limit hollow fibre abrasion and clogging. The second activated carbon was from Pica Charbons Actifs (Picasorb suspension). Its trade name is Picasorb 14. Its median diameter was between  $15$  and  $35\ \mu\text{m}$  and its apparent density was  $400\ \text{kg m}^{-3}$ . Raw water from the Seine River (France) was concentrated by settling up to  $200$  NTU and  $700$  NTU, respectively named Seine 200 and Seine 700, and used as the feed water. All the suspensions were stored at  $-20\ ^\circ\text{C}$  and defrosted at room temperature before the tests.

## Evolution and technical criteria

In order to evaluate all the devices, different criteria were developed. The aim was to obtain a value between 0 and 5 whatever the type of parameters was (qualitative or quantitative). These parameters were gathered according to their technical (T) or financial (F) characteristics. (T<sub>1</sub>) Ease of use: the analysis can be run physically and instrumentally quickly, but there can be a lot of preparation. This criterion represented the ease of use by taking into account the speed of preparation and its execution, the tool startup, the methodology suggested by the supplier, the representativeness of one test on the information obtained and the necessary workload for an operator. This parameter was evaluated at the end of this study to take into account all the results. (T<sub>2</sub>) Particles: samples were either synthetic or natural suspensions. Depending on the tool, there were some risks of denaturation of the samples (evaporation, aggregation/degradation of particles). The problems were linked to the experimental set-up, the concentration and the mean size of particles. The notation considered the presence of particles of size greater or smaller than  $100\ \mu\text{m}$ , the sensibility of the response for concentrations less than  $1\ \text{g L}^{-1}$ , the structuring of particles in stratum during the test and the modification of the composition during analysis (evaporation, coagulation, etc.). (T<sub>3</sub>) Repeatability: the tests were performed with the supplier's advices. The aim was to

minimize the variability of experimental data with the minimum of development work. The equipment could perform a repeatable state analysis (notation of 5) or with a methodology acquired during the device loan (notation of 4). It could be repeated if there was a simple methodology development (notation of 2.5) or an important one (notation of 1). Otherwise, the device was not adapted and was not repeatable (notation of 0). (T<sub>4</sub>) Accuracy: the accuracy was evaluated as a function of the information about the equipment. When the analysis was repeated, the result accuracy was associated with the coefficient of variation (CV). Otherwise, it was estimated with the suppliers' information on the various device components to obtain a CV. The notation was 5 if CV was smaller than 0.1% and 0 if CV was greater than 20%. (T<sub>5</sub>) Potential: the acquisition of a new equipment was integrated in the global evolution of the research department. Its flexibility and its transversality were an important aspect in the case of a first depreciation of materials. Thereby, the equipment modularity was evaluated. The notation of 5 was applied when the analysis duration, accuracy and repeatability are maximum, and if the equipment could be used for other applications thanks to an easy adaptation of the method enabling the development of a new type of analysis. (F<sub>1</sub>) Device price: the acquisition of new equipment required a first investment to obtain the basic configuration. The cost, the workforce for installation and the training of the operators were included for each device. A notation of 5 and 0 corresponds to a free device and the most expensive equipment respectively. (F<sub>2</sub>) Cost of consumables: depending on the requirement in terms of consumables, the interest in new equipment depended on its usage. This criterion represented the cost of one analysis. In this criterion, only the cost was considered in the operating expenditure. It was weighted by the maximum cost of consumables. (F<sub>3</sub>) Analysis duration: another operating expenditure integrated the duration between two possible analyses but human intervention was not integrated in this criterion. A notation of 5 would indicate that analysis would be instantaneous. (F<sub>4</sub>) Implementation: the environment required by the device was directly linked with the quality of the results. It could suggest the investment of peripheral equipment to guarantee the data validity. Each condition or additional equipment involved a new budget and the installation more difficult on site. A notation of 5 implied that the equipment was optimal without a particular environment. The notation decreased if the equipment needed improvements, air-conditioned room, climate chamber with controlled temperature and hygrometry, external system of temperature regulation (cryostat), fluid



utility (air) or precision balance. (F<sub>5</sub>) Transversality: once purchased, the new equipment was interesting in terms of predicted use beyond its current application. To provide the transversality notation, the acquisition of the equipment thinking about its flexibility in the context of this study but also the research themes of the department in which it will be installed. In relation to the future projects, samples could evolve toward samples without water as carrier fluid. Finally, the transversality had to be considered in terms of the chore of measurement and the equipment's ability to be readapted in a new context. The equipment had a notation of 5 if all items were met. Technical and financial criteria could evaluate qualitative or quantitative aspects. In financial criteria, the combination of the device price and cost of consumables could provide information about the real investment cost at medium and long term for the laboratory. In this case, an estimation of the number of analyses was a prerequisite and the integration of human time was more correct in the evaluation of the global cost of equipment.

### Statistical tools

The slurry ranking required the consideration of the abrasive potential as a value estimated by the measurement performed with the equipment. For each suspension in stable experimental conditions, there is an intrinsic value of abrasive potential. Calculating the average allows estimating the intrinsic value by the statistical treatment. By including the dispersion indicator of raw data, which is the variance, it is possible to quantify the risk of a misclassification and the reliability of a correct classification on the tested suspension. The descriptive statistics were calculated to evaluate the overall trend. Then, an analysis of variance (ANOVA) with one and two controlled factors was done to understand the cause of the variability and if the tested suspensions and the identified factors were significantly discriminated randomly (variability unexplained by a factor). The significance was observed by comparing the limit Fisher factor for  $\alpha = 0.05$  with the probability associated with the experimental observation  $p$ -value (Pearson 1900; Fisher 1925a). Finally, for some experimental conditions, the data for each suspension was compared in three steps to assess the quality of a proposed classification based on the measured values. For each suspension, the variance study was performed with Fisher test (Fisher 1925a). In experimentation conditions, a risk of first species  $\alpha$  was fixed to 0.05. The F-test was carried out bilaterally because there was no indication on a source of measurement

variability. If F-test conclusions indicated a difference of variance, the means of experimental measurement were compared with the Aspin Welch test (Welch 1947). Otherwise, the Student test was more appropriate (Student 1908; Fisher 1925b). Using Aspin Welch test and Student test, the normality distribution was assumed for the experimental data. Each average measurement of abrasive solution was compared with a risk of first species  $\alpha$  of 0.05. The quality and the reliability of the classification were quantified with the calculation of the risk of second species  $\beta$ . The type II error  $\beta$  should reflect the probability of ending in a low ranking due to misclassification of suspensions. Thereby, the statistical power  $(1-\beta)$  was deduced and quantified the reliability of the classification with a quality indicator to quantify the ranking between each suspension.

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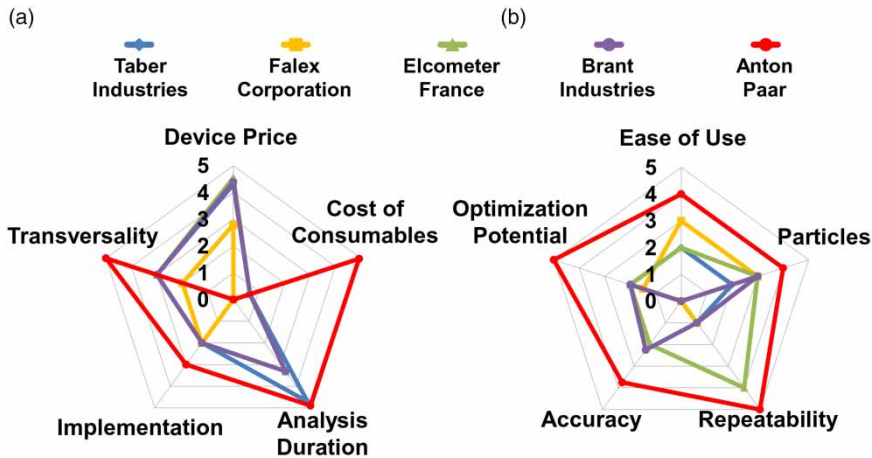
## RESULTS AND DISCUSSION

### Identification of the most pertinent instrument

All notations were collected and compared through a visual representation in relation to the technical or financial characteristics in Figure 2. In Figure 2 the surface can be assimilated to the performance and adequacy of the equipment with the determination of the abrasive potential of the suspension. Nine out of ten criteria showed that the Anton Paar's device was the most appropriate with a global notation greater than 4 compared to the four other devices. Whatever the financial or technical criterion was, Anton Paar's equipment appears to be the most efficient. But the device's price can be an obstacle to performing these analyses as routine. The Anton Paar device becomes financially interesting from 400 analyses onward. In a development context, this number of analyses would be quickly reached considering the test of validation and repeatability. This number appears very low when ranking or mapping different waters in terms of chemistry and geographical origin. In the context of this study, Anton Paar's device was the most appropriate one and was then selected for the next steps of the study. Thus, only the results obtained with this apparatus are described below.

### Stribeck curve of slurries (Stribeck 1901, 1902; Jacobson 2003)

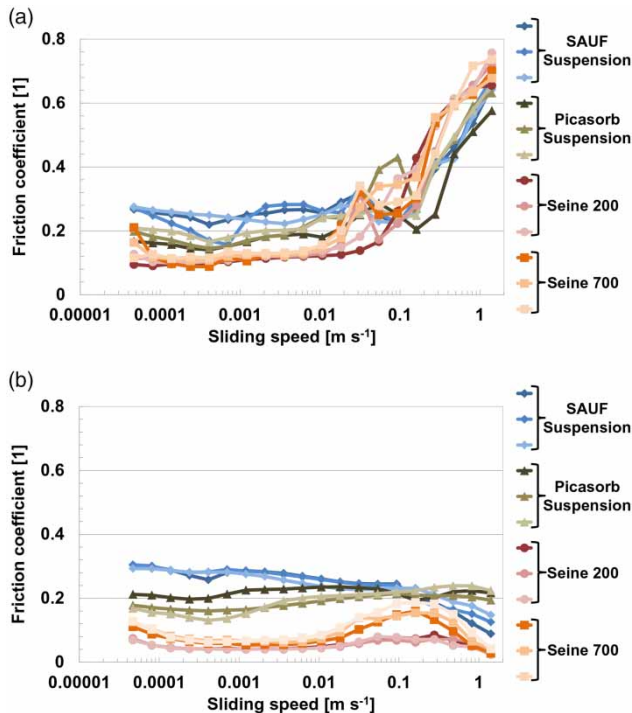
The MCR302 with Anton Paar's tribological cell was identified as the most relevant device. The measurement of friction coefficient with different suspensions using Stribeck



**Figure 2** | Analysis of criteria on five points: (a) financial criteria; (b) technical criteria.

curves was determined three times for each abrasive suspension, and for each type of plates (Figure 3). From Figure 3(a), the friction coefficient hardly changed until a sliding speed of  $0.01 \text{ m s}^{-1}$ , and it measured between 0.1 and 0.3. Visually, the activated carbon suspension appeared to be correctly separated, whereas Seine 200 and Seine 700 were really close. Beyond a sliding speed of  $0.01 \text{ m s}^{-1}$ , the friction coefficient lay between 0.2 and 0.8, but the identification of suspensions was difficult on steel plates. With Teflon plate (Figure 3(b)), each suspension was well identified, with a change in the

ranking order for  $0.16 \text{ m s}^{-1}$ . Although the friction coefficient was not above 0.4, the suspensions were better identified. The range in decrease of friction coefficient between steel and Teflon plates was explained by different analyses. With steel, a surface lapping in contact was necessary to decrease the frictions (Jisheng & Gawne 1997; Kato 2002) and to better assess the effect of suspensions used as lubricant. Another explanation was identified by the property of the Teflon to be a self-lubricant (Sreenilayam-Raveendran *et al.* 2013). The decrease of friction in sliding mechanical forces between steel and Teflon was a phenomenon already observed (Khamatkar *et al.* 2015). Compared with the steel plate, the range of lubrication regimes would change with Teflon plate toward a more important sliding speed. Thus, when the measurements on steel plates began to increase together with the sliding speed, the regime would change too. But the increase observed with the friction coefficient of the steel plate was linked to the transition between elasto-hydrodynamical and hydrodynamical lubrication regimes. However, for the same sliding speed, the measurements on Teflon plates always lay in the mixed regime (before the elasto-hydrodynamical regime) with a decreasing friction coefficient (Frene *et al.* 2010). In the range of the study, experimental measurements of friction coefficient on Teflon plates remained only within the lubrication regime and mixed regime boundaries whereas, for the highest sliding speed, the measurement of steel plates was in the hydrodynamic lubrication regime.



**Figure 3** | Stribeck curve: (a) on three steel plates, (b) on three Teflon plates.

### ANOVA on friction coefficient data

Obtaining a secure and robust classification requires the estimation of the experimental average of friction coefficient.



But as this average is an estimation, there must be an indicator of the dispersion of raw data. Therefore, the variance was determined by considering the type of suspension at first, and then by integrating a possible impact of the sliding speed. For each test, the suspension was analysed three times for 20 different sliding speeds. Table 3 shows the descriptive statistics for steel plates and Teflon plates respectively. Table 3 shows that all the average values for Teflon plates were lower than the average values for steel plates. The experimental variance was also lower. For Teflon plates, the CV was about twice lower than for the steel plate. This low level of variability was interesting for the suspension ranking. Teflon was known to have a low friction coefficient (Lauer 1999) compared to steel. The improvement of the lubrication phenomenon with a Teflon surface can explain these lowest variances. For an identical sliding velocity, the analysis on steel plates linked to surface roughness and steel hardness are noisier. In the case of the combination steel plates/steel ball, the highest asperities of each side were progressively eroded until a stabilized state. In the analysis, this last stabilized state was not obtained. The lubricants used with steel surface were oil rather than water lubricants (Studt 1989; Lauer 1999; Yoo & Kim 2013). However, in a tribological context, Teflon was often used with water as lubricant because of its low friction coefficient and wear (Deleanu *et al.* 2007, 2008; Deleanu & Georgescu 2015). With an ANOVA with one controlled

factor (suspension in this case), the variances were analysed to quantify the proportion of variability explained by the suspension compared to the variability linked to random (residue). F-test was used to verify that the variability of the identified factor was significantly higher than the variability of residue, corresponding to variability inside each suspension. Table 4 shows the results of ANOVA with the suspensions as controlled factor for steel and Teflon plates. For ANOVA with the suspensions as controlled factor, the sum of squared deviations (SSD) was calculated for each factor (suspension and residue). The SSD was weighted with the degrees of freedom factor to obtain the variance. The variance follows a chi-square distribution as the probability law. To compare two random variables, the ratio of two variances was calculated. The ratio study follows a Fisher-Snedecor law as the probability law. Thus, F-test allows identification of the relevant factor with a significant threshold fixed to  $\alpha=0.05$ . In Table 4 for steel plate,  $F_{\text{experimental}} < F_{\text{limit}}$  indicated that the suspension factor only was not significant to distinguish the different suspensions with analyses on steel plates ( $p\text{-value} = 0.277$ ). But on the Teflon plate (Table 4), ANOVA with one factor shows that the suspension factor could be significant with  $F_{\text{experimental}} > F_{\text{limit}}$  ( $p\text{-value} = 1.5 \cdot 10^{-87}$ ). Nevertheless, Stribeck curves in Figure 3 show an evolution with the sliding speed. Even if the resulting relevance with Teflon plate is highlighted, sliding speed could have a

**Table 3** | Descriptive statistics for assay on steel plates and Teflon plates

Material	Groups	Number of samples	Sum	Average	Variance
Steel plate	SAUF suspension	60	18.286	0.305	0.014
	Picasorb suspension	60	16.094	0.268	0.019
	Seine 200	60	14.743	0.246	0.042
	Seine 700	60	15.825	0.264	0.038
Teflon plate	SAUF suspension	60	14.5935	0.2432	0.0026
	Picasorb suspension	60	11.9103	0.19851	0.00079
	Seine 200	60	3.18253	0.05304	0.00021
	Seine 700	60	5.6835	0.0947	0.0016

**Table 4** | One-factor ANOVA on steel plates and Teflon plates

Material	Source of variations	SSD	Degrees of freedom	Variances	F-exp	p-value	F-limit
Steel plate	Suspension	0.110	3	0.036	1.29	0.277	3.17
	Residue	6.725	236	0.028			
	Total	6.834	239				
Teflon plate	Suspension	1.4083	3	0.4694	358	$1.5 \cdot 10^{-87}$	3.17
	Residue	0.3091	236	0.0013			
	Total	1.7174	239				

significant impact on the measurement of friction coefficient. An ANOVA with two controlled factors (suspension and sliding speed) was performed to quantify the impact of each of them on the measurement of friction coefficient. Table 5 shows the results of ANOVA with two controlled factors respectively for steel plates and Teflon plates. For two plates, variances were broken down in relation to both factors (suspension and sliding speed), the interaction between the factors and the part of residue. The interaction between both factors was important if the influence of the first factor was modified in presence or in absence of the other factor. The three sources of variations of the ANOVA (sliding speed, suspension and interaction) were compared with the residue with F-test (threshold  $\alpha = 0.05$ ). For all the comparisons,  $F_{\text{exp}} > F_{\text{limit}}$  indicated that all the factors could distinguish the measurement of the friction coefficient. The  $p$ -values confirmed that probabilities were extremely low. In conclusion, it was necessary to control the sliding speed to rank suspensions with measurements of the friction coefficient. In order to know the proportion of impact of factors on the friction coefficient measurement, experimental variabilities (SSD) were calculated and are displayed in Figure 4. The experimental data on three steel plates were mostly controlled by experimental conditions. With over 80% of the variability explained by the sliding speed for steel plate, only 1.6% of the variability was induced by the suspensions. Despite this low value, the suspensions were identified as a significant factor if there is a control of the sliding speed. For the steel plate, it is possible to obtain an f suspension ranking as a function of its abrasive potential, but the suspension factor was minor in the explanation of the global variability. In contrast, for Teflon plate, the suspension factor explained over 80% of the variability. The ANOVA with two controlled factors showed the sliding speed and the interaction with suspension represented

about 15% of variability over 90% on the steel plate. For Teflon plate, suspensions were a discriminating factor and the variability was mainly explained with a risk of first species lower than 1% ( $p\text{-value} < <0.01$ ).

### Evaluation of ranking by the abrasive potential

The four suspensions were evaluated for multiple sliding speeds. ANOVA of two factors had shown that the analysis completed on Teflon plates allowed the discrimination of suspensions with a weak impact of device environment (sliding speed). Nevertheless, no indications were calculated on the capability of the experimental set-up to do a low ranking (no difference between two suspensions) and to see a difference when two different suspensions were analysed. The corresponding probability of type II error  $\beta$  should reflect the probability to do a low ranking due to the inversion of two suspensions in the ranking. The power  $(1-\beta)$  could inform on the ranking reliability, namely on the possibility to see a difference when two different suspensions were analysed. The statistical analysis was conducted on Teflon plates data, for three different sliding speeds identified as very different: the beginning of analysis with a sliding speed of  $4.7 \cdot 10^{-5} \text{ m s}^{-1}$  (Table 6), the most complex sliding speed for  $0.161 \text{ m s}^{-1}$  (Table 7) and the end of analysis at  $1.41 \text{ m s}^{-1}$  (Table 8). The variance ( $s^2$ ) and the mean ( $m$ ) were compared with  $\alpha = 0.05$ . The values of the statistical powers were evaluated assuming that if two suspensions were different, the probability that two suspensions are identical also has to be assessed. Tables 6–8 represent the results of different statistical treatments. Generally, the variance of all the suspensions was identical for each comparison (represented by  $s^2$ ). For the most complex case with the sliding speed of  $0.161 \text{ m s}^{-1}$  (Table 7) none of the suspensions displayed a significant difference on the mean measurement

**Table 5** | Two-factor ANOVA on steel plates and Teflon plates

Material	Source of variations	SSD	Degrees of freedom	Variance	F-exp	p-value	F-limit
Steel plates	Sliding Speed	6.00	19	0.31	279.5	$3.1 \cdot 10^{-112}$	1.815
	Suspension	0.110	3	0.036	32.5	$1.8 \cdot 10^{-16}$	3.198
	Interaction	0.5400	57	0.0094	8.4	$1.1 \cdot 10^{-26}$	1.505
	Residue	0.1809	160	0.0011			
	Total	6.8338	239				
Teflon plate	Sliding Speed	0.06521	19	0.00343	13.5	$1.9 \cdot 10^{-24}$	1.82
	Suspension	1.40834	3	0.46945	1849	$6.5 \cdot 10^{-124}$	3.20
	Interaction	0.20327	57	0.00357	14.0	$1.2 \cdot 10^{-39}$	1.50
	Residue	0.04062	160	0.00025			
	Total	1.71744	239				

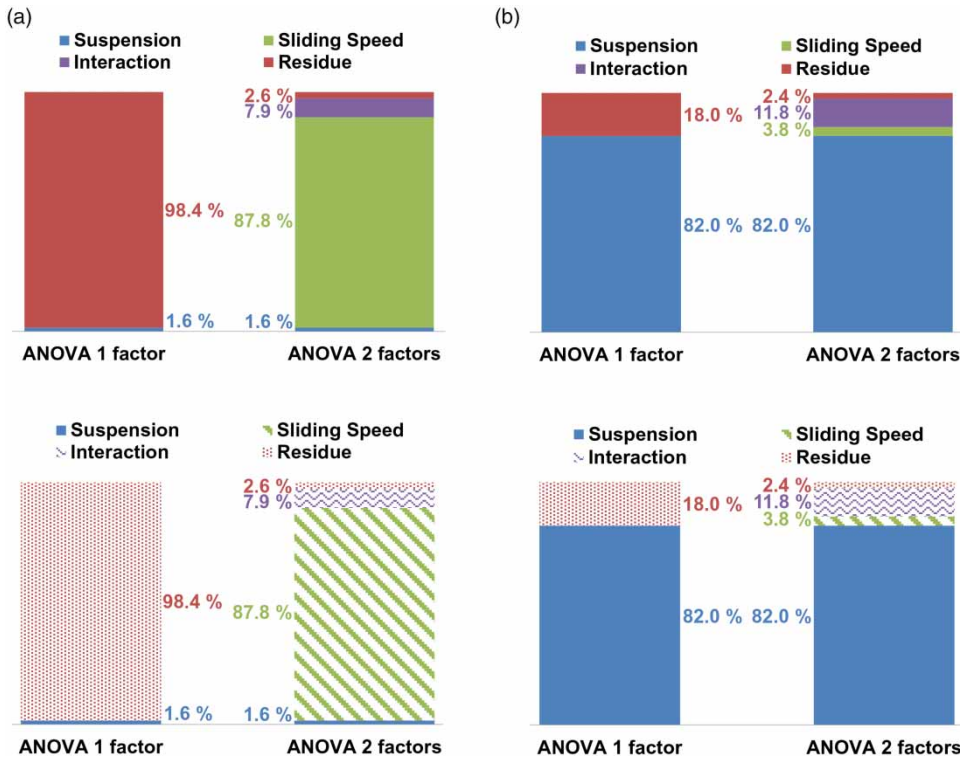


Figure 4 | Origin of experimental variability with the sum of square deviation: (a) on three steel plates, (b) on three Teflon plates.

Table 6 | Comparison of abrasive potential between two suspensions for the weakest sliding speed ( $4.7 \cdot 10^{-5} \text{ m s}^{-1}$ ) on Teflon plates

m1; s1\m2; s2	SAUF suspension	Picasorb suspension	Seine 200	Seine 700
SAUF suspension		$s^2 = ; m \neq ; 0.999$	$s^2 = ; m \neq ; 0.999$	$s^2 = ; m \neq ; 0.999$
Picasorb suspension	$s^2 = ; m \neq ; 0.989$		$s^2 \neq ; m \neq ; 0.989$	$s^2 = ; m \neq ; 0.921$
Seine 200	$s^2 = ; m \neq ; 0.999$	$s^2 = ; m \neq ; 0.999$		$s^2 = ; m \neq ; 0.999$
Seine 700	$s^2 = ; m \neq ; 0.999$	$s^2 = ; m \neq ; 0.996$	$s^2 = ; m \neq ; 0.991$	

Table 7 | Comparison of abrasive potential between two suspensions for the most complex of sliding speeds ( $0.161 \text{ m s}^{-1}$ ) on Teflon plates

m1; s1\m2; s2	SAUF suspension	Picasorb suspension	Seine 200	Seine 700
SAUF suspension		$s^2 = ; m = ; 0.109$	$s^2 = ; m \neq ; 0.996$	$s^2 = ; m = ; 0.746$
Picasorb suspension	$s^2 = ; m = ; 0.109$		$s^2 = ; m \neq ; 0.998$	$s^2 = ; m \neq ; 0.925$
Seine 200	$s^2 = ; m \neq ; 0.999$	$s^2 = ; m \neq ; 0.999$		$s^2 = ; m \neq ; 0.999$
Seine 700	$s^2 = ; m = ; 0.855$	$s^2 = ; m \neq ; 0.868$	$s^2 = ; m \neq ; 0.993$	

Table 8 | Comparison of abrasive potential between two suspensions for the highest of sliding speeds ( $1.41 \text{ m s}^{-1}$ ) on Teflon plates

m1; s1\m2; s2	SAUF suspension	Picasorb suspension	Seine 200	Seine 700
SAUF suspension		$s^2 = ; m \neq ; 0.943$	$s^2 = ; m \neq ; 0.946$	$s^2 = ; m \neq ; 0.922$
Picasorb suspension	$s^2 = ; m \neq ; 0.993$		$s^2 = ; m \neq ; 0.999$	$s^2 = ; m \neq ; 0.999$
Seine 200	$s^2 = ; m \neq ; 0.999$	$s^2 = ; m \neq ; 0.999$		$s^2 = ; m = ; 0.602$
Seine 700	$s^2 = ; m \neq ; 0.999$	$s^2 = ; m \neq ; 0.999$	$s^2 = ; m = ; 0.263$	

of friction coefficient. Visually, as statistically, the SAUF and Picasorb suspensions were the least discriminated with their mean identified as equal (represented by m), and a really low statistical power of 0.109. Despite a sliding speed disadvantageous for the identification of all the suspensions as different, two-thirds of the comparison allowed the detection of the difference with a higher statistical power. In a context of determination of a robust ranking, two rankings could be identified in relation to the value of the sliding speed used to calculate the friction coefficient. Up to  $0.161 \text{ m s}^{-1}$ , these four suspensions had the following ranking by ascending order of abrasive potential: Seine 200 < Seine 700 < Picasorb suspension < SAUF suspension. Table 6 shows a really good discrimination of each suspension with a very good statistical power higher than 0.9. Above a sliding speed of  $0.161 \text{ m s}^{-1}$ , the ranking is modified by the change of lubrication regimes between the steel ball and the Teflon plates. Beyond  $0.161 \text{ m s}^{-1}$ , the suspension ranking by ascending order of abrasive potential was: Seine 200 < Seine 700 < SAUF suspension < Picasorb suspension. The abrasiveness index thus developed must be compared to another measurement that shows that the contact of a real membrane with feed water may result in important damage. Based on such calibration performed on different waters used in drinking water production plants and the abrasion measurements done on the membranes that filter these waters, it will then be possible to recommend pre-treatments required to reduce feed water abrasivity and acceptable for contact with membranes.

## CONCLUSION

This study focused on the evaluation of the real contribution of the abrasion phenomena to the abrasive potential of waters (naturally turbid surface waters and groundwater containing suspensions of powdered activated carbons). The methodology consisted of benchmarking five different types of apparatus already marketed for other applications like rock excavation or the manufacturing of toothpaste. The devices were evaluated on their capacity to be adapted to particles commonly found in most drinking water treatment plants, but at low concentrations (less than  $1 \text{ g L}^{-1}$ ). In order to rank the abrasive potential of different waters, it is necessary to identify an experimental set-up with a good compromise between technical and financial parameters. After comparing 10 criteria, the MCR302 with the tribological cell of Anton Paar was identified as the most relevant device. The data produced with this

experimental set-up was analysed statistically in different steps. An ANOVA allowed identification of the parameters influencing the measurement of friction coefficient (assimilated to the abrasive potential). This analysis enabled the identification of the best plates (in Teflon) with which the waters (suspensions) had the major impact on the measurement. Finally, the relevance of ranking was evaluated with a comparison of each suspension measurement with the calculation of the statistical power. In the conditions of the study, the relevance of ranking obtained with the experimental set-up of Anton Paar was higher than 90% in a correct range of sliding speed of the device. The newly developed methodology could quantify the abrasive potential of natural waters used in membrane filtration. The abrasiveness index thus developed must be calibrated with tests on a real polymeric membrane with such feed water, which may display significant damage. Based on such calibration, it will then be possible to recommend pre-treatments required to reduce water abrasivity to an acceptable level at the inlet of membrane systems. This methodology will be extended to sea water for the pre-treatment of reverse osmosis.

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