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EUROMBRA

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1 Introduction

Since its early applications the MBR technology has been considered as an extremely long-SRT process, due to the membrane-based solid/liquid separation which results in a mutual independence of sludge age and HRT. Early studies report steadily good performances in terms of COD and ammonia removal even under complete solids retention. Houten and Eikelboom (1997) compared the specific sludge production in a pilot-scale side-stream MBR in a variable range of F/M ratio (0.029÷0.15 kgCOD $kgMLVSS^{-1}$ d^{-1}); with a MLSS concentration of ~ 50 kgMLSS m^{-3}, the authors report an observed ranging between 0 and 0.19 kgMLVSS $kgCOD_{rem}^{-1}$. Similar results have been earlier published by Müller *et al.* (1995) in a bench-scale MBR operated with no sludge wasting (F/M ratio ~ 0.02 kgCOD $kgMLVSS^{-1}$ d^{-1}) and at a MLSS concentration of 40÷50 kgMLSS m^{-3}. More recently Bhatta *et al.* (2004) reported the results of an experimental study carried out at a sludge age ranging between 90 and 500 days, with a gradual decrease of MLVSS in the biological compartment and an observed yield coefficient from 0.16 kgMLVSS $kgBOD_{5r\,em}^{-1}$ down to 0.07 kgMLVSS $kgBOD_{5r\,em}^{-1}$.

Under the above mentioned condition, the MLVSS/MLSS ratio has been generally reported to be in the range 65÷70%, thus leading to consider the (low) amount of surplus sludge to be already stabilized and not needing further digestion process to remove the volatile fraction. However, most of the cited studies have been carried out at either bench- or pilot-scale. Indeed, currently two main categories of MBR are reported to be in operation at full-scale all around the world: centralized MBR, generally used for upgrading or retrofitting conventional activated sludge plants and decentralized MBRs which are pretty common in new installations where the choice of technology is driven by lack of space, need of compact solutions and quick startup operation. In terms of sludge age, such categorization reflects in two completely different processes: while centralized MBR are more and more moving towards conventional values of SRT (lower than 30 days for nitrification/denitrification), the second kind of membrane bioreactors is frequently operated in a very wide range of sludge age, which is also determined by seasonal fluctuations in the influent loading (tourist areas). In this case, the actual need for further stabilization of the excess sludge should be investigated, also by taking into account the effect of the additional digestion SRT on the physical properties of the sludge for post-processing.

The report is a collection of contributions coming from partners UNITN and UM2 regarding the impact of some operational parameters (namely F/M ratio and sludge age) on the respiration rate measured or calculated by process modelling. Particularly, the experimental activity reported by UNITN relates to the aerobic digestion of surplus sludge produced at a 20÷25 days SRT large pilot-scale MBR, with a special attention to the impact of two different sludge ages in the aerobic digestion on the biological and physical characteristics of the sludge. The UMII contribution is focused on the impact in the oxygen uptake rate by different operational conditions (SRT and F/M ratio).

2 Experimental activity

2.1 *UNITN*

Surplus sludge collected from a large pilot-scale MBR operated at a sludge age of ~20÷25 days has been fed to two bench scale aerobic digesters, with an additional SRT of 10 days and 50 days respectively. Both biological and physical properties of the sludge have been routinely monitored in order to asses the impact of the SRT on VSS removal, biomass activity, sludge dewaterability and filterability.

2.1.1 Materials and methods

A large pilot-scale MBR has been operated since Oct. 2006. The plant is located at the WWTP of Lavis (Trento, WWTP of Lavis (Trento, Italy) and is fed with pre-screened (2 mm) municipal wastewater pumped after the grit pumped after the grit chamber of the full scale installations. The flow-diagram and a picture of the setup are of the setup are shown in

Figure 1.

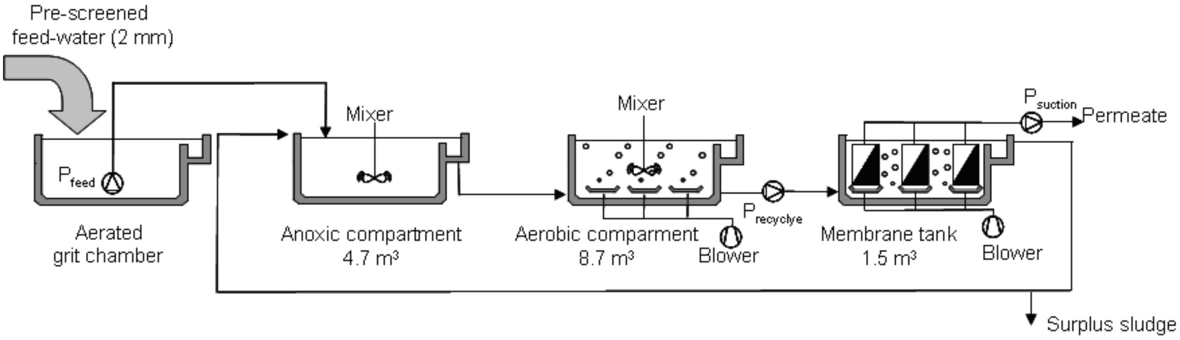

Figure 1: Large pilot scale MBR

The overall process volume is 14.9 m^3, 10% of which being due to the membrane compartment in which three membrane modules (GE Zenon ZW500d) are immersed. The membrane material is a patented hydrophilicized PVDF, with a nominal pore size of 0.04 µm and an overall membrane surface area of ~100 m^2. Both hydraulics- and biology-related parameters are monitored on-line including permeate flux, TMP, MLSS (in biotank and membrane chamber), dissolved oxygen and effluent ammonia and nitrate. After the transient phase at the plant start up, the system has been usually operated at a 20-25 days SRT (Solids Retention Time) which resulted in a pseudo steady state MLSS concentration of 6.8 ± 0.9 kg m^{-3} and; being the recycle ratio in the range 3-4, the MLSS concentration in the membrane compartment was correspondingly between 8.3 ± 1.0 kg m^{-3}. Except for some specific experiments for the critical flux assessment (data not shown) the permeate flux has been tuned in the sub critical region, i.e. at values ranging between 10 and 20 L m^{-2} h^{-1}. The influent wastewater characteristics have been monitored twice a week, by collecting 24 hours samples analysed according to the APHA Standard Methods; the values for the main macropollutants for the considered experimental period are listed in Table 1.

Table 1: Feedwater composition in the considered experimental period

Parameter	Unit	Value
COD	g m^{-3}	636.9 ± 422.3
Soluble COD	g m^{-3}	172.4 ± 160.2
TKN	g m^{-3}	50.9 ± 18.1
$N\text{-}NH_4^+$	g m^{-3}	32.2 ± 13.9
Total P	g m^{-3}	7.1 ± 2.2
TSS	g m^{-3}	323.1 ± 102.6
VSS/TSS	%	82.7 ± 5.5

Excess sludge has been fed to two bench-scale aerobic digesters (50 L each; Figure 2-a) which have been operated at 10 days and 50 days by means of daily sludge wasting; the experimental apparatus was installed indoor and the sludge temperature in the two stabilization tank ranged between 14 and 21°C over the whole duration of the whole experimentation. A blower supplied the digesters with air continuously in order to keep a dissolved oxygen concentration higher than 3 g m^{-3}. Sludge was sampled from both compartments periodically (2-3 times a week) and was characterized in terms of maximum OUR after a spike addition of sodium acetate, biomass viability, capillary suction time, specific resistance to filtration and COD content in the liquid phase. Respirometric tests were carried out in a flowing-gas static-liquid jacketed respirometer with online DO-data acquisition and OUR calculation. Sodium acetate was added to the sludge in order to obtain a S_0/X_0 ratio ranging between 0.08 and 0.1 gCOD $gCOD^{-1}$; moreover, in order to inhibit the nitrifiers activity, ATU was added before each test.

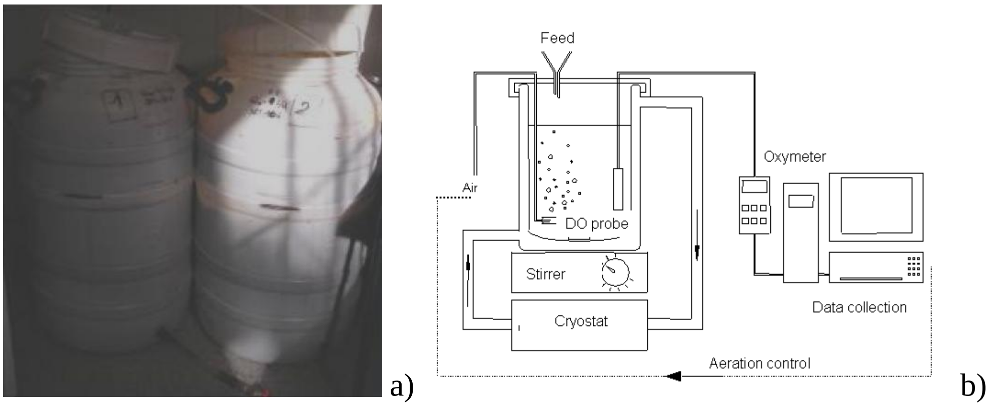

Figure 2: Lab-scale digestors and respirometer used for biomass characterization

The MLSS and MLVSS analysis was carried out by gravimetric methods according to the APHA Standard Methods. Batch filtration tests were performed in a Sartorius cell to determine the sludge filterability from both sites. Filterability was expressed in terms of (αC) in the V *vs.* t/V plot, by means of the Carman-Kozeny equation:

$$\frac{t}{V}=\frac{\mu\cdot\alpha C\cdot V}{2A^2\cdot P}\cdot V+\frac{\mu\cdot R_m}{A\cdot P}$$

Where:
t (s) is the time variable,
V is the permeate volume (m^3),
μ is the dynamic viscosity of permeate (Pa·s),
α (m^{-1} kg^{-1}) is the specific resistance to filtration,
C (kg m^{-3}) is accumulated matter per unit of permeate volume,
A is the membrane surface area (m^2),
P is the operational pressure (Pa),
R_m is the intrinsic membrane (polysulphone) resistance (m^{-1}).

Sludge dewaterability was quantified as capillary suction time CST (s) according to the APHA Standard Method 2710G with a portable apparatus (Triton 304B; chromatography paper Whatman no. 17).

2.1.2 Results

The trend of MLVSS concentration in both digesters is shown in Figure 3: as reported in the graph, the longer SRT slightly impacted the VSS reduction which increased from 30% to 35% (measured at the end of the test). The difference in VSS concentration occurred since the beginning of the trial and reached an almost steady value after three weeks for both sludges.

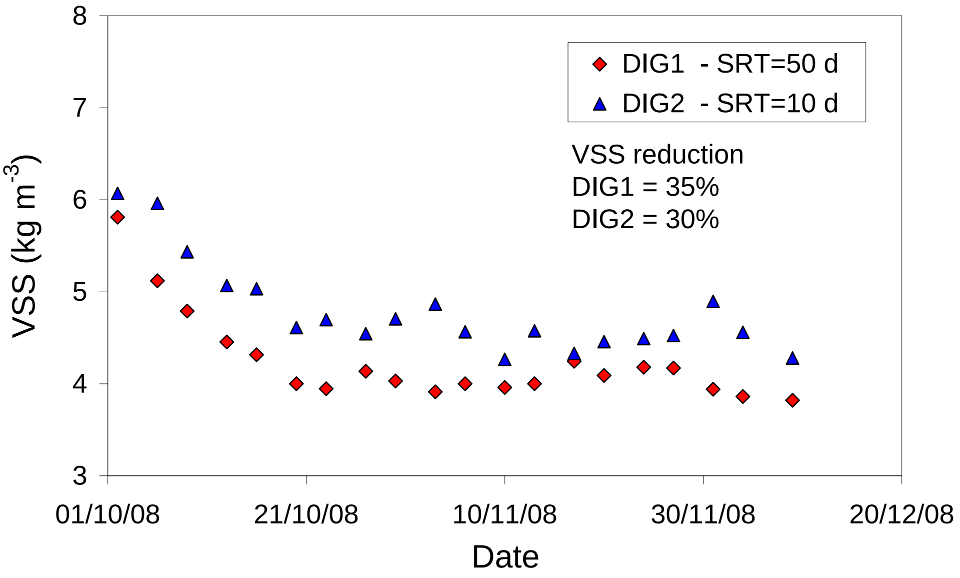

Figure 3: Trend of MLVSS in digester 1 and 2

At the same time, the maximum specific OUR decreased rapidly at a value of around 4 mgO_2 $gVSS^{-1}$ h^{-1} and 2 mgO_2 $gVSS^{-1}$ h^{-1} for the 50 days and 10 days SRT respectively (Figure 4). More in detail, at the longer sludge age the respiration rate of heterotrophs dropped down to almost 0 mgO_2 $gVSS^{-1}$ h^{-1} thus confirming the indications provided by the VSS trend.

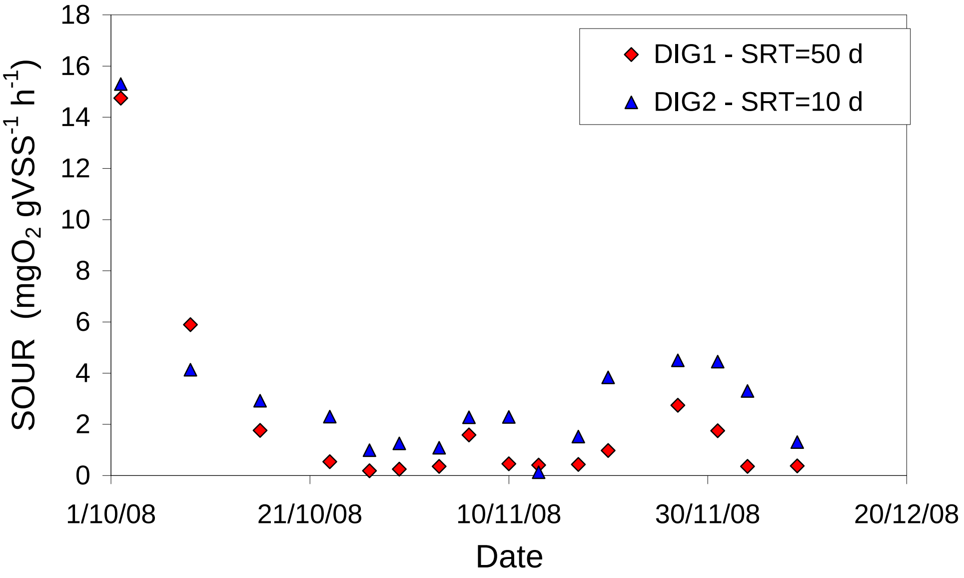

Figure 4: Specific oxygen uptake rate for heterotrophic organisms after addition of sodium acetate

The organic content of the liquid phase of the sludge was also investigated, by measuring the filtered COD (Figure 5).

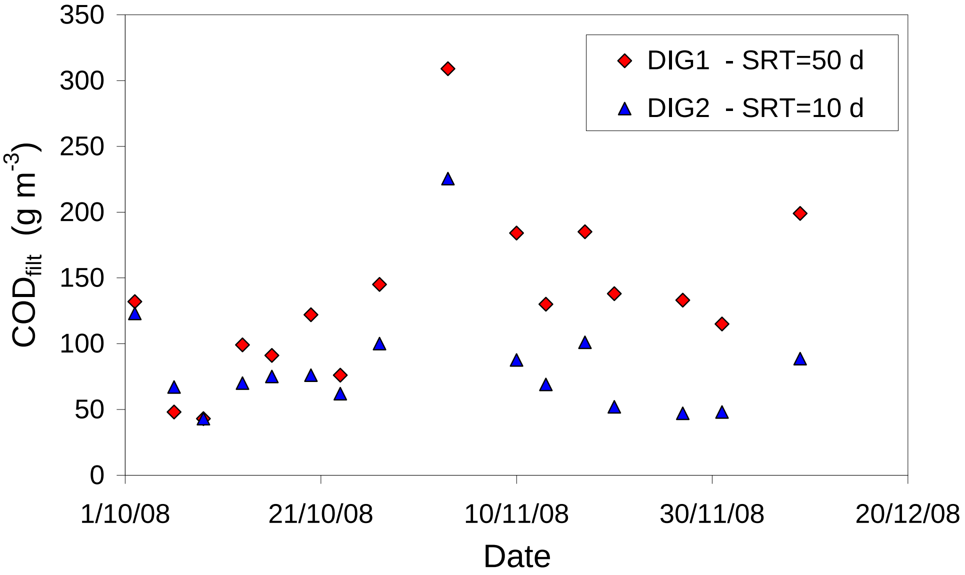

Figure 5: Filtered COD in sludge sampèles collected form the two digesters

As shown in the graph, the higher sludge age resulted in a generally higher concentration of organics in the liquid phase which are responsible for the worse sludge filterability and dewaterability, as depicted in Figure 6 and Figure 7.

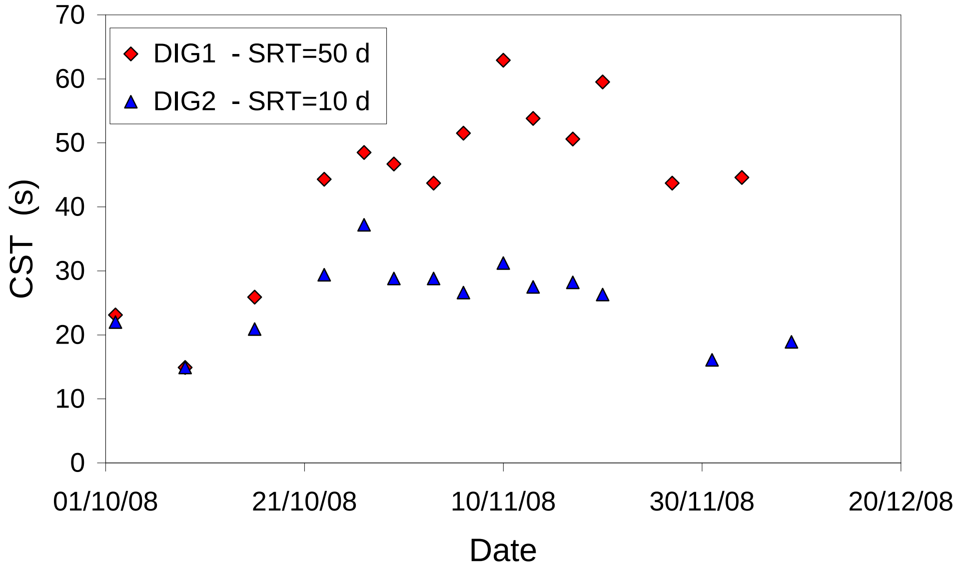

Figure 6: Capillary Suction Time on sludge samples collected form the aerobic digestion tanks

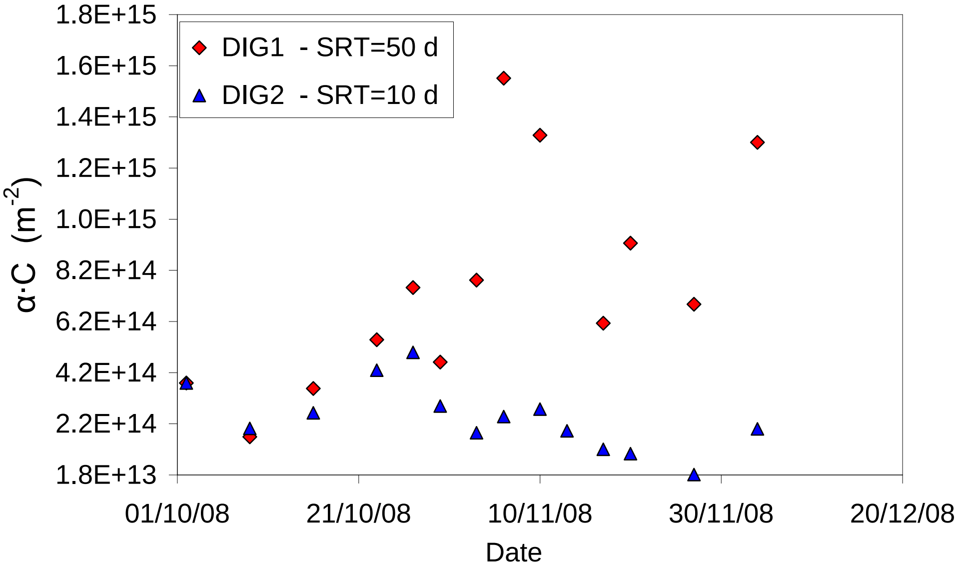

Figure 7: Trend of $(\alpha{\cdot}C)_{sludge}$ for both digesters

2.1.3 Conclusions

Two values of additional sludge age for aerobic digestion were tested in a bench scale on waste sludge collected from a large pilot-scale MBR for municipal wastewater treatment. The impact of different SRT was investigated in terms of volatile solids reduction and sludge properties for post-processing. Although a slight further reduction of VSS was observed at

SRT=50 days, the negative impact of longer sludge age due to the higher release of soluble microbial product suggested that a very short additional SRT should be adopted in aerobic digestion of surplus sludge produced at “centralized MBR”, in order not to affect the post-processing steps of sludge handling.

2.2 UM II

2.2.1 Materials and methods

Experiments were developed in an membrane bioreactor with a total volume of 50 L. The reactor was ring-shaped, similar to an oxidation channel. An impeller was introduced to provide a horizontal circular flow of the suspension, preventing sedimentation of particles and favoring the homogenization of the media. The membrane bioreactor was inoculated with a mixed culture taken from a wastewater treatment plant. Aeration, conducted by air injection supplied the necessary oxygen for biological activity, and created turbulence around the submerged membrane modules.

The reactor, operated under aerobic conditions, steadily fed with a soluble and easily biodegradable synthetic organic substrate. Substrate feed and permeate extraction were respectively carried out by the peristaltic pumps (ISMATEC VC-MS/CA) and (WATSON MARLOW 505S), at constant rate. The experiments were conducted under non-limiting oxygen condition, the minimal average concentration of oxygen dissolved in the reactor being 2 mgO_2/l.

Oxygen Uptake Rate (OUR) measurements were performed by sampling 600 mL of sludge to follow the evolution of the respiration rate and to estimate the oxygen needs. The experimental set-up used at UM2 for OUR consists in a 600 mL vessel closed on the top, equipped with a dissolved oxygen probe (WTW OXY 340) and a stirrer; data acquisition is performed via Hyperterminal every 30 seconds.

2.2.2 Modelling activities

2.2.2.1 ASM 1

Oxygen demand was linked to growth according to the ratio $Y_H/(1-Y_H)$. As in the stabilized phase, the rate of growth was equal to the rate of death, the Oxygen Uptake Rate is given as (Heran et al. 2008) :

$$rO_2 = OUR = \frac{1-Y_H}{Y_H} \times \left(b_H + \frac{1}{SRT}\right) \times Xb = (1-Y_H) \times \frac{OLR}{1 - \frac{Y_H (1-fp) b_H}{b_H + \frac{1}{SRT}}} \quad [\,gCOD.m^{-3}.d^{-1}\,] \qquad \text{Eq (1)}$$

Thus the Oxygen demand is proportional to organic volumetric load.

However, it is possible to split this OUR into two part: (i) one due to the exogenous substrates, and (ii) another part linked to the endogenous respiration:

$$OUR = OUR_{SubstrateConversion} + OUR_{EndoneousProcesses}$$

$$OUR_{SC} = (1-Y_H) \times OLR \quad [\,gCOD.m^{-3}.d^{-1}\,] \qquad \text{Eq (2)}$$

$$OUR_{EP} = (1-Y_H)(1-fp) b_H \; X_B = \frac{(1-Y_H) OLR}{\frac{b_H + \frac{1}{SRT}}{Y_H (1-fp) b_H} - 1} \quad [\,gCOD.m^{-3}.d^{-1}\,] \qquad \text{Eq (3)}$$

2.2.2.2 ASM 3

The same mathematical development was done with the ASM3 equation (Lobos 2008).
The results are :

$$OUR_{SC} = \frac{Q}{V} \cdot (1-Y_{STO}) \cdot (S_{S0} + X_{S0}) + \frac{(1-Y_H)}{Y_H} \cdot \left(b_H + \frac{1}{SRT}\right) \cdot X_{BH} \qquad \text{Eq (4)}$$

$$OUR_{ER} = (1-f_{XI}) \cdot b_H \cdot X_{BH} + b_{STO} \cdot X_{BH} \frac{\left(b_H + \frac{1}{SRT}\right) \cdot K_{STO}}{\mu_m - \left(b_H + \frac{1}{SRT}\right)} \qquad \text{Eq (5)}$$

With the active biomass equation:

$$X_{BH} = \frac{Y_{STO} \; Y_H \; OLR}{\left(b_H + \frac{1}{SRT}\right) \cdot \left(1 + \frac{Y_H \; b_{STO} \cdot K_{STO} \; SRT}{(\mu_m - b_H) SRT - 1}\right)} \qquad \text{Eq (6)}$$

2.2.3 Results

2.2.3.1 Case-study No. 1

No biomass extraction was carried out, apart from sampling. The long-term treatment performance of the system was investigated for three volumetric organic loads: 0.41, 0.82 and 0.93 $kg_{COD}.m^{-3}.d^{-1}$, in order to study the biological behavior (physiological state of the biomass, growth kinetics, anabolic transformations) under conditions of high sludge age and total biomass retention. The overall operating conditions are given in Table 2.

Table 2: Operating conditions

Hydraulic conditions		
Reactor volume	50 L.	
Hydraulic flow rate	24 $L.d^{-1}$	
Hydraulic retention time	2.08 d	
Sludge retention Time	No extraction	
Influent conditions Acetate concentration (Ss) and Volumetric loading (Cv)		
Run I :	Ss = 900 $gCOD.m^{-3}$	
Run II :	Ss = 1700 $gCOD.m^{-3}$	
Run III :	Ss = 2000 $gCOD.m^{-3}$	
Run I :	OLR = 0.41 $kgCOD.m^{-3}.d^{-1}$	OUR = 0.19 $kg_{O2}. m^{-3}.d^{-1}$
Run II :	OLR = 0.82 $kgCOD.m^{-3}.d^{-1}$	OUR = 0.50 $kg_{O2}.m^{-3}.d^{-1}$
Run III :	OLR = 0.93 $kgCOD.m^{-3}.d^{-1}$	OUR = 0.53 $kg_{O2}.m^{-3}.d^{-1}$

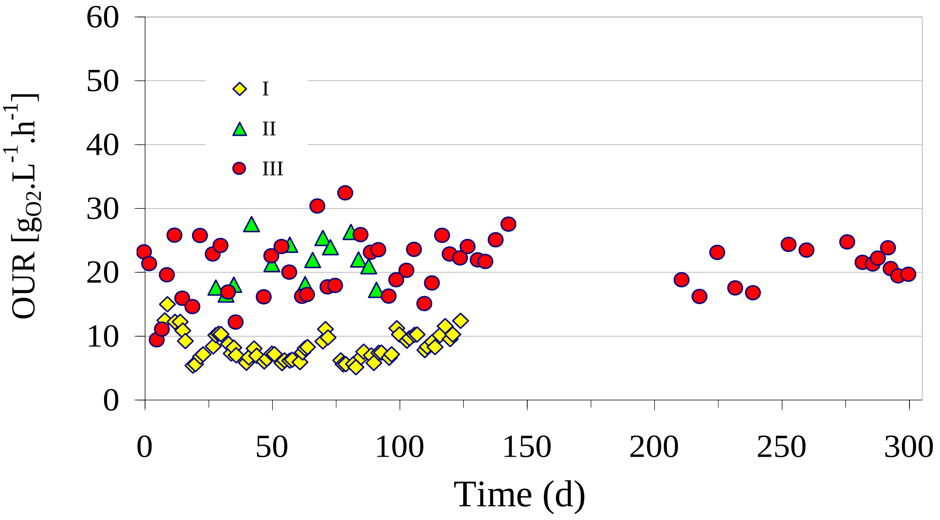

Figure 8: Oxygen uptake rate evolutions during the three running periods

Oxygen uptake remained relatively constant during each experimental period despite a steady increase in VSS concentration. Therefore the increase in VSS did not correspond to an increase in oxygen demands. Consequently, the quantity of bacteria that was active, or that had respiratory activity, was supposed unchanged in the reactor for the duration of the series despite the net increase in VSS. It appeared that VSS could not be associated to active biomass, which was confirmed by microscopic observation where a lot of cell debris was observed. Independently of VSS concentration, the oxygen uptake rate measured seemed proportional to volumetric load. These observations went against the equation proposed by Eckenfelder where the amount of oxygen uptake for maintenance (endogenous respiration) is proportional to the concentration in VSS. Nevertheless, its confirms the ASM equation, where the steady state OUR only depends on organic load.

2.2.3.2 Case-study No.2

The substrate was representative of a complex effluent (acetate + extract of meat). For the extract of meat a commercial product (Viandox) was used. Nitrate of ammonium ((NH4)NO3) and Di-ammonium hydrogen phosphate ((NH4)2HPO4) were added to maintain an acceptable COD/N/P ratio and so, to avoid any nutrient limitation. The pH was adjusted using NaHCO3 at 0.5 g/L for the complex feed. The average feed concentrations are given in Table 3.

Table 3: Mean concentrations of the feed solutions

	COD_{Total} (mgCOD/L)	$COD_{Soluble}$ (mgCOD/L)		$COD_{Particular}$ (mgCOD/L)	*MES* (mg/L)	*MVS* (mg/L)
		acetate	viandox	viandox		
Influent	1830	1500	239	91	93	56

Table 4: Operating conditions

Hydraulic conditions	
Reactor volume	50 L.
Hydraulic flow rate	24 $L.d^{-1}$
Hydraulic retention time	2.08 d
Sludge retention Time	No extraction
Influent conditions Acetate concentration and Viandox	
Run I : Ss = 1830 $gCOD.m^{-3}$	
Run I : OLR= 0.88 $kgCOD.m^{-3}.d^{-1}$	OUR = 0.504 $kg_{O2}.\ m^{-3}.d^{-1}$

Figure 9 shows the VSS, SS, ISS (Inorganic Suspended solids) and OUR evolution. Cycles of growth and pseudo-stabilisation in the MLVSS concentration were observed. Nevertheless a constant increase of TSS is observed.

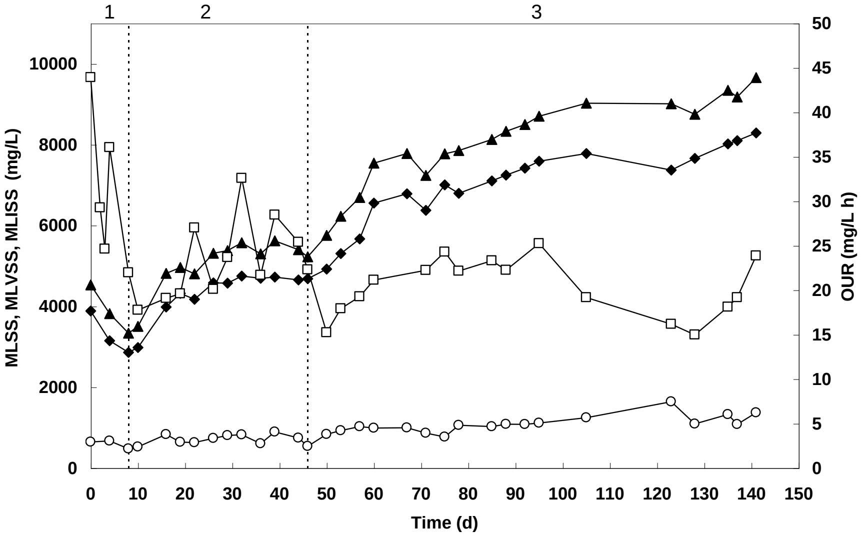

Figure 9: TSS (▲), VSS (♦), ISS (○) and OUR (□) evolution during the MBR operation with the acetate-Viandox® substrate

Even if an increase in the MLVSS concentration was observed, the MLVSS/MLSS ratio remained constant during the whole experimental period. However, a decrease of the COD_P/MLVSS ratio was observed, from an initial value of **1.47 COD_P/MLVSS** to a final value of **1.32 COD$_P$/MLVSS**. The decrease of this ratio can be attributed to a large microbial change in the sludge composition. The oxygen uptake rate (OUR) was quantified during the whole experimental period in order to estimate the viability of the bacteria thought the respiratory activity. It was first observed a decrease phase, possibly due to the biomass adaptation, and then a stabilisation by the 60-operation day at **21 mg$_{O2}$/L·h** (0.504 kg$_{O2}$. m^{-3}.d^{-1}). This result could signify that the part of respiring cells remained constant during the whole experiment.

2.2.3.3 Case-study No.3

The substrate was representative of a acetate and salt in order to respect the COD/N/P ratio of 100/5/1. The operand parameters are summarized Table 5.

Table 5: Operating conditions

Parameters	Units	C I	C II		C III	C IV
Period	days	1 24	50 - 81	106 - 127	149 – 227	278 - 361
Organic Load ORL	kgCOD.m^{-3}.d^{-1}	1,5	1,5	6,0	1,5	1,5
HRT	days	No extraction	40	No extraction	No extraction	No extraction
Jw	LMH	10,0	10	10	20	20
TSH	hours	13,6	11,4	11,4	5,7	5,7
Influent substrate	gCOD.m^{-3}	840	700	700	300	300

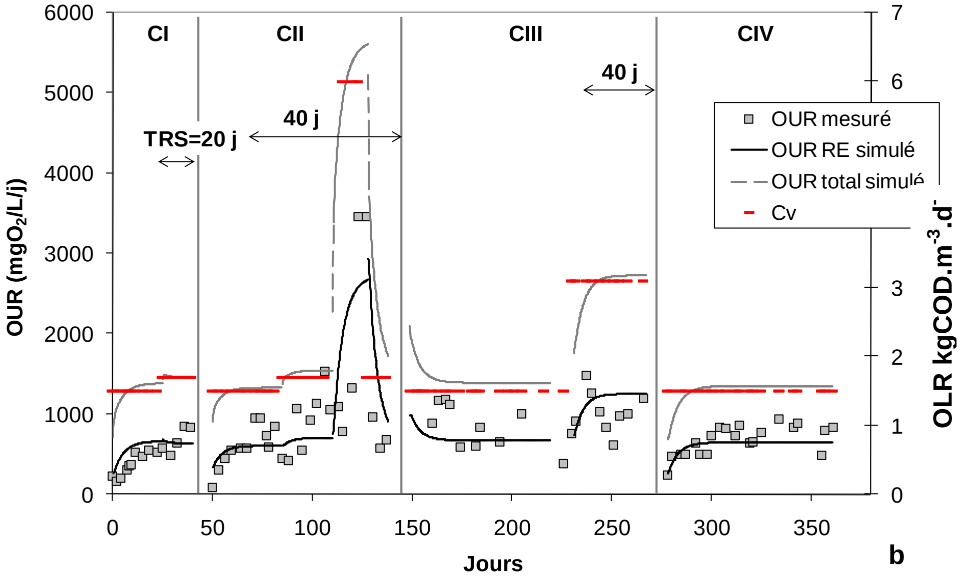

Figure 10: OUR (□) evolution and ▬ Organic loading rate during

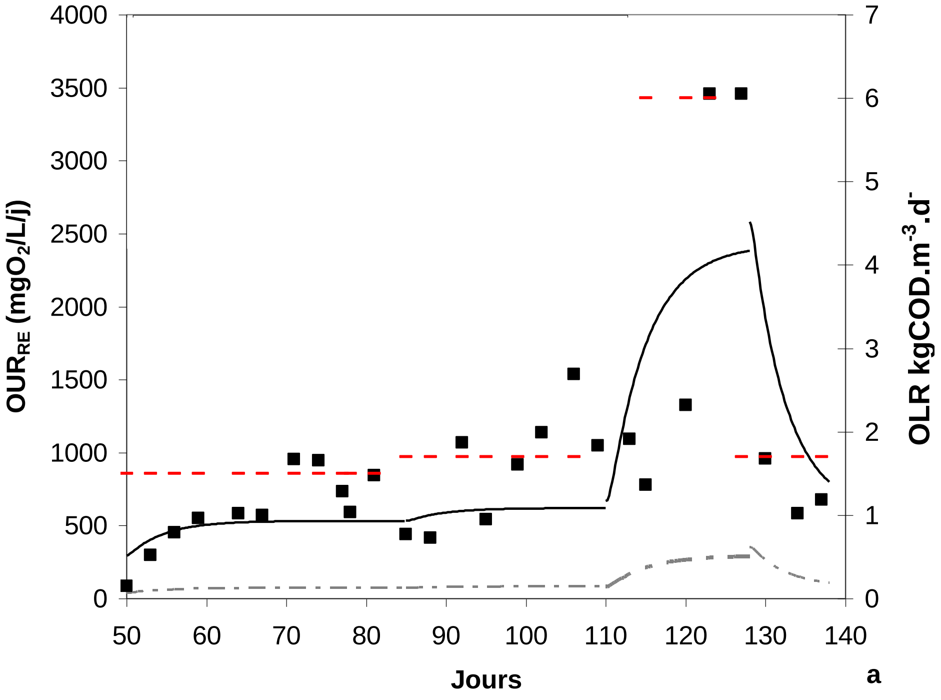

Figure 11: OUR (■) evolution and ▬ Organic loading rate during the MBR operation with the acetate substrate

Table 6: Operating conditions

OLR= 1.5 kgCOD.$m^{-3}.d^{-1}$	OUR = 0.656 kg_{O2}. $m^{-3}.d^{-1}$
OLR= 3 kgCOD.$m^{-3}.d^{-1}$	OUR = 1.143 kg_{O2}. $m^{-3}.d^{-1}$
OLR= 6 kgCOD.$m^{-3}.d^{-1}$	OUR = 3.470 kg_{O2}. $m^{-3}.d^{-1}$

Figure 10 shows OUR evolution during the overall operation. The Figure 11 is a zoom of the campaign number two where the OLR was increased until 6 kgCOD $m^{-3}d^{-1}$.

2.2.3.4 Case-study No.4

A biomass extraction of 1 L.d^{-1} was carried out with an average sludge age of 50 day. However, as the pilot volume is only 50 Liter, it is difficult to conclude that the pilot has worked with this constant 50 day of sludge age. The volumetric organic loads was increased each long-term treatment performance of the system was investigated for three volumetric organic loads: 0.41, 0.82 and 0.93 $kg_{COD}.m^{-3}.d^{-1}$, in order to study the biological behavior (physiological state of the biomass, growth kinetics, anabolic transformations) under conditions of high sludge age and total biomass retention. The overall operating conditions are given in Table 7.

Table 7: Design and operational conditions

Hydraulic conditions	
Reactor volume	50 L.
Hydraulic flow rate	104 $L.d^{-1}$
Hydraulic retention time	11h20
Sludge retention Time	50 d.
Influent conditions Acetate concentration (Ss) and Volumetric loading (Cv)	
Run I (20 days) :	Ss = 710 $gCOD.m^{-3}$
Run II (30 days) :	Ss = 1180 $gCOD.m^{-3}$
Run III (10 days) :	Ss = 1890 $gCOD.m^{-3}$
Run I : Cv = 1.5 $kgCOD.m^{-3}.d^{-1}$	OUR = 0.60 $kg_{O2}. m^{-3}.d^{-1}$
Run II : Cv = 2.5 $kgCOD. m^{-3}.d^{-1}$	OUR = 0.95 $kg_{O2}.m^{-3}.d^{-1}$
Run III : Cv = 4 $kgCOD. m^{-3}.d^{-1}$	OUR = 1.47 $kg_{O2}.m^{-3}.d^{-1}$

2.2.3.5 *Data compilation*

The Table 8 gives the values of the default values which are used in the OUR prediction.

Table 8: Default values used for OUR prediction

Default values			
ASM 1		ASM 3	
mu	6	b_H	0.2
fp	0.08	b_{STO}	0.2
Yh	0.67	K_{STO}	1
bh	0.62	Y_{STO}	0.85
		Y_H	0.63
		u_m	2
		f_I	0.2

The default value (Table 8) for ASM 1 and 3 allows with the help of equation (3 and 5) the OUR calculation (Fig.13) which are listed in Table 9. It is very difficult to find a relation between OUR and SRT. Indeed, during case 3, different sludge ages were tested (Figure 10) but no significant variation were found. Except the last point (High OLR), a linear relation could be draw (Figure 12).

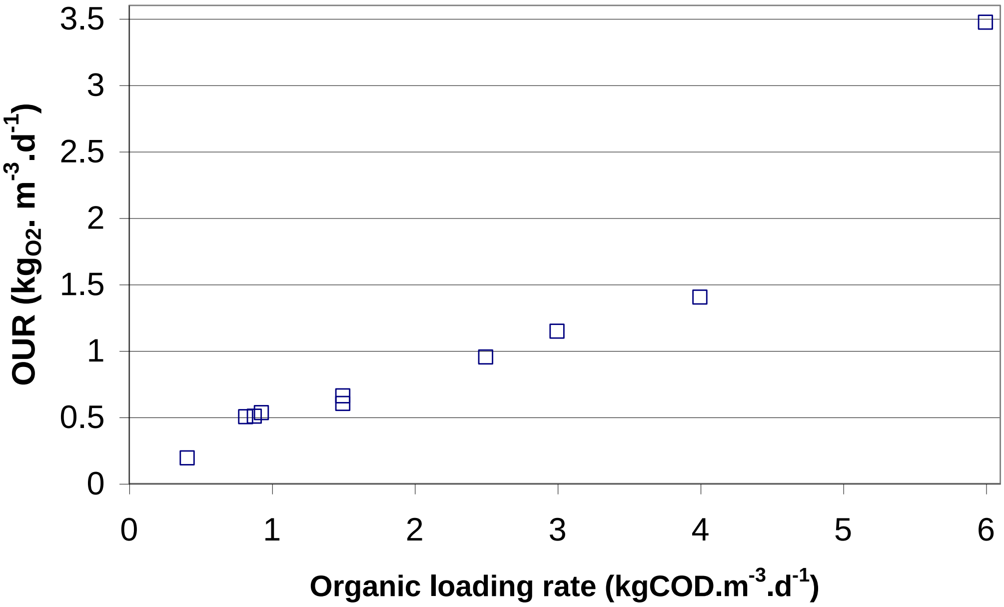

Figure 12: Influence of OLR on OUR value

Table 9: Influence of OLR on OUR value

OLR kgCOD.m^{-3}.d^{-1}	0.41	0.82	0.88	0.93	1.5	1.5	2.5	3	4	6
OUR kg$_{O2}$.m^{-3}.d^{-1}	0.190	0.500	0.504	0.530	0.600	0.656	0.950	1.143	1.400	3.469
HRT (2)	48h	11.4		11.4	11.4		5.7	11.4		11.4
SRT (d)	No extraction	50		No extrac.	50		40	50		40
OUR ASM1	12.1%	15.4%	9.9%	9.5%	22.3%	17.9%	28.7%	25.9%	39.7%	17.0%
OUR ASM1b	17.3%	37.1%	33.1%	32.7%	-7.9%	-12.4%	-3.1%	-4.6%	5.2%	37.1%
X_B ASM3	1001	2002	2148	2270	3410	3661	5683	6667	9093	13334
OUR ASM3	-3.7%	26.8%	22.1%	21.7%	5.0%	2.0%	10.5%	8.1%	20.0%	28.8%

In order to adjust the theoretical values (for ASM 1) corresponding to the measured parameters, default values Y_H (a heterotrophic growth yield) was replaced by the specific conversion yield dedied to acetate $Y_H = 0.45$.

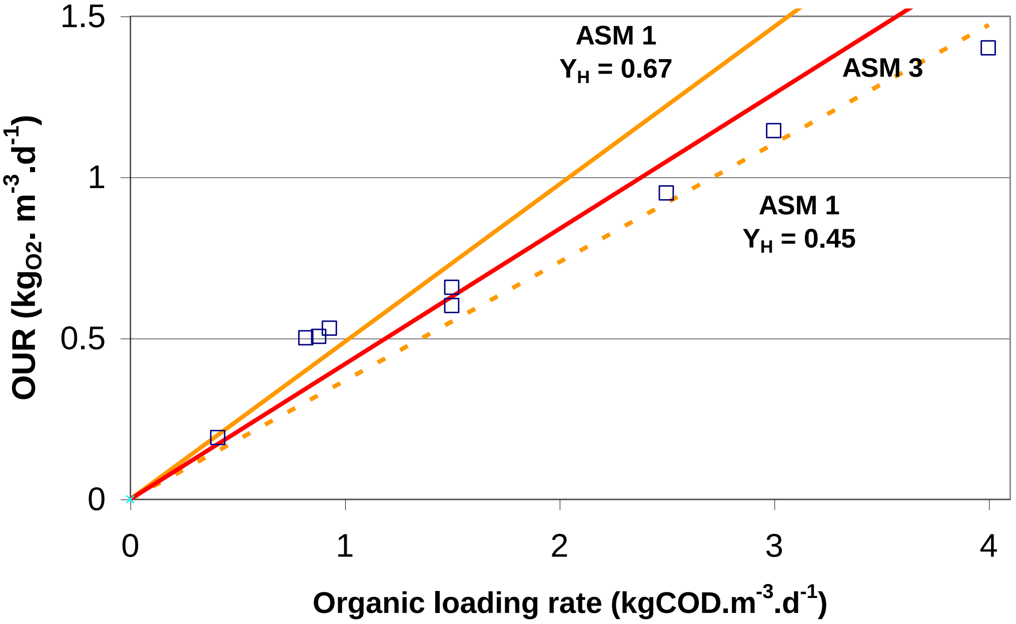

Figure 13: OUR prediction according to ASM 1 and 3

2.2.4 *Conclusions*

This work was centred on monitoring biological activity through oxygen demands of a submerged membrane bioreactor which was fed with a constant easily biodegradable organic substrate under controlled sludge age. At a constant organics load corresponds a constant active biomass concentration. In these operating conditions, the active biomass could not be assimilated to VSS, which was largely composed of bacterial or cellular residues with no metabolic activity. The use of the ASM model, after adjusting the conversion rate of the acetate substrate used in this study, provided a good simulation of the behaviour of the media.

Moreover, several points could be draw:

- A linear Volatile Suspended Solids concentration increase was observed whereas a stabilisation of respiratory activity was achieved.
- Thus, the active biomass could not be assimilated to VSS concentration that was largely composed of dead bacterial or cellular residues with no metabolic activity.
- ASM was adapted for OUR prediction if the OLR is in the usual range (from 1 to 3 kgCOD.m^{-3}.d^{-1}), nevertheless, ASM 3 presents better results.

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