Impact of Suspended Solids Concentration on Sludge Filterability in Full-scale Membrane Bioreactors

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Abstract

The relation between activated sludge filterability and mixed liquor suspended solids (MLSS) concentration in membrane bioreactors (MBRs) is framed in a single hypothesis, explaining results seemingly contradictory. A total of 44 activated sludge samples were collected and analyzed on a variety of parameters, i.e. filterability, MLSS concentration, soluble microbial products (SMP) concentrations and particle size distribution in the range of 2-100 μm and of 0.4-5.0 μm. The sludge filterability was assessed by using the Delft Filtration Characterization method (DFCm). In order to investigate the impact of MLSS concentration, identical samples were diluted with permeate. Results showed that dilution of the samples led to an increased activated sludge filterability, but only when the starting MLSS concentration was below the apparent critical value of 10.5 g/L. As opposed, the filterability of sludge with MLSS concentrations above 10.5 g/L, and which was characterized by a moderate to good filtration quality, i.e. $\Delta R_{20}<1$ [$\times 10^{12} m^{-1}$], worsened when diluted. The specific resistance times the particle concentration of a cake layer obtained when filtrating sludge of moderate to good filterability and MLSS concentration above the apparent critical value, was 5.5 times smaller compared to the cake layer of sludge with MLSS concentration below the critical value. Results from SMP assessment and particle counting in the range 2-100 μm showed that reduction in sludge mass and de-flocculation occurred, upon dilution of all samples. However, when diluting sludge samples with MLSS concentrations exceeding 10.5 g/L and which were characterized by a moderate to good filtration characteristics, there was also release of particles below 0.4 μm, opposite to dilutions of samples with MLSS concentrations below 10.5 g/L. We postulate that sludge, which is characterized by a moderate to good filterability, having an MLSS concentration above the apparent critical value of 10.5 g/L, is likely to retain particles smaller than 0.4 μm in its mass, as opposed to sludge with MLSS concentration below the apparent critical value. Our work indicates that there are optimal MLSS concentration ranges in MBR technology, to promote good filterable sludge quality in order to avoid fouling.

Keywords

Membrane bioreactors; filterability; mixed liquor suspended solids concentration.

1. INTRODUCTION

One of the main advantages of membrane bioreactor (MBR) technology is the possibility to work at high mixed liquor suspended solids (MLSS) concentrations (Judd, 2008). However, one of the main constraints is the need to control membrane fouling (Judd, 2008). Membrane fouling can be defined as the sum of processes leading to flux deterioration due to surface or internal pore blockage of the membranes (Judd, 2006). In the early stages of MBR technology, MLSS concentration was considered one of the possible fouling parameters (Yamamoto et al., 1989). Nowadays, it is generally accepted that MLSS concentration alone is a poor indicator of biomass fouling propensity (Jefferson et al., 2004). However, it is also generally understood that biological flocs, i.e. the suspended fraction of the activated sludge, play a key role in the fouling layers built up (Le-Clech et al., 2006). In short, MLSS concentration alone is considered to be a poor indicator of fouling propensity, but the suspended fraction of the sludge does play a role in the build-up of the cake layer.

Literature reports on the effect of MLSS concentration on membrane filtration provide apparently contradictory results. Meng et al. (2007) observed that membrane fouling increased exponentially with increasing MLSS concentrations. Le-Clech et al. (2003) saw no effect on fouling for a shift from 4 to 8 g/L in MLSS concentration, but a significant increase in critical flux occurred for MLSS concentrations of 12 g/L. The relation between the fouling propensity and MLSS concentration is therefore not clarified. In practice, in membrane tanks of full-scale MBRs, MLSS concentrations are mainly determined by the membranes manufacturers. Optimal MLSS concentrations for MBR activated sludge, aiming at an optimal sludge filtration quality, are not defined.

The Delft Filtration Characterization method (DFCm) was defined to characterize the sludge fouling potential, by measuring sludge filterability through a pre-defined procedure (Evenblij et al., 2005). The DFCm was applied in several MBR installations scattered around Europe (Moreau, 2010, Lousada-Ferreira, 2011, Krzeminski, 2013). The boundaries of the DFCm were clarified, namely on data processing, accuracy, reproducibility, reliability and applicability, leading to the conclusion that the DFCm was a convenient tool to research how filterability can be influenced by activated sludge characteristics such as MLSS concentrations (Lousada-Ferreira et al., 2014). In our previous work, the membrane tank sludge filterability and MLSS concentrations were measured at four full-scale MBRs scattered around Europe, during weekly campaigns at summer and winter seasons (Lousada-Ferreira et al., 2011). The filterability was measured according to the DFCm, which provides ΔR_{20} results. The ΔR_{20} presents the cake layer resistance obtained after extracting 20 L of permeate per membrane area. Further information on the DFCm is provided in the materials and methods section. A classification linking the assessed ΔR_{20} and activated sludge filterability was defined, showing that for values of $\Delta R_{20} < 0.1$ [×10 ^{12}m^{-1}] sludge filterability is good (Geilvoet, 2010). Figure 1 presents the results obtained at the 4 full-scale European MBRs, with sludge collected at the membranes tanks and measured immediately after collection.

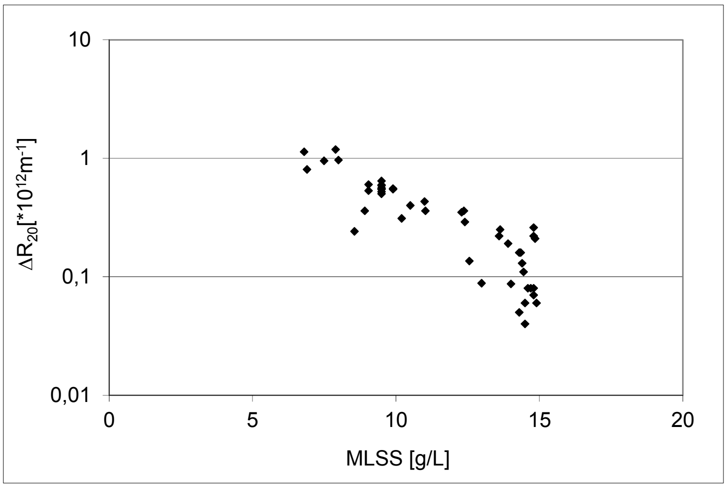

Figure 1 Filterability and MLSS concentration at full-scale MBRs

Figure 1 shows that the sludge filterability can be good, i.e. filterability assessed by the DFCm with a ΔR_{20} value below 0.1 [×10 m^{-1}], at MLSS concentrations around 15 g/L. The lower range is insufficiently captured by the data, however for higher ranges there is a clear improvement of filterability with increasing MLSS concentrations. Nevertheless, Chang and Kim (2005) measured an increased cake resistance with an MLSS concentration shift from 0.09 to 3.7 g/L, Fang and Shi (2005) an increase in total resistance with a shift from 2.4 to 9.6 g/L and Psoch and Schiewer (2006) an increased fouling potential with a shift from 3 to 10 g/L in MLSS concentration. Therefore, it seems logical to assume that good sludge filterability might also be possible in sludge with low MLSS concentrations. The above results lead us to the following hypothesis.

In our present research, we hypothesize that the relation between filterability and MLSS concentration can be explained by the characteristics of the activated sludge mass. In sludge with an MLSS content below an apparent critical concentration, fouling particles are available in the free water of the activated sludge bulk. As opposed, in activated sludge with a high MLSS content, i.e. above a *critical* concentration, and moderate to good filterability, fouling particles become entrapped in the sludge matrix. The critical MLSS concentration is understood as the MLSS concentration above which the entrapment of particles results in a filterability improvement. Figure 2 clarifies the definitions used in the above hypothesis and henceforth in this research.

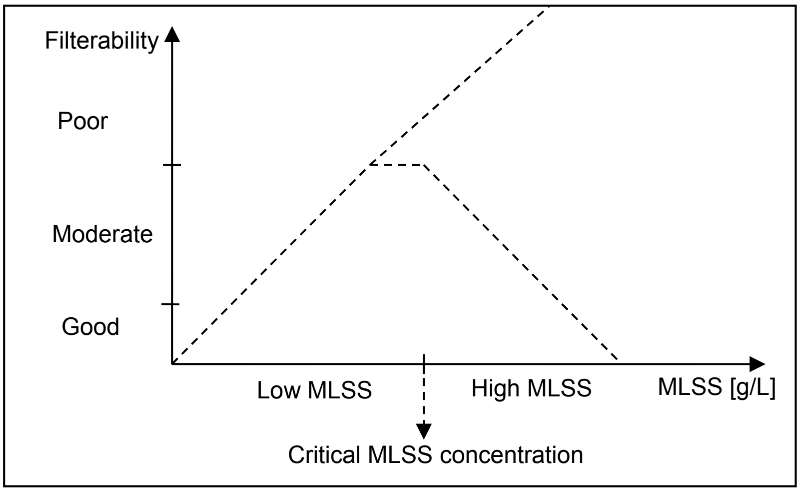

Figure 2 Schematics of concepts (Lousada-Ferreira, 2011)

In Figure 2 two MLSS concentration ranges are defined, namely, ''Low MLSS'' and ''High MLSS'' as below and above the critical MLSS concentration. According to the hypothesis, increasing the MLSS concentration of sludge with ''High MLSS" and moderate to good filtration quality, leads to a filterability improvement because more fouling particles remain entrapped in the activated sludge mass. As opposed, sludge in the "Low MLSS'' range improves its filterability when the MLSS concentration is reduced because the amount of fouling particles will also be reduced. To test the aforementioned hypothesis, sludge with different MLSS concentrations were collected at full-scale MBRs and further diluted to obtain sludge with lower MLSS concentrations. If our hypothesis is proven true then there are optimal MLSS concentrations in MBR systems to promote good filterable sludge quality in order to avoid fouling.

2. METHODOLOGY

In this research, 44 activated sludge samples, further referred to as non-diluted samples, were collected from the membrane tanks of full-scale MBRs and submitted to the following measurements: filterability, MLSS concentration, soluble microbial products (SMP) concentrations and particle size distribution in the range of 2-100 µm and of 0.4-5.0 µm. Identical samples were diluted with permeate, in a fast procedure of about 30 minutes, to obtain sludge with different MLSS concentrations, further referred to as dilution samples. The dilution samples were then submitted to the same analyses as the original, non-diluted, samples.

The experiments were organized in sets, namely 44 sets of experiments. For each set, a minimum of three filtration measurements were performed, followed by the abovementioned physical-chemical analyses. One filtration measurement was performed with the original non-diluted sample and the other two with diluted samples. The diluted samples were prepared according to the methodology described in Lousada-Ferreira *et al.* (2010). The experiments were performed with the following goals: (i) set 1 to 22 to characterize the MBR activated sludge; (ii) set 23 to 28 to compare sludge from membrane and aeration tanks, within one full-scale MBR installation; (iii) set 29 to 32 to compare sludge from different MBRs.

The sludge samples were collected at four full-scale installations further referred to as MBR A to MBR D. Details of the MBR installations are provided in Table 1. In this research, 88% of the sludge samples were collected at MBR A. All samples were collected at the upper-decks of the MBR membrane tanks, in central areas, with the following exception. MBR A has two separate membrane tanks plus 6 other tanks, intended for carbonaceous and nutrient removal. At MBR A, before reaching the membrane tanks the sludge is subjected to extra aeration in a tank further designated as aeration tank. Sludge samples for set 23 to 28 were collected simultaneously at the membrane and aeration tank of MBR A.

Table 1 Characteristics of full-scale MBRs.

WWTP		MBR A	MBR B	MBR C	MBR D
Population Equivalent	-	13,000	28,000	18,500	23,150
Wastewater treatment	-	CAS+MBR	CAS+MBR	CAS+MBR	MBR
Location of the membranes	-	Submerged in separate tanks	Submerged in separate tanks	Side-stream membranes	Submerged in separate tanks
Membrane Supplier	-	Toray (Flat sheet)	Zenon (Hollow Fiber)	Norit (Multi-tube)	Zenon (Hollow Fiber)
Membrane pore size	μm	0.08	0.04	0.03	0.04
Total membrane area	m^2	4,110	10,560	2,436	20,160
Flux (design)	$L/m^2.h$	24.3	33.6	55	37.5
Flux (average operation)	$L/m^2.h$	18.2	33	45	25
MLSS	g/L	12	11	9.2	10.2
Return ratio $^{(1)}$	-	1.5	5.8	-	-
Sludge age	d	20	21	42	33

Key: $^{(1)}$ return ratio from membrane tanks to carbonaceous or nutrient removal tanks.

2.1 Filterability

The filterability was measured according to the Delft Filtration Characterization method (DFCm), defined by Evenblij et al. (2005) and further clarified in Lousada-Ferreira et al. (2014). The DFCm measures filterability of activated sludge through a single transmembrane (TMP) filtration measurement at constant flux and cross-flow velocity, performed in a side-stream membrane installation with a defined operation and cleaning protocol (Evenblij et al., 2005). The DFCm provides three types of results: ΔR_{20} parameter, $\alpha_R \times c_i$ product and s coefficient. The ΔR_{20} is the cake layer resistance obtained after extracting 20 L of permeate per membrane area; the α_R is the specific cake resistance which is multiplied by c_i indicating the concentration of the cake layer particles; the s coefficient is the compressibility coefficient of the cake layer (Geilvoet, 2010, Lousada-Ferreira et al., 2014). The ΔR_{20} parameter, contrary to the $\alpha_R \times c_i$ product and s coefficient, does not have a direct physical meaning. The parameter was defined to simplify the comparison between filtration curves. A classification linking the assessed ΔR_{20} and activated sludge filterability was defined (Geilvoet, 2010), as follows. For values of $\Delta R_{20} < 0.1$ [$\times 10^{12} m^{-1}$] sludge filterability is good; if $1 > \Delta R_{20} > 0.1$ filterability is moderate; if $\Delta R_{20} > 1$ filterability is poor.

2.2 Particle counting

The particle counting in the range of 2-100 μm were performed as described in our previous work (Lousada-Ferreira et al., 2011). The particle counting measurements in the range of 0.4-5.0 μm were performed in a HIAC ChemShield particle counter. MBR activated sludge was filtered through a 589^2 Schleicher & Schuell paper filter, with pore size between 7 and 12 μm. The free water, obtained through the aforementioned filtration step, was then diluted, by a factor of 100, with demineralized water, immediately before being submitted to particle counting measurements. The obtained particle counting results were normalized, i.e. the concentration of particles in a given size range was divided by the size interval and the particle size presented in logarithmic scale. The β value, i.e. the slope of normalized particle counting data, was used to compare between different sludge samples.

2.3 MLSS and SMP concentrations

MLSS concentration was determined according to standard methods. The SMP concentrations, namely, proteins and polysaccharides concentrations, were determined according to the methods proposed by Lowry et al. (1951) and Dubois et al. (1956), respectively.

3. RESULTS AND DISCUSSION

3.1 Filterability and cake layer parameters

The average results obtained in sets 1 to 22, analyzed to characterize the MBR activated sludge structure, are shown in Figure 3. Figure 3 shows that the filterability of the diluted samples, varies according to the MLSS concentration of the non-diluted samples. In this research, the apparent critical

MLSS concentration was found to be 10.5 g/L. MBR activated sludge with low MLSS concentration, i.e. MLSS ≤10.5 g/L, has increased filterability when diluted. In contrast, diluting MBR activated sludge with high MLSS concentration, i.e. MLSS>10.5 g/L, provides samples with worse filterability. The obtained results are in agreement with our previous work (Lousada-Ferreira et al., 2010) and support the initial hypothesis.

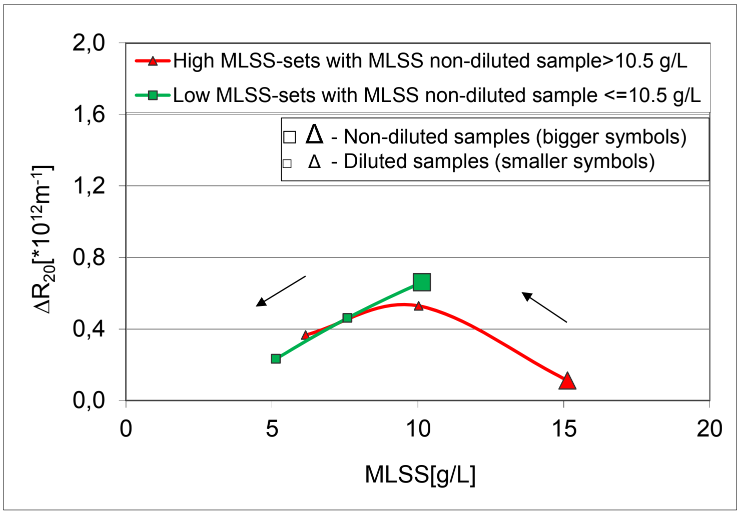

Figure 3 Average filterability vs. average MLSS concentration of membrane tank sludge (Lousada-Ferreira, 2011)

The MLSS concentration has a direct impact on sludge viscosity, i.e. samples with lower MLSS concentration will have a lower viscosity (Rosenberger et al., 2002, Hasar et al., 2004). Suspensions with low viscosity will more easily create turbulent regimes, which are considered preferable for membrane filtration (Rosenberger et al., 2002). Therefore, theoretically, an improvement in filterability is expected when sludge is diluted. In our present research, filterability of original samples with MLSS concentration above 10.5 g/L and good to moderate filtration quality, does not improve when diluted.

Statistical parameters for "Low MLSS" and "High MLSS" sets are presented in Table 2 for non-diluted, dilution 1 and dilution 2 samples. Table 2 shows that, in "Low MLSS" sets, the filterability of the non-diluted samples varied between 1.6 and 0.3 [$\times 10^{12} m^{-1}$], i.e. between poor and moderate filterability (Geilvoet, 2010). No non-diluted samples with good filterability, i.e. with ΔR_{20} below 0.1, and MLSS concentration ≤10.5 g/L were obtained in this research. Nevertheless, an improvement of filterability when sludge is diluted agrees with the theoretically expected result, as explained in the previous paragraph. Therefore, it can be assumed that an increase in filterability by diluting occurs in MBR activated sludge samples with low MLSS concentration, regardless of the filtration quality of the original sludge.

Table 2 Statistical parameters for MLSS concentration and ΔR_{20} results in sets 1 to 22.

	Non-diluted		Dilution 1		Dilution 2	
	MLSS [g/L]	ΔR_{20} [$\times 10^{12} m^{-1}$]	MLSS [g/L]	ΔR_{20} [$\times 10^{12} m^{-1}$]	MLSS [g/L]	ΔR_{20} [$\times 10^{12} m^{-1}$]
			"Low MLSS"(1)			
Number of samples	4	4	4	4	4	4
Average	10.1	0.7	7.6	0.5	5.1	0.2
St. deviation [%]	4	91	4	68	28	32
Maximum	10.5	1.6	8	0.9	6.3	0.3
Minimum	9.7	0.3	7.4	0.3	3.2	0.2
			"High MLSS"(2)			
Number of samples	18	18	18	18	18	18
Average	15.1	0.1	10	0.5	6.2	0.4
St. deviation [%]	12	110	24	97	45	94
Maximum	18.3	0.5	15.2	1.8	10.7	1.4
Minimum	10.8	0.01	6	0.08	2.2	0.02

Key: (1) sets with MLSS of original sample >10.5 g/L; (2) sets with MLSS of original sample ≤10.5 g/L.

In "High MLSS" sets, the filterability of the original samples varied between 0.5 and 0.01 [$\times 10^{12} m^{-1}$], as shown in Table 2. No non-diluted samples with poor filtration quality, i.e. with ΔR_{20} above 1, and MLSS concentration above 10.5 g/L were obtained. An increase in viscosity in activated sludge with increased MLSS concentration should lead to decreased filterability. However, whereas some authors indeed measured increasing fouling with a shift in MLSS concentration from 4 to 18 g/L (Meng et al., 2007), others reported higher critical flux in sludge with 12 g/L (Le-Clech et al., 2003). Our own results show that MBR activated sludge with MLSS concentrations above 10.5 g/L, and good to moderate filtration quality, provides diluted samples with worse filterability.

Table 3 shows the statistics of the cake layer parameters, namely the $\alpha_R \times c_i$ product and s coefficient. As explained in our previous research the DFCm produces a hardly compressible cake layer (Lousada-Ferreira et al., 2014). In the DFCm the compressibility coefficient results are mainly between 0 and 0.3 (Lousada-Ferreira, 2011, Krzeminski, 2013), while theoretically it varies between 0 and 1, indicating no compression to total compression, respectively. In the DFCm the cake layer mass and specific cake resistance are the main contributors of the total measured resistance (Lousada-Ferreira et al., 2014).

Table 3 Statistical parameters for the $\alpha_R \times c_i$ product and s coefficient results in sets 1 to 22.

	Non-diluted		Dilution 1		Dilution 2	
	$\alpha_R \times c_i$ [$\times 10^{-3}$m^{-2}]	s	$\alpha_R \times c_i$ [$\times 10^{-3}$m^{-2}]	s	$\alpha_R \times c_i$ [$\times 10^{-3}$m^{-2}]	s
			“Low MLSS”$^{(1)}$			
Number of samples	4	4	4	4	4	4
Average	33	0.02	24	0.05	12	0.04
St. deviation	29	0.03	15	0.04	4	0.04
			“High MLSS”$^{(2)}$			
Number of samples	18	18	18	18	18	18
Average	6	0.03	26	0.08	21	0.15
St. deviation	7	0.06	24	0.08	15	0.12

Key: $^{(1)}$ sets with MLSS of original sample <10.5 g/L; $^{(2)}$ sets with MLSS of original sample >10.5 g/L.

The non-diluted samples’ results in Table 3 show that “High MLSS” samples have an average $\alpha_R \times c_i$ which is 5.5 times lower than “Low MLSS” samples. The latter results would not be possible if samples with higher MLSS concentration would always result in increased fouling, as obtained by Meng et al. (2007).The $\alpha_R \times c_i$ of the dilution samples shows that while for “Low MLSS” samples the $\alpha_R \times c_i$ decreases with dilution, for “High MLSS” the result is opposite, i.e. dilution samples have an $\alpha_R \times c_i$ product at least 3.5 times higher than the non-diluted samples. Our results show that diluting sludge with high and low MLSS concentration, even when comparing sets where the original, non-diluted sludge had the same filterability, does not produce dilutions with comparable cake layer parameters.

Geilvoet (2010) describes the membrane cake layer concerning the $\alpha_R \times c_i$ parameters, as follows. For a determined value of the $\alpha_R \times c_i$ product, the cake layer is either thinner with a higher specific cake resistance, either thicker with a lower specific cake resistance. Moreover, it can be speculated that the thickness of the cake layer should in principle be determined by the size of the attached flocs, i.e. bigger flocs should result in thicker cake layers and *vice-versa*. Diluting should, in principle, result in a de-flocculation of particles obtaining sludge with smaller flocs. Consequently, when submitting dilution samples to membrane filtration experiments, the obtained cake should be composed by smaller flocs. Eventually, due to a reduction of the sludge mass, also the amount of material attached to the membrane should be reduced. The results in table 3 of “High MLSS” sets show that dilution samples have a higher $\alpha_R \times c_i$ than original samples. Therefore, if due to diluting the cake layer should be thinner and composed by smaller flocs, then the α_R of the diluted sample has to be higher compared to the α_R of the original non-diluted sample. It can be speculated that if “High MLSS” samples release extra fouling particles when diluted, such as colloidal particles previously attached to the flocs, these particles could fill the cake layer pores and increase the α_R. In contrast, if “Low MLSS” samples contain less fouling particles or not at all, the α_R either remains basically the same or it is reduced. Our results indicate that “High MLSS” sludge has a different composition than “Low MLSS” sludge, which might result in a particular structural arrangement, supporting our initial hypothesis. The differences in structure might be revealed by particle counting measurements.

Figure 4 shows results of single filtration tests, identified by sets. Figure 4A shows the filterability and MLSS concentration performed with sludge collected simultaneously in the membrane and aeration tanks of MBR A, identified by the respective pairs of sets. A second order polynomial curve was fitted to each pair of sets and the respective R^2 shown in Figure 4A. Figure 4B shows the results obtained with sludge from MBRs B, C and D.

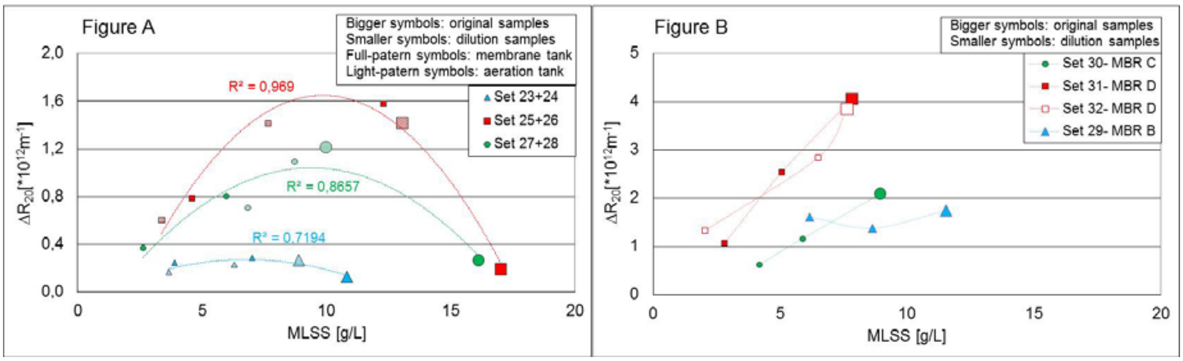

Figure 4 Filterability vs. MLSS concentration of sludge collected from: A- aeration tank and membrane tank of MBR A; B- membrane tanks of MBRs B, C and D.

Figure 4A shows that the sludge filtration quality in the membrane tank, i.e. the filterability of the membrane tank original, non-diluted samples, is better than in the aeration tanks. Simultaneously, the MLSS concentration in the membrane tanks is higher than in the aeration tanks. The observed results can be explained by the sludge concentration effect occurring during sludge filtration in the membrane tank of MBR A, where a low sludge return flow to the carbonaceous and nutrient removal tanks is applied (Table 1).

Based on our hypothesis, we postulate that there is a critical MLSS concentration for MBR filtration, potentially resulting in good to moderate sludge filtration qualities at both low and high MLSS concentrations. The curves depicted in Figure 4A indeed confirm our hypothesis. The fitting of each curve to the results of diluted and original, non-diluted samples, simultaneously collected in both tanks, are shown by the R^2. Figure 4A shows that the obtained R^2 is between 0.72 and 0.97, which is relatively high.

Figure 4B confirms the results obtained at MBR A, i.e. sludge with MLSS concentration below the apparent critical value produces dilutions with improved filterability. Strikingly, in set 29- MBR B, original and dilution samples have approximate similar filterability results, i.e. varying from 1.74 to 1.38 [$\times 10^{12} m^{-1}$]. It should be noted that, when the set 29 sludge was collected, an unusual growth of filamentous bacteria, namely *Nocardia amarae*, was reported (Moreau, 2010), which did not occur in the remaining sets. Possibly, the presence of these filamentous bacteria negatively determines the sludge filterability, irrespective the degree of dilution. Overall, the samples collected at other MBRs confirmed the results obtained at MBR A, supporting our initial hypothesis.

Filterability and MLSS concentration results obtained by other researchers (Moreau, 2010, Gil et al., 2011) support the existence of a critical MLSS concentration around 10 g/L. The latter references researched several pilot and full-scale MBR installations with municipal and industrial wastewater, applying the DFCm as method to measure the sludge filtration quality. Moreau (2010), in field campaigns at 5 pilot-scale and 3 full-scale municipal MBRs, shows a maximum of the $\alpha_R \times c_i$ product and s coefficient at MLSS concentrations of 10 g/L. Gil et al. (2011), in sludge samples of 11 full-scale industrial MBRs, measured filterability improvement in sludge samples with MLSS concentration above 10 g/L. Therefore, it does not seem likely that the critical MLSS concentration is scale, site or feed water specific. Nevertheless, our own research and the aforementioned references all apply the DFCm. It is possible that, if the sludge quality is measured through other methods, the absolute numbers of a critical MLSS concentration are different. The value of 10,5 g/L as MLSS critical concentration should be used as reference, but not extrapolated without further research.

3.2 SMP

The SMP, in particular proteins and polysaccharides, have been identified as fouling particles in MBR activated sludge (Lesjean et al., 2005, Rosenberger et al., 2005). The statistical parameters of the proteins and polysaccharides results are shown in Table 4.

Table 4 Statistical parameters for proteins and polysaccharides in sets 1 to 22.

	Non-diluted		Dilution 1		Dilution 2	
	Proteins [mg/g MLSS]	Polysaccharides [mg/g MLSS]	Proteins [mg/g MLSS]	Polysaccharides [mg/g MLSS]	Proteins [mg/g MLSS]	Polysaccharides [mg/g MLSS]
			“Low MLSS”(1)			
Number of samples	1	1	1	1	1	1
Average	2.12	1.67	2.72	2.94	4.61	1.98
St. deviation	-	-	-	-	-	-
			“High MLSS”(2)			
Number of samples	16	17	17	17	17	17
Average	1.06	0.66	1.3	1.01	2.19	1.56
St. deviation	0.44	0.45	0.64	1.08	1.22	2.05

Key: (1) sets with MLSS of original sample >10.5 g/L; (2) sets with MLSS of original sample ≤10.5 g/L.

The results of proteins and polysaccharides concentrations shown in Table 4 indicate that both in “Low MLSS” as in “High MLSS” sets, dilution samples have more SMP than original samples. However, in this research dilutions are produced with permeate, which has SMP and no suspended particles. Therefore, it is possible that the increase in SMP concentration from original to dilution samples is a consequence of the applied methodology as well as from de-flocculation of the original, non-diluted samples.

In this research no linear relation was found between filterability and SMP results (Lousada-Ferreira, 2011). The latter conclusion was also obtained by authors monitoring filterability and SMP in full-scale installations (Lyko et al., 2008, Moreau, 2010). Successful predictions of filterability were only obtained when a combination of parameters was taken into account (Van den Broeck et al., 2011). The results shown in Table 4 seem to indicate that “Low MLSS” sets have more SMP than “High MLSS” sets. However, there was only one set measured at “Low MLSS” and in the latter set the non-diluted sample had a poor filtration quality (result not shown), which was not obtained in any of the “High MLSS” sets. Therefore, in this research, clear conclusions regarding the amount of SMP in “Low MLSS’’ and “High MLSS” sets are not warranted.

3.3 Particle counting in the range of 2-100 μm

Figure 5 shows the average number of particles in “Low MLSS“ and “High MLSS“ sets in the range 2-100 μm. The particle counter Met One PCX applied in this research, counts particles in intervals of 0.5 μm, starting at 2 μm and ending at 100 μm. Our previous work showed that MBR activated sludge particles in the range 2-10 μm are not successfully counted by the Met One PCX (Lousada-Ferreira et al., 2011). Therefore, Figure 5 shows average results in the identified reliable range, which is 10-100 μm. The average is calculated from single samples results, directly obtained in each sample measurement by the particle counter software. The number of samples used for each average is indicated in the figure legend. Figure 5A and 5B show that dilution and de-flocculation effects occurred in both groups of sets. A dilution operation causes a reduction in the number of counted particles in all size ranges, which is depicted in Figure 5 by full arrows. A de-flocculation process causes higher counts in the smaller size ranges, while lower numbers are counted in the larger size ranges, depicted in Figure 5 by the dashed arrows. Dilution and de-flocculation effects were observed in all sets measured in this research with the exception of set 29, with sludge collected at MBR B (result not shown), where at the time of collection, an unusual growth of filamentous bacteria was reported (Moreau, 2010). It can be concluded that, through particle counting in the range of 2-100 μm, no difference was detected in the MBR activated sludge structure. However, particle counting in the range 2-100 μm did show that through the applied methodology, i.e. a fast dilution of original samples with permeate, a de-flocculation process occurred in all samples. Furthermore, if our hypothesis is correct and original, non-diluted samples of “High MLSS” sets with good to moderate filtration quality entrapped fouling particles in its structure, as opposed to “Low MLSS” sets, then the aforementioned fouling particles are certainly smaller than 2 μm.

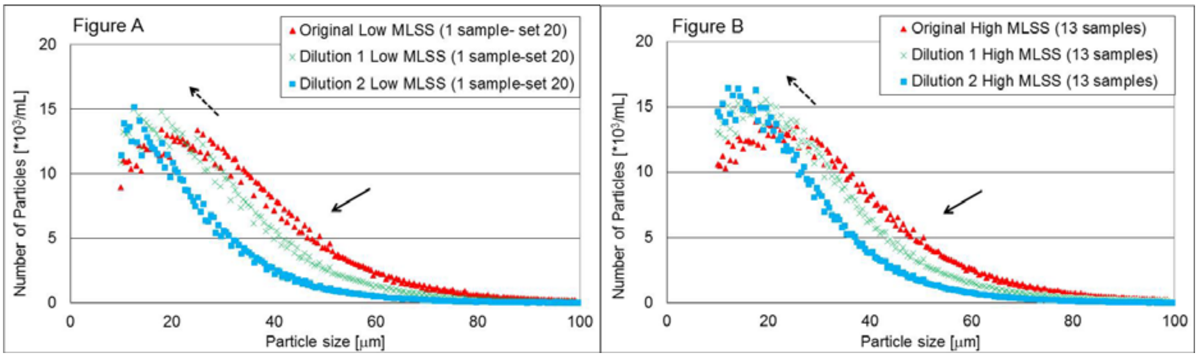

Figure 5 Average number of particles/mL per particle size in "Low MLSS" (A) and "High MLSS" sets (B).

3.4 Particle counting in the range of 0.4-5 µm

Our previous work showed that particles in the range 0.4-0.5 µm were not successfully counted by our equipment, a HIAC ChemShield particle counter (Lousada-Ferreira, 2011). Furthermore, the total number of particles in the range 1-5 µm constitutes 1 % of the total number of particles in the range 0.4-5 µm. Therefore, in this research we assume the reliable and significant range for particle counting in the range of 0.4-5 µm as 0.5 to 1 µm. Moreover, a particle size distribution can be presented as a power law function:

$$\log n(d_P) = \log A - \beta \log(d_P)$$

where d_P represents the particle diameter; $n(d_P)$ the derivative, at any point, of the cumulative number distribution; β and $\log A$ coefficients of the power law function and β the slope of the power law function. In this research, the value of β , i.e. the normalized distribution slope, in the range 0.5 to 1 µm is constant. Table 5 shows the statistical parameters of β obtained in "Low MLSS" and "High MLSS" sets.

Table 5 Statistical parameters of β values for particle counting in the range 0.4-5 µm.

		Low MLSS [1]			High MLSS[2]		
		[#] [3]	β average	β st. deviation	[#] [3]	β average	β st. deviation
Sets 1 to 22: MBR A	Non-diluted	1	13.7	-	9	8.4	2.8
	Dilution 1	1	12.5	-	9	9.1	2.5
	Dilution 2	1	10.1	-	9	12.8	6
Sets 2 to 28: membrane and aeration tanks	Non-diluted	1	9.5	-	2	10.6	1
	Dilution 1	1	9.3	-	2	10.5	0.7
	Dilution 2	1	9.2	-	2	10.7	1.2
Sets 29 to 32: MBR B,C and D	Non-diluted	3	10.5	0.2	-	-	-
	Dilution 1	3	10.5	0.6	-	-	-
	Dilution 2	3	9.3	0.5	-	-	-

Key: [1] sets with MLSS of original sample ≤10.5 g/L; [2] sets with MLSS of original sample >10.5 g/L; [3] Number of results used to calculate the average.

Table 5 shows that in "Low MLSS" sets, β is stable or slightly decreases with dilution, opposite to "High MLSS" sets where β is stable or increases with dilution. Lawler (1997) theoretically defined the possible particle size distributions, showing that β can be constant or variable, within certain limits. Ceronio et al. (2005) concluded that for full size range measurements a variable β is fundamentally more correct but a constant β can be accurate in certain size range fractions of the particle size distribution. In our research, each measurement in the size range 0.4-5 µm has a constant β. However, the hypothetical full-range measurement should have a variable β. Therefore, a decrease in β for the dilution samples actually means that the peak of the hypothetical full-range particle size distribution, constituted by particles with a smaller size than 0.4 µm, is decreasing. By opposition, an increase in β in dilution samples means that the aforementioned peak is increasing. Consequently, in "High MLSS" sets we measure an increase in particles smaller than 0.4 µm with dilution, opposite to "Low MLSS" sets.

Several authors stressed the importance of sub-micron particles in membrane filtration, as follows. Ivanovic *et al.* (2008) concluded that the relevant particle size range for membrane filtration is below 0.1 µm. Geilvoet (2010) stresses the importance of particles smaller than 0.5 µm to determine the volume of the cake layer. Considering that membrane filtration is a size exclusion separation technique, it seems logical that the most relevant particle sizes, determining filtration performance, are close to the membrane pore size. Our research shows that de-flocculation of particles smaller than 0.4 µm occurs in “High MLSS” sets, opposite to “Low MLSS” sets, supporting our initial hypothesis.

4. CONCLUSIONS

In our present work we validated our hypothesis that due to structural differences, activated sludge, with moderate to good filterability and MLSS concentrations exceeding an apparent critical value, would retain fouling particles in its matrix, as opposed to activated sludge with MLSS concentrations below that apparent critical value. The conclusions obtained in this research, can be summarized as follows:

- Filterability of sludge with moderate to good filtration quality and MLSS concentration above 10.5 g/L worsens when diluted, as opposed to filterability of sludge with MLSS concentration below 10.5 g/L.
- For the investigated sludge samples, the apparent critical MLSS concentration was 10.5 g/L. The value of 10,5 g/L should not be extrapolated without further research.
- The specific resistance times the particle concentration of a cake layer obtained when filtrating sludge of moderate to good filterability and MLSS concentration above the critical value, is 5.5 times smaller compared to the cake layer of sludge with MLSS concentration below the critical value.
- Due to the methodology applied, i.e. a fast production of dilution samples by adding permeate, a reduction of the sludge mass and a de-flocculation process took place both in samples with MLSS concentration above and below the apparent critical value. The latter conclusion is supported by SMP and particle counting results, in the range 2-100 µm. However, sludge with MLSS concentration above the apparent critical value and moderate to good filtration quality when diluted, also releases particles with sizes below 0.4 µm, opposite to samples with MLSS concentrations below the critical value. Activated sludge, with moderate to good filterability and MLSS concentration above the apparent critical value is likely to retain particles, smaller than 0.4 µm, in its sludge mass.

Our results indicate that to promote good filterable sludge quality in MBR systems to avoid fouling, we should rely on optimal MLSS concentration ranges.

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