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### Sustainable Flux Fouling in a Membrane Bioreactor: Impact of Flux and MLSS

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## Sustainable Flux Fouling in a Membrane Bioreactor: Impact of Flux and MLSS

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**Abstract:** The long term sustainable flux behavior of a submerged membrane bioreactor operated under a steady state conditions at a range of mixed liquor suspended solids (MLSS) concentrations has been examined. Comparison of fouling rates at a number of imposed fluxes has been made between long term filtration trials and short term tests using the flux step method. Results indicate an exponential relationship between fouling rate and flux for both long and short term trials, although the value was an order of magnitude lower during long term tests. Moreover, operation during long term trials is characterised by a period of pseudo stable operation followed by a catastrophic rise in TMP at a given critical filtration time ( $t_{\text{filt}}$ ) during trials at  $6 \text{ g} \cdot \text{L}^{-1}$ . This time of stable operation,  $t_{\text{filt}}$ , is characterised by a linear relationship between fouling rate and flux. Results have been compared with the literature. Data for membrane fouling prior to the end of  $t_{\text{filt}}$  yielded a poor fit with a recently proposed model. Trends recorded at  $t > t_{\text{filt}}$  revealed the fouling rate to follow no definable trend with flux, contrary to the notion that fouling beyond the critical filtration time relates to solids deposition.

**Keywords:** Submerged membrane bioreactor, sustainable flux, fouling

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## INTRODUCTION

It has become evident through a number of studies that even at very low fluxes some fouling occurs in MBRs (1–4), such that the original definition of critical flux where there is a flux below which fouling (or permeability decline) does not occur (5) does not apply to MBRs. A more suitable indicator and definition would be “transitional flux,” thus representing the change from relatively low and stable fouling to higher fouling rate at the onset of significant fouling. This value is often used as a guide to establish an operating flux for a given system. There is a certain degree of interpretation of values which represent “low” and “high” fouling.

How well these values relate to sustainable long term operation is not fully understood since fouling rates (i.e.  $dP/dt$  values under constant flux conditions,  $P$  being the transmembrane pressure, TMP) recorded for long term operation have been reported as being between 10 and 100 times lower than those measured for short term trials (4). Evidence of fouling at low fluxes is provided from long-term studies, where a characteristic pattern of behavior over time appears to evolve in some reported cases. An initial rapid but small increase in TMP takes place followed by a very small TMP rise over an extended time period. In some studies (2–4, 6, 7) a noticeable increase in  $dP/dt$  arises after some critical time period. The ability to predict the onset of this high fouling period would be advantageous and the application of a recently proposed fouling model (2) is investigated.

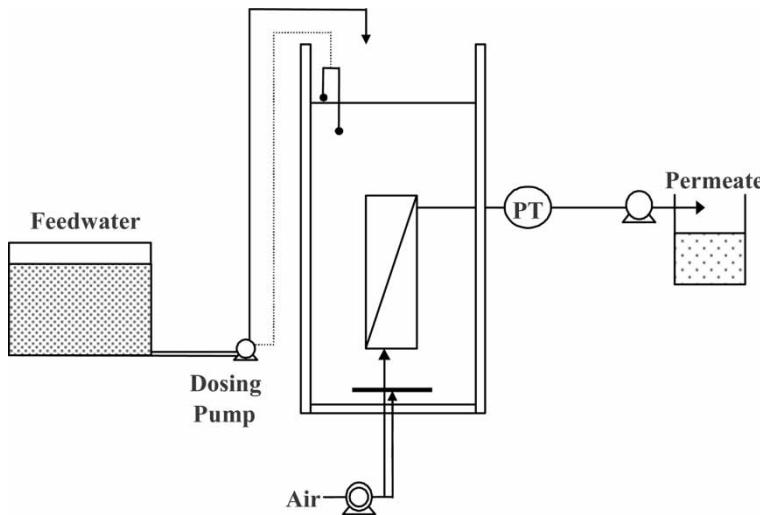
The present study aims to investigate further the changes in low-flux (i.e. sustainable flux) fouling during long-term operation under constant flux conditions in an MBR operated under steady-state conditions fed with a produced water analog. Produced water may be defined as the wastewater that is brought to the surface during oil and gas production. This water tends to be of varying salinity with key trace pollutants comprising dispersed and dissolved hydrocarbons such as BTEX compounds (Benzene, Toluene, Ethylbenzene, Xylene) and polycyclic aromatic hydrocarbons.

## METHODS

### Pilot Scale Rig and Feed-Water

A submerged membrane bioreactor, 40 L in volume (Fig. 1), was operated under steady-state conditions. The vertically mounted tubular membranes used in the current study were supplied by X-flow and had a total area of  $0.15 \text{ m}^2$  and nominal pore size of  $0.03 \mu\text{m}$  (Table 1).

The produced water analog employed had a target COD of  $1575 \pm 100 \text{ mg} \cdot \text{L}^{-1}$ , and contained methanol, ethylene glycol, BTEX compounds (Benzene, toluene, ethylbenzene, and xylene), polycyclic aromatic hydrocarbons (acenaphthene, biphenyl, phenanthrene), naphthalene,



**Figure 1.** MBR rig schematic.

phenol, carboxylic acids (acetic acid, propionic acid, valeric acid), and sodium chloride (1%). Nutrients of urea and orthophosphate required to support micro-organism growth were also dosed into the feedwater. The bio-reactor was operated at a HRT of 24 hours. All flux step tests and long-term fouling trials were carried out following membrane cleaning with 0.5% Ultrasil 75, soaking at 50°C for 24 hours.

### Experimental Analysis and Data Handling

Biomass from all systems was collected and analyzed for solids content, size, viscosity, dewaterability, and organic content in terms of EPS (extracted

**Table 1.** Membrane characteristics

Type	X-flow 11PEFr5385
Material	PVDF
Module length	1 m
Lumen internal diameter	8 mm
Number of lumens	7
Total membrane working area	0.15 m <sup>2</sup>
Pore size	0.03 µm
Module air flow	7 L · min <sup>-1</sup>
Module air loading	0.2 m · s <sup>-1</sup>

extracellular polymers) and SMP (soluble microbial products) according to methods outlined previously by Le-Clech et al. (4), based on the method of Zhang et al. (8). The MLSS and MLVSS (mixed liquor suspended solids and mixed liquor volatile suspended solids respectively) content were determined according to Standard Methods (9). TMP trends from long-term operation were processed by applying a 60 minute moving average so as to smooth fluctuations.

## RESULTS AND DISCUSSION

Although the two-stage fouling phenomena discussed earlier has been exhibited in other studies (2–4, 6, 7) a lack of comprehensive data prevented any correlation between applied flux and either fouling rate or time of stable operation ( $t_{\text{filt}}$ ) being produced by these authors. The only other study of sustainable flux fouling rates and flux has been by Wen et al. (10).

Operation at fluxes between 2 and  $10 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$  for the system having a biomass concentration of  $6 \text{ g} \cdot \text{L}^{-1}$  MLSS demonstrated fouling dynamic behavior to depend on flux (Fig. 2). At a flux of  $10 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$  no stable low fouling period was exhibited, but rather a linear fouling from the beginning of experiment (corresponding to  $dP/dt = 0.036 \text{ mbar} \cdot \text{min}^{-1}$ ) followed by rapid fouling up to 130 mbar after 2 days of operation. When operated at the lowest flux of  $2 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$  the TMP was stable for up to 10 days without the onset of accelerated fouling noted earlier. However, the TMP increased from 2 to 12 mbar within the 10 first days of filtration ( $dP/dt = 7.10^{-4} \text{ mbar} \cdot \text{min}^{-1}$ ) confirming that even at very low fluxes some fouling still takes place.

Fouling rates recorded at other fluxes reveal the same distinct pattern with a period of stable operation ( $t_{\text{filt}}$ ) before a sudden increase in fouling rate. The first stage of gradual but steady increase in TMP is thought to be due to dissolved/colloidal solute interacting with the membrane surface leading to gradual pore closure and plugging. Initial membrane permeability values for all trials were  $550\text{--}600 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$  compared with  $70\text{--}80 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$  when filtration was stopped. In all results cited in the literature experiments have been carried out at a relatively low MLSS concentration of  $6 \text{ g} \cdot \text{L}^{-1}$  or below (Table 2). In the current study the two-stage fouling phenomenon was only exhibited at the lowest biomass concentration investigated of  $6 \text{ g} \cdot \text{L}^{-1}$ . Results from operation at  $12 \text{ g} \cdot \text{L}^{-1}$  and  $18 \text{ g} \cdot \text{L}^{-1}$  displayed no such characteristic discontinuity suggests that the two-stage fouling phenomenon is a feature only of low biomass concentrations and/or short solids retention times and possibly only specific feedwater matrices.

Figure 3 shows  $t_{\text{filt}}$  to decrease linearly with increasing fluxes. If the foulant is assumed to be entrained in the flow of liquid through the membrane (i.e. not substantially removed by back diffusion) then it may be postulated that this

**Table 2.** Long-term operation data from Results of published studies

System type and volume (membrane)	Feed	Flux $L \cdot m^{-2} \cdot h^{-1}$	MLSS $g \cdot L^{-1}$	$t_{filt}$ hr	1st phase $dp \cdot dt^{-1}$ ( $t < t_{filt}$ ) $mbar \cdot min^{-1}$	2nd phase $dp \cdot dt^{-1}$ ( $t > t_{filt}$ ) $mbar \cdot min^{-1}$	Ref.
SS MBR 20L Tub. (0.05 $\mu m$ )	Syn. Mun.	10	1.8	550	$<8.3 \times 10^{-3}$	0.45	(2)
SS UASB Bench FP (0.22 $\mu m$ )	Syn. Mun.	30	0.3–0.55	360	$<6 \times 10^{-3}$	0.046	(3)
Subm. 40 L Tub. (0.1 $\mu m$ )	Mun.	9	3	240	$7 \times 10^{-4}$	0.01	(4)
Subm. 40 L Tub. (0.1 $\mu m$ )	Syn. Mun.	7	3	100	0.001	0.028	(4)
SS MBR 50 L HF. (0.22 $\mu m$ )	Syn. Mun.	22	5–6	1220	$1.8 \times 10^{-3}$	0.71	(10)
SS MBR 50 L HF. (0.22 $\mu m$ )	Syn. Mun.	25	5–6		$4 \times 10^{-3}$	0.045	(10)
SS MBR 50 L HF. (0.22 $\mu m$ )	Syn. Mun.	30	5–6		0.012	0.03	(10)
Subm. 40 L Tub. (0.03 $\mu m$ )	Syn. Ind.	4	6	192	$2.2 \times 10^{-3}$	0.14	Current study
Subm. 40 L Tub. (0.03 $\mu m$ )	Syn. Ind.	6	6	137	$5.1 \times 10^{-3}$	0.031	Current study
Subm. 40 L Tub. (0.03 $\mu m$ )	Syn. Ind.	8	6	74	0.1	0.047	Current study

linear relationship may be associated with a critical filtration volume or mass of foulant deposited per unit membrane area, with fouling increasing rapidly once this critical mass has been reached. It has been suggested that the period beyond  $t_{\text{filt}}$  reflects solids (i.e. sludge) deposition (2, 3). This being the case, and based on Darcian flow, an increase in flux would be expected to produce a proportional increase in  $dP/dt$  at  $t > t_{\text{filt}}$ . However, recorded data do not reflect this (Fig. 2 and Table 2), with the post- $t_{\text{filt}}$  fouling rate varying little with flux. Non-Darcian flow would be expected to further increase  $dP/dt$  with flux due to cake compression, solids migration, etc. In contrast it has been reported by Wen et al. (10) that the post  $t_{\text{filt}}$  fouling rate can actually decrease with applied flux. It must therefore be concluded that fouling at  $t > t_{\text{filt}}$  cannot be attributed solely to solids deposition. Differences in the cake structure and porosity will affect the rate of fouling and given the low number of long term tests conducted more work will need to be undertaken to further examine the phenomena.

Fouling rates measured during the flux step experiments both reveal an exponential relationship between  $dP/dt$  and flux, as found by other authors (4, 11). The fouling rates from the long-term trials at different fluxes also exhibit the same exponential relationship, with the value of the exponent being about the same as for that obtained for the flux-step experimental data but with the actual fouling rates being around an order of magnitude lower, viz.:

- $dP/dt = 0.0031e^{0.49J}$  for flux-step studies cf.  $0.0003e^{0.47J}$  for the long-term trials at  $6 \text{ g} \cdot \text{L}^{-1}$ ;

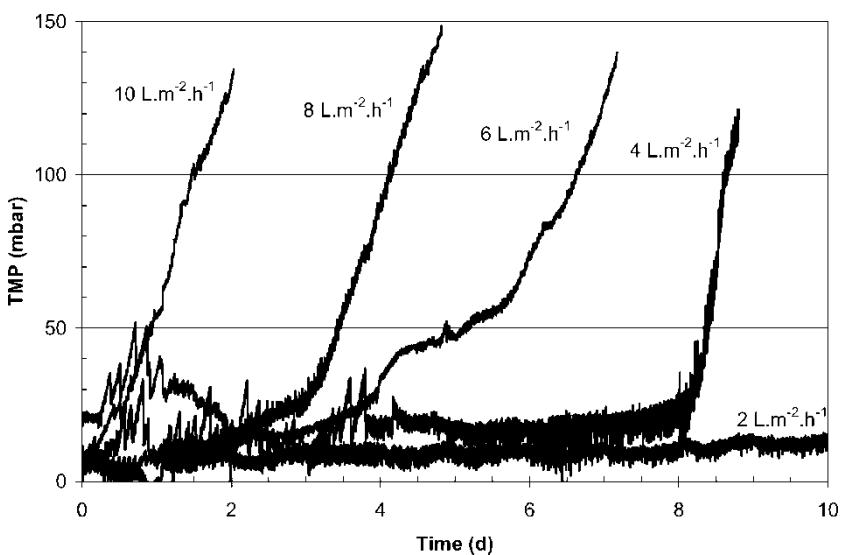


Figure 2. TMP transients for long term trials at a range of fluxes,  $6 \text{ g} \cdot \text{L}^{-1}$

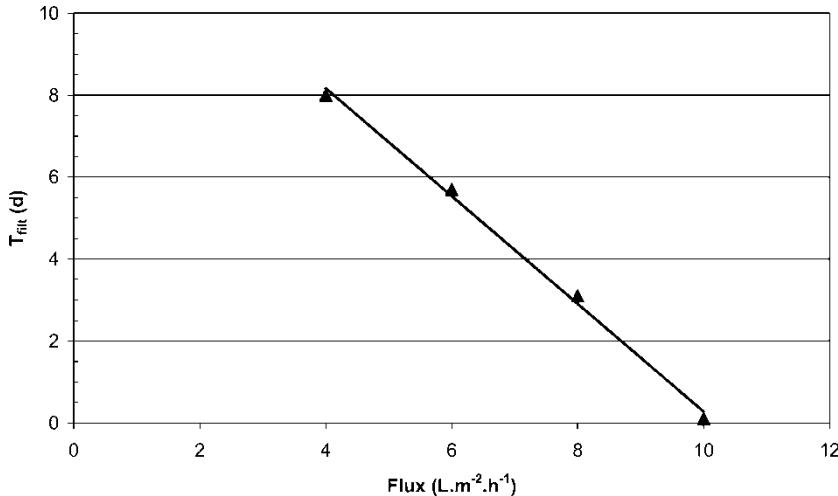


Figure 3.  $t_{\text{filt}}$  for a range of fluxes,  $6 \text{ g} \cdot \text{L}^{-1}$ .

- $dP/dt = 0.0073e^{0.30J}$  for the flux-step studies cf.  $0.0005e^{0.36J}$  for the long-term trials at  $12 \text{ g} \cdot \text{L}^{-1}$ ;
- $dP/dt = 0.0052e^{0.26J}$  for the flux-step studies cf.  $0.0009e^{0.26J}$  for the long-term trials at  $18 \text{ g} \cdot \text{L}^{-1}$ .

This relationship appears to apply at all flux values studied (Figs. 4, 5), such that the concept of a critical flux is questionable for these data.

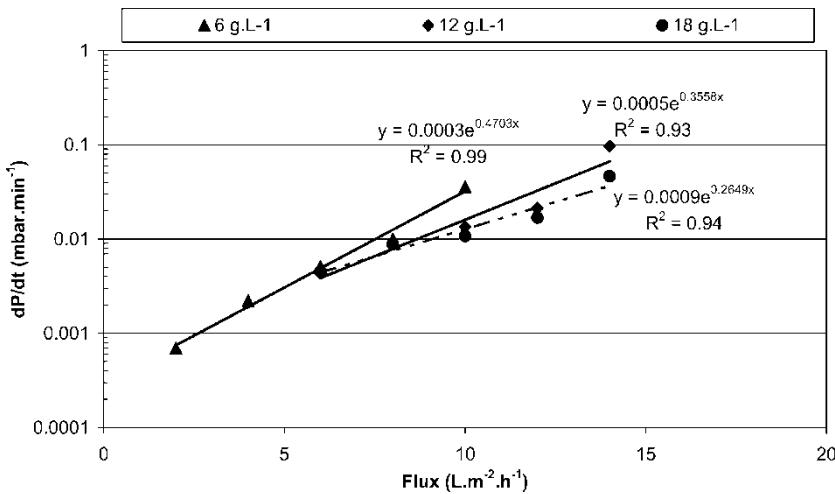


Figure 4. Long term fouling rates.

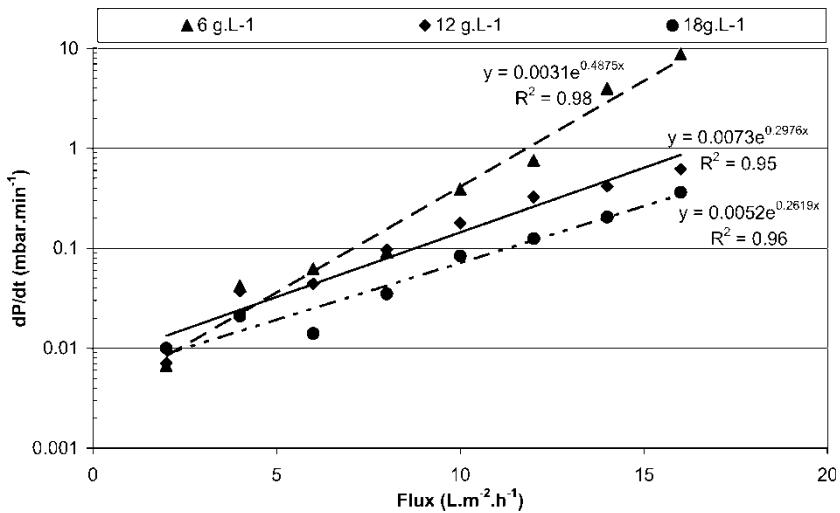


Figure 5. Flux step fouling rates at a range of MLSS concentrations.

Data from flux step tests carried out at all MLSS concentrations (Fig. 5) reveal MLSS levels of  $12\text{ g}\cdot\text{L}^{-1}$  and  $18\text{ g}\cdot\text{L}^{-1}$  to yield a similar permeability decline relationship. The benefit of operating at the higher MLSS concentrations only becomes apparent at higher fluxes, since very similar fouling rates are observed during both long term trials and short term flux step tests at fluxes below  $8\text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$  and  $6\text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$  for trials at  $12\text{ g}\cdot\text{L}^{-1}$  and  $18\text{ g}\cdot\text{L}^{-1}$  respectively.

Extracellular polymeric substances (EPS) have been identified as the primary foulants in MBRs (12). However, according to the biomass characteristics data from the current study (Table 3) there is no clear relationship between EPS levels (as defined by the soluble microbial products (SMP) and steam-extracted EPS concentrations) and fouling rate. This suggests that these parameters, as defined by standard extraction methods (8), are not a reasonable indicator of fouling propensity in this instance. Data shows an increase in total SMP concentrations at higher biomass concentrations and greater EPS protein concentration at longer sludge ages, lower EPS carbohydrate are evident. The actual chemical characteristics of the foulants are thus unclear from these studies.

### Application of an Existing Fouling Model

Data for membrane fouling prior to the end of stable operation ( $t_{\text{filt}}$ ) was used to ascertain the validity of a model recently proposed by Ognier et al. (2). The conceptual frame-work of the model can be briefly summarized as follows: when the membrane operates at values lower than the critical flux, the

**Table 3.** Biomass characteristics at MLSS 6, 12 and 18 g·L<sup>-1</sup>

Determinant	Concentration (st.dev.)		
	6 g·L <sup>-1</sup>	12 g·L <sup>-1</sup>	18 g·L <sup>-1</sup>
Suspended Solids (mg·L <sup>-1</sup> )	6.2 (0.8)	12.55 (1.8)	17.4 (1.3)
Volatile Suspended Solids (mg·L <sup>-1</sup> )	5.4 (0.7)	11.36 (1.5)	(15.9) (1.2)
Capillary suction time (s)	148.8 (82.5)	46.1 (27.3)	61 (21.1)
Particle size d <sub>50</sub> (μm)	86.7	105.4	75.1
Soluble microbial products (SMP <sub>c</sub> )–Carbohydrate (mg·L <sup>-1</sup> )	11.35 (7.4)	17.26 (4.9)	22.7 (7.2)
Soluble microbial products (SMP <sub>p</sub> )–Protein (mg·L <sup>-1</sup> )	18.7 (9.6)	34.52 (8.2)	48.5 (13.7)
Extracellular polymeric substances (EPS <sub>c</sub> )–Carbohydrate (mg·g <sup>-1</sup> )	13.6 (2.7)	10.5 (3.8)	9.3 (5.1)
Extracellular polymeric substances (EPS <sub>p</sub> )–Protein (mg·g <sup>-1</sup> )	20.3 (5.8)	31.1 (8.64)	37.3 (12.4)

soluble particles interact with the membrane surface, thus reducing the number of open pores really involved in the filtration ( $n_p$ ). When the actual flux in open pores becomes equal to the critical flux (previously determined with flux-step tests) a sudden increase in TMP takes place due to solids accumulation and cake formation on the membrane surface.

The model is expressed as:

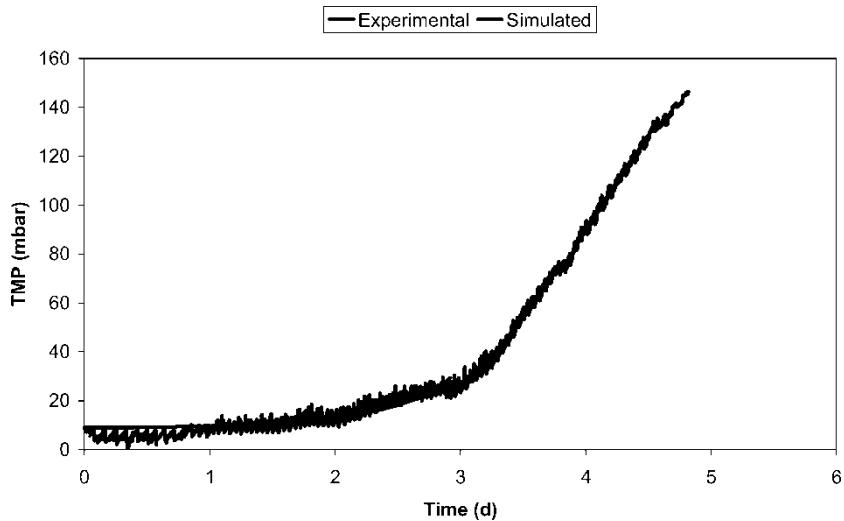
$$TMP(t) = \frac{TMP_0}{1 - ((\alpha \cdot TMP_0 \cdot t^2)/2)} \quad (1)$$

where  $TMP_0$  is the transmembrane pressure at the start of the filtration and  $\alpha$  is an adjustable parameter incorporating both membrane and suspension characteristics. Particularly,  $\alpha$  includes two proportionality constants respectively referring to the mass transport of foulants towards the membrane surface and the gradual decrease in open pore area;  $\alpha$  takes into account geometric and physical membrane properties (pore section  $S_p$  and hydraulic resistance  $R_p$ ). The value of  $\alpha$  can be determined from experimental data recorded prior to the critical filtration time.

Different sets of experimental data obtained under process steady-state conditions have been used to calibrate and then validate the model. The model calibration has been carried out with data obtained from the test at 8 L m<sup>-2</sup> h<sup>-1</sup> and 6 g L<sup>-1</sup> MLSS, by estimating the  $\alpha$  parameter value that minimizes the sum (RSS) of the square differences between measured and calculated data:

$$RSS = \sum_{j=1}^N (TMP_{j,measured} - TMP_{j,calculated})^2 \quad (2)$$

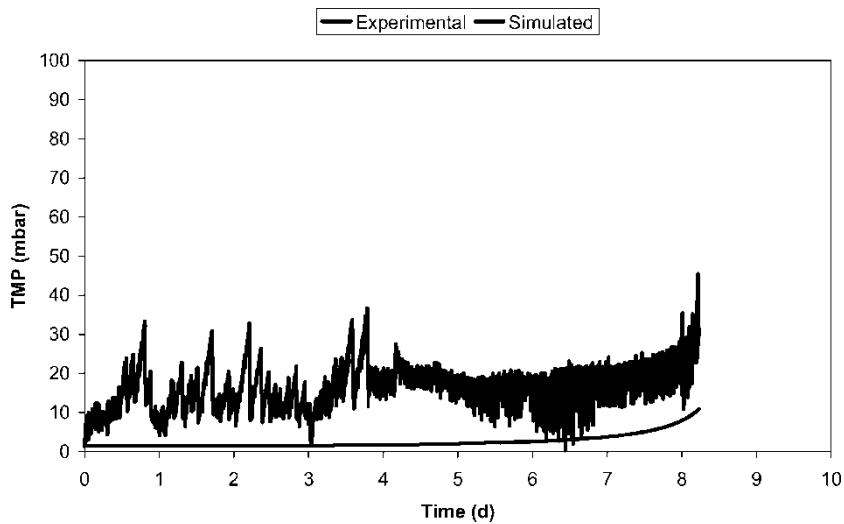
Calibration provided a  $\alpha$  value of  $2.46 \times 10^{-12}$  m kg<sup>-1</sup> (Figure 6).



**Figure 6.** Mathematical calibration of the  $\alpha$  value at  $8 \text{ L m}^{-2} \text{ h}^{-1}$ .

Commonly, the prediction effectiveness of a mathematical model requires a validation phase aimed to evaluate the model behaviour when the mathematically determined value of a given parameter ( $\alpha$  in this case) is used to predict another series of data.

Therefore the  $\alpha$  value obtained at  $8 \text{ L m}^{-2} \text{ h}^{-1}$  was adopted in order to verify the capability of the model to predict long term fouling profiles at



**Figure 7.** Model validation at  $4 \text{ L m}^{-2} \text{ h}^{-1}$ .

**Table 4.** Optimal values of the  $\alpha$  parameter for different long fluxes

$J \text{ L m}^{-2} \text{ h}^{-1}$	Best fitting $\alpha$ value $\text{m} \cdot \text{kg}^{-1}$
2	$3.70 \cdot 10^{-13}$
4	$5.36 \cdot 10^{-12}$
6	$2.51 \cdot 10^{-13}$
8	$2.46 \cdot 10^{-12}$

other flux values ( $2 \text{ L m}^{-2} \text{ h}^{-1}$ ,  $4 \text{ L m}^{-2} \text{ h}^{-1}$ ,  $6 \text{ L m}^{-2} \text{ h}^{-1}$ ). The percent error, estimated according to Equation (3), ranged between 23.3% and 84.5%, thus showing a poor fit of the model with the experimental data collected at different fluxes but under otherwise identical conditions of calibration in terms of MLSS, SRT, and membrane cleaning conditions.

$$\%Error = \frac{\sum_{j=1}^N |TMP_{measured} - TMP_{simulated}|}{\sum_{j=1}^N TMP_{measured}} \quad (3)$$

The trend of simulated and experimental data at  $4 \text{ L m}^{-2} \text{ h}^{-1}$  is described in Fig. 7 and it demonstrates the relevant underestimation of the model with respect to the real TMP profile. Table 4 shows the best fitting values of  $\alpha$  for the series of data under consideration.

Recorded experimental data are somewhat scattered, partly accounting for the poor fit. Another possible contributing factor may lie with the conceptual approach of the model. Many components are incorporated into the  $\alpha$  parameter, which is assumed to be constant with time. Given that operation is over a prolonged period and fouling constituents are likely to change because of the heterogeneity of the feed suspension this assumption may not be valid, such that  $\alpha$  should more correctly be expressed as a function of time.

## CONCLUSIONS

Experiments conducted on the application of a submerged tubular membrane bioreactor for treatment of a BTEX-laden produced water analog have revealed a number of facets of fouling behavior.

- The flux step data yields the same exponent value for the  $dP/dt: J$  correlation as the long-term experiments. Short-term fouling experiments may thus be helpful in detecting fouling under sustainable flux operation and allow comparative studies of different feeds, operating conditions, etc., but true sustainability of low flux operation is only revealed through long-term trials where fouling rates may be an order of magnitude lower than that obtained for short term flux step tests.

- Fouling rate  $dP/dt$  is exponentially related to flux for both short term and long term trials and no change is apparent above or below the notional transitional flux for  $dP/dt$  vs flux relationship.
- A two-stage fouling phenomenon is only exhibited at the lowest biomass concentration studied of  $6\text{ g}\cdot\text{L}^{-1}$ , but is not evident at higher biomass concentrations.
- There is no clear relationship between SMP/EPS levels and fouling rate according to data produced from this study.
- The critical filtration time for low-fouling operation ( $t_{\text{filt}}$ ) decreases with increasing flux and may be related to total filtrate volume, hence possibly a critical amount of solids/solute deposited/adsorbed per unit membrane area.
- An equation describing dynamic trend in  $P$  vs.  $t$  provided by Ognier et al. (2) does not appear to predict fouling behaviour, as defined by data recorded in the current study. This may be due to non-steady behaviour impacting on the parameter  $\alpha$ .
- For  $t > t_{\text{filt}}$ , recorded pressure trends are inconsistent with solids deposition according to simple Darcian relationships where fouling rate due to solids accumulation should increase with flux.

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