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Filtration charaterization methods in MBR systems: A practical comparison

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ABSTRACT

Three different methods for filtration characterization in Membrane Bioreactor (MBR) systems were compared. These were the Delft Filtration Characterization Method (DFCm), the Berlin Filtration Method (BFM) and an *ex situ* side-stream filtration test cell for the determination of the critical flux. The *ex situ* filtration test cell and the DFCm filter activated sludge from a tank, while the BFM works *in situ* with a test cell directly submerged into the biological tank at similar operational conditions to a typical MBR plant. The mixed liquor of four different MBR units was characterised several times with the three filtration methods. The three tested methods seemed to agree in the classification of the tested mixed liquors in terms of filterability except for one of the tested activated sludges. Additionally, three critical flux protocols were studied using the BFM filtration test cell. The first consisted in the classical flux-step method, the second included relaxation between filtration steps and in the third protocol, 2 min filtration at a fixed flux were performed before every filtration step. The last protocol was selected as the most representative of full scale MBR operation and the most interesting one for giving valuable information about the irreversibility of the fouling.

Keywords: Membrane bioreactors (MBR); Filterability; Fouling

1. Introduction

The lack of design and method standardization in Membrane Bioreactor (MBR) systems has been identified as one of the hindrances of a general MBR technology acceptance by the wastewater treatment market [1]. The absence of a standard method for the determination of the filterability of the activated sludge in MBRs has led to the development of a large quantity of different and more or less elaborated filtration characterization methods in the last years by the numerous research institutions and end-users working with this technology. These methods can be divided into those which measure fouling rates and their development over a certain time, and the critical flux measurement methods. The DFCm (Delft Filtration Characterization Method [2]) and the MBR-VFM (VITO Fouling Measurement) [3] belong to the most known methods belonging to the former. In the determination of the critical flux, flux-step methods are usually used in laboratory investigations [4]. While the main objective of the DFCm lies in the comparison of the filterability of different activated sludges measured under well defined hydrodynamic conditions, the objective of the MBR-VFM is the on-line monitoring of the plant. In the case of the MBR-VFM, the determination of the filtration resistance is carried out at constant pressure, whereas the DFCm uses constant flux, which is the common operating modus in the real MBR plants. It has been demonstrated that fouling mechanisms are different at constant pressure than at constant flux, showing in experiments at constant flux more complex mechanisms [5]. By applying relaxation, the MBR-VFM provides

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information about the irreversibility of the fouling. The idea of the DFCm is to "accelerate" fouling by applying a supercritical flux, providing a quick evaluation of the quality of the sludge in terms of filtration resistance.

The concept of critical flux, introduced in 1995 by Field et al. [6] is very widespread in the field of membrane technology, but also very much debated, especially for MBR applications. The lack of a standard protocol is here also one of the main hindrances of its application.

In this study, a new method called BFM (Berlin Filtration Method) is introduced which final purpose is the fouling monitoring of the plant. This method uses critical flux determinations for the evaluation of the filterability of the activated sludge via an *in situ* test cell. The novel method was compared and evaluated with the DFCm and a filtration test cell for the determination of the critical flux. In order to select the most appropriate protocol for critical flux determinations with the *in situ* test cell, three flux-step protocols were compared and evaluated.

2. Materials and methods

2.1. In situ test cell

The BFM uses a UF flat-sheet made of PES (Micro dyn—Nadir) of dimensions 9 cm × 13 cm (filtration surface 0.025 m²). The whole equipment was kept as simple as possible in order to facilitate the assembling in the plant and the transport to the units. The measurements are carried out in the anoxic chamber of the units except for MBR4, which measurements take place in the aerobic chamber because of space issues. The frame of the membrane module is perforated at the bottom, which provides the module with an integrated aeration via an air blower. The permeate side is connected to a pressure sensor so that the evolution of the pressure on the permeate side is monitored continuously during the measurement. The SAD applied to the test cell was 3.5 m³/(m²h). Due to higher width/height ratio of the flat sheet used in the test cell in comparison to a typical MBR flat sheet module, much more aeration must be applied per membrane surface in the test cell to achieve a similar superficial gas velocity. The spacing between plates was fixed at 7 mm, a typical value of spacing between membrane in flat sheet MBR modules. A scheme of the apparatus can be seen in Fig. 1.

Membrane permeability was regularly checked by filtering deionised water. Chemical cleaning was performed by soaking the membrane in a 1% active chlorine solution when the permeability loss was greater than 10%.

2.2. The delft filtration characterisation method (DFCm)

The unit is represented schematically in Fig. 2 and described in detail in [7] and [2]. The heart of the installation is formed by a single sidestream ultrafiltration membrane tube (X-flow, diameter 8 mm, nominal pore size 0.03 μm).

The standard protocol filters about 30 activated sludge which is kept aerated during the experiment. The sample is recirculated with a cross flow velocity of 1 m/s. Permeate can be extracted with any desired flux. Membrane cleaning is performed after every experiment using forward flush, back flush or chemical cleaning (or a combination of them). Clean water resistance is determined after and before the experiment to verify that the membrane has been cleaned properly. If that is not the case, supplementary chemical cleaning can be applied.

The filterability of the mixed liquor is expressed as a ΔR_{20} value which represents the increase in additional membrane filtration resistance after extraction of 20 Ll/m² of permeate under the protocol described below. Normally a ΔR_{20} value below 0.2 10^{12} m^{-1} is typical for good filterable mixed liquors, whereas ΔR_{20} values above 1 10^{12} m^{-1} are characteristics of activated sludge with bad filterability. The data presented in this paper were obtained with a constant permeate flux of 80 L/(m²h) and corrected at a temperature of 15°C.

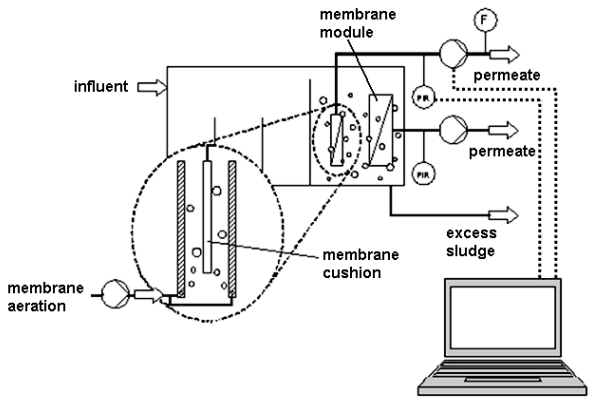

Fig. 1. Scheme of the *in situ* BFM test cell.

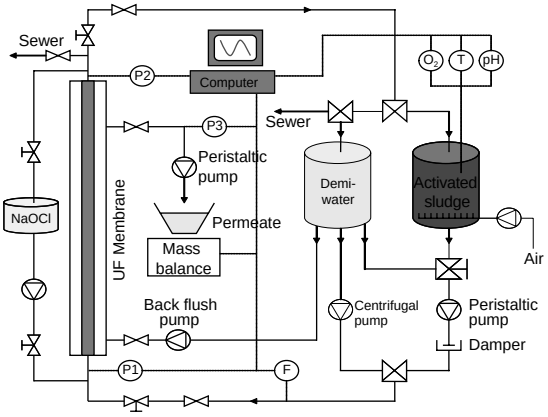

Fig. 2. Filtration characterisation installation.

Table 1
Operation conditions of the investigated MBR units during the monitoring campaign (January–July 2008).

	SRT (d)	TS* (approx.) (g/L)	COD supernatant* (mg/L)	COD influent (mg/L)	Wastewater influent	Temperature (°C)
MBR1	12	8–9	30–110	500–1500	Municipal	17–20
MBR2	12	6–12	30–110	500–1500	Municipal	17–20
MBR3	25	9–22	150–300	750–2500	Domestic	10–25
MBR4	35**	8–10	100–400	750–2500	Domestic	10–25
MBR5***	39	8	175	400	Synthetic	18

*of mixed liquor taken from the membrane tank (in Berlin, TS ~ MLSS + 1 g/L (salt)).
**at the end of the monitoring campaign. Before that, no excess sludge withdrawal (SRT>200 d).
***Mean values for April, when the critical flux protocol comparison was performed.

2.3. Ex situ test cell

The filtration test cell uses a virgin MF PVDF flat sheet membrane of dimensions 5.5 cm × 16 cm for each experiment. The activated sludge is stored in a 8-L tank which is continuously aerated. The cross-flow velocity used is 20 cm/s and the aeration is set at 7.2 $m^3/(m^2h)$. Further details about the test cell are provided in [8] and [9].

2.4. MBR units

The BFM was included in the monitoring program of four MBR units operated in Berlin. One of them is an MBR pilot plant constructed within the EU Project AMEDEUS, which works with two units in parallel (MBR1 and MBR2). The other one (MBR3) is a demonstration plant from the project ENREM, designed to perform enhanced nutrients removal. A second unit (MBR4) was installed in January 2008 side by side to the MBR3 unit, and was designed to perform COD removal and nitrification. In the pilot plant, the two units MBR1 and MBR2 have an approx. volume of 1.5 m^3 and are equipped with a submerged flat sheet microfiltration (MF) membrane module each (22 m^2). Both are fed with municipal wastewater; in one of them (MBR2) a flocculant for permeability enhancement is added periodically. The demonstration plant (MBR3) treats domestic effluent for approximately 250 p.e. in a reactor volume of about 10 m^3 and is operated with one MF module with a membrane surface of 31 m^2. MBR4 has a reactor volume of about 5 m^3 and is equipped with 20 m^2 membrane surface. MBR5 is a 50-L lab scale unit fed with synthetic wastewater simulating municipal water and was used for the critical flux comparison. The characteristics of the plants are described in Table 1.

2.5. Critical flux protocols

The protocols followed for the determination of the filterability consisted of modified flux-step methods for the determination of the critical flux. Three protocols were tested:

2.5.1. Classical protocol

Described in [10] and represented in Fig. 3 (left). The flux is increased stepwise and decreasing until the initial value is reached.

2.5.2. Filtration/Relaxation protocol

Basically the classical protocol with relaxation between filtration steps (Fig. 3 (II)). The objective is to make the filtration regime more similar to the plant operation conditions, in which relaxation commonly takes place and the membrane virtually recovers from all reversible fouling that accumulated during filtration. This was the protocol used with the BFM during the first month of investigation and the protocol used in the *ex situ* test cell during the whole study.

2.5.3. Pre-step protocol

A modification of the Filtration/Relaxation protocol is introduced here by filtering at a constant low value

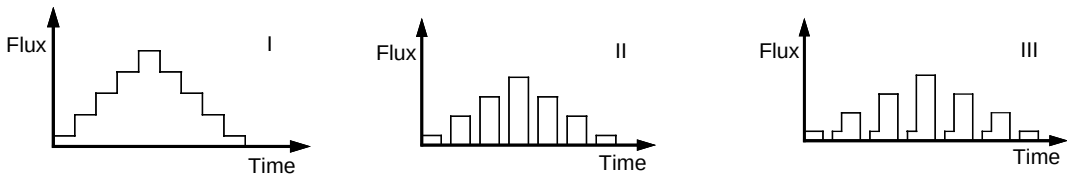

Fig. 3. (I) Classical protocol, (II) *Filtration/Relaxation* protocol, (III) *Pre-step* protocol.

(5 L/(m²h)) for 2 min before every filtration step (Fig. 3 (III)). The objective is to obtain by the evolution of the transmembrane pressure (TMP) in this pre-filtration step a parameter which could give information about the irreversible fouling, i.e. the fouling remaining after the relaxation step. This protocol is similar to the one developed by van der Marel *et al*. [11] but it reaches complete relaxation. The pre-step was found to be too low at 5 L/(m²h) for detecting any irreversible fouling with precision and it was increased to 10 L/(m²h) from May onwards. In this case, no pre-step was done for the first filtration steps until the flux achieved 10 L/m²h. This protocol was introduced in the BFM on 01.02.08 and maintained during the whole study.

The duration of the whole protocol was less than two hours for the classical protocol and between two and three for the other two protocols. The flux step for both protocols was 3 L/m²h, and the initial step selected was variable, from 5 to 8 in some periods. In some cases, the critical flux was already achieved at the beginning of the experiment, and the first step represent in these cases the "detection limit" of the method (between 5 and 8 L/(m²h)). The end of the experiment was also variable, from 31 to 40 L/(m²h) depending on the monitoring period. Filtration time was 5 min in all cases and the relaxation time was 2 min.

Critical flux in the BFM test cell was determined as the flux step at which dP/dt was greater than 0.2 mbar/min. The TMP data were corrected with viscosity data (ηp) as follows:

$$TMPc = TMP \cdot \frac{\eta_{ref}}{\eta_{act}} \quad (1)$$

η_{ref} = dynamic viscosity at 20°C $(Pa \cdot s)$
η_{act} = dynamic viscosity during the measurement $(Pa \cdot s)$
TMP_c = corrected transmembrane pressure $(mbar)$

$$\eta_p = \frac{479 \cdot 10^{-3}}{(T + 42.5)^{1.5}} \quad (2)$$

T = temperature (°C)

In the *ex situ* test cell, a threshold value of 1 mbar/min was selected as the most appropriate for the determination of the critical flux. The value recommended by Le-Clech et al. in [10] of 0.1 mbar/min was reached already in the first early steps and the for this low pressure variations the pressure sensor was not able not give a reliable signal. Therefore this value was found too low for our operating conditions and equipment. As the storage tank was maintained at a constant temperature of 20°C, no temperature correction was needed.

3. Results and discussion

3.1. Critical flux protocol comparison

For the selection of the critical flux protocol, three critical flux experiments were performed in the same day *in situ* in MBR5 with the BFM test cell. This comparison was repeated with activated sludge from MBR1 in the laboratory (measurement *ex situ*) and with activated sludge from MBR4 *in situ*. The activated sludge was aerated during and between experiments *ex situ* in order to avoid significant changes in its nature.

The results from this comparison are shown for the activated sludge from MBR5 in Figs. 4 and 5. As expected, the results from the *filtration/relaxation* protocol and *pre step* protocol are quite similar, and differs from the *classical* protocol (protocol I) in terms of average TMP in the filtration steps. The results from the experiments mentioned below with activated sludge from MBR1 and MBR4 are not shown here but they showed similar patterns. It is remarkable that in terms of fouling rate and critical flux, all protocols exhibited very similar results. The Jc values obtained with the three protocols gave the same values with a mistake of maximally one flux step for the selected criteria (dP/dt < 0.2 mbar/min). Due to the fouling history, we could have expected higher fouling rate for protocol I, at least for fluxes above the critical flux (due to higher TMP after at least 30 min of continuous filtration without relaxation). On the other hand, a classification effect might have taken place during the protocols with relaxation, because the increment of flux is quite high in the last steps, going up to 30 L/(m²h). This effect was reported for solutions of colloidal silica [12], where it was shown that a great flux increase at once led to a higher dP/dt value due to a faster developed and therefore more chaotic fouling layer than the obtained by a slowly increasing of the flux. The contrary effect was found from other authors [13]. These two effects (classification effects on the one hand and relaxation neglecting the membrane history) might be both negligible, or might have cancelled each other out and the differences between protocols from the results of this study are therefore not significant.

The *filtration/relaxation* protocol (protocol II) and *pre-step* protocol (protocol III) were selected as the most interesting ones for giving valuable information about the irreversibility of the fouling by looking at the TMP average evolution. When using the *classical* protocol (protocol I), an accumulation of the cake on the membrane occurs along the cycle after the critical flux is reached and a hysteresis phenomenon always takes place (Fig. 4). The membrane history influences the measurement because no relaxation take place, so that the results are very dependent of the flux-step height and

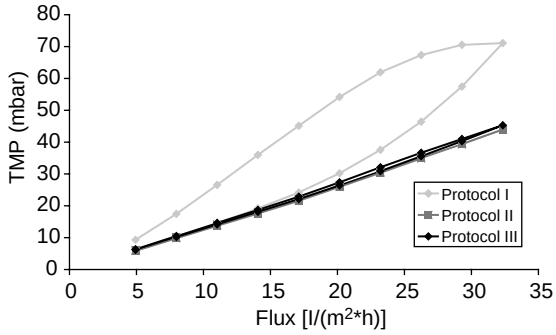

Fig. 4. Evolution of the $TMP_{c,ave}$ versus Flux: *ex situ* measurement with the BFM with activated sludge from MBR5 (synthetic wastewater).

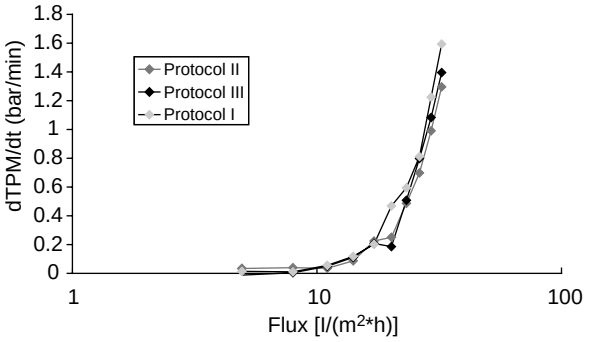

Fig. 5. Fouling *rates* in the protocol comparison: *ex situ* measurement with the BFM with activated sludge from MBR5 (synthetic wastewater).

duration. With the other two protocols, the relaxation between steps makes the membrane start the following filtration step without being influenced from the membrane history so that the pressure by increasing the flux must be the same as the pressure by going down in the flux for the same flux. In some cases, a hysteresis can be observed also with protocols II (*filtration/relaxation*) and III (*pre-step* protocol) due to remaining fouling in the membrane which could not be eliminated with the relaxation, as it was shown in [14] for activated sludge showing poor filterability. This was called irreversible fouling. Protocol III, due to the short pre-step at low flux, offers the following specific advantages: it enables a quicker stabilisation of the pressure value when applying the higher flux step, therefore provides cleaner data, but more important it provides a direct measure of irreversible fouling during the pre-step, straight after the relaxation phase.

3.2. DFCm and BFM comparison

The DFCm was applied during one week in the determination of the filtration resistance of the four investigated MBR units. The BFM was applied in every plant between 2 and 3 days of the same week in order to enable a comparison of the three methods. The total solid content (TS) was determined for some of the days of the measurement campaign. Table 2 sums up the results obtained during the comparison week.

The results from Fig. 6 show that there are some differences due to differences in configuration, operating conditions and membrane material. However, the critical flux obtained with the BFM and the filtration resistance measured with the DFCm seemed to agree with a correlation coefficient of 0.65 for three of the biological sludges (MLSS range 7–16 g/L), although more measurements would be needed in order to confirm these data. For MBR4, the DFCm and BFM gave totally different results, showing the activated sludge by the DFCm a good filterability while having low critical flux values obtained with the BFM. The reason could be the unstable operation during the start-up phase of the unit and the adaptation phase after seeding, or a low solid concentration of the plant (4 g/L MLSS): for these conditions, these two methods do not seem to be comparable.

Table 2
MBR filtration characterization during the comparison campaign week.

	MBR1			MBR2			MBR3			MBR4		
	DFCm	BFM	TS	DFCm	BFM	TS	DFCm	BFM	TS	DFCm	BFM	TS
Date	ΔR_{20}	Jc [Lm²h]	g/L	ΔR_{20}	Jc [L/m²h]	g/L	ΔR_{20}	Jc [L/m²h]	g/L	ΔR_{20}	Jc [L/m²h]	g/L
28.01.08	0.86	-	8.2	0.86	-	9.2	1.56	14	16	0.71	5	5.06
29.01.08	0.9	-		0.98	-		1.5	14	16.4	0.86	11	5.74
30.01.08	0.87	22		1.12	19		2.96	-	15.6	1.53	-	5.21
31.01.08	0.79	22	9.7	1	25	10.4	2.86	-	14.9	1.32	-	5.4
01.02.08	1	19		1.07	25		1.26	-	17.4	1.7	-	6.4

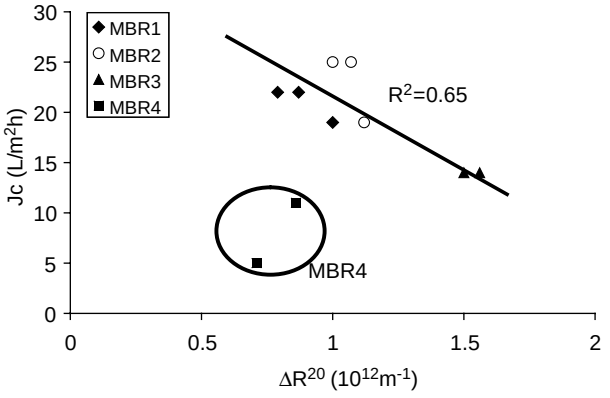

Fig. 6. Comparison of the results of Jc and ΔR_{20} values obtained in the comparison campaign.

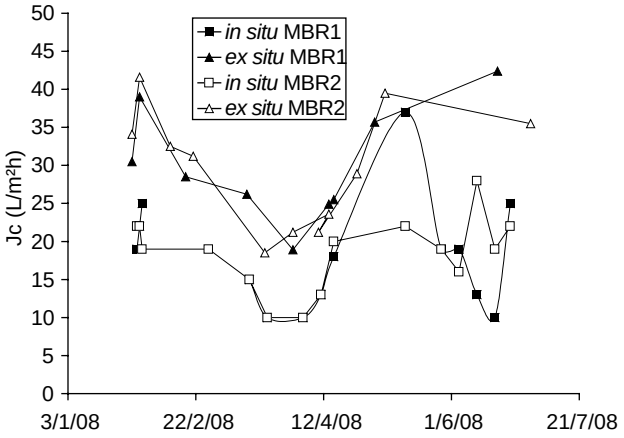

Fig. 7. Comparison of the results of the BFM and the *ex situ* filtration test cell during the regular monitoring of the plants.

3.3. BFM and test cell ex situ comparison

Fig. 7 reflects the comparison performed between the two filtration test cells used in this study to measure critical flux (*in situ* BFM and *ex situ*) during more than six months in MBR1 and MBR2. A direct comparison is difficult because the measurements were done on different days but it can be noticed that the same trend is followed by both of them, although the results are not strictly equivalent probably due to the differences in the operating conditions. The higher values in the *ex situ* test cell may be attributed to the lower pore size of the membrane in the case of the BFM, although some authors have found lower fouling rates in MF membranes than in UF membranes caused by pore clogging of the MF membranes by large particles, and protection of the UF membrane by the large particles acting as a secondary membrane [15]. Another reason can be that the parameter used to determine the critical flux results in more conservative results in the case of the *in situ* test cell. In the *ex situ* test cell, the sludge circulates at high cross-flow velocities through the membrane, which causes high shear forces and high fouling rates so that the parameter selected for the determination of the critical flux (dP/dt < 1 mbar/min) is much higher that in the BFM (dP/dt < 0.2 mbar/min), in which the sludge circulates with the action of the air scouring.

The advantage presented by the *ex situ* test cell is the use of a new membrane in every experiment so that there is no influence in the measurement of the membrane history. However, this method is *ex situ*, which involves transport and storage of the sludge in a tank, where the measurements will take place. In contrast to that, the BFM uses an *in situ* aerated test cell which can be submerged directly in the bioreactor. This way, it eliminates problems like sludge transport and storage or different fluid dynamic conditions typically encountered in common filterability measurements [4].

4. Conclusion

Three different methods for the filtration characterization in MBR were compared in four MBR units. The three tested methods seemed to agree in the classification of the activated sludge in terms of filterability except for one of the tested MBR units. That was attributed to the low MLSS of the unit and the unstable operation of the plant. When comparing the results from the monitoring of the MBR units during six months with the *in situ* (BFM) and *ex situ* test cell, the same trend could be found for both test cells, showing the *ex situ* test cell leads to higher values than the BFM. This and other differences were attributed to the different operating conditions, membrane material and critical flux determination method. From the results of this study, the three filtration characterization methods were found appropriate for the measurement of the filterability in MBR. Additionally to this comparison, three critical flux protocols were studied using the BFM filtration test cell with activated sludge from three MBR plants. The results were quite similar in terms critical flux values and fouling rates for the selected determination criteria (dP/dt < 0.2 mbar/min), although the classical protocol was found to be influenced by the membrane history and this way influenced by characteristics of the protocol like first step, step height and step duration. Therefore and for providing information about the irreversibility of the flux, protocol III with relaxation and pre-step was selected as more interesting for critical flux determination.

Acknowledgements

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