

# Final MBR-Network Workshop

**“Salient outcomes of the European R&D  
projects on MBR technology”**

## Presentation handouts

**31 March – 1 April, Berlin 2009 (Germany)**



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# 12. AERATION AND FOULING DYNAMICS IN HF SMBR: DETERMINING CRITERIA AND PHENOMENA

*J. Lebègue, M. Héran, A. Grasmick*

# Aeration and fouling dynamics in HF SMBR : Determining criteria and phenomena

Julie Lebegue, [Marc Heran](#) and Alain Grasmick

- 1- FOULING CLASSIFICATION IN TERM OF INTENSITY AND REVERSIBILITY vs AERATION
- 2- MODELLING AND OPTIMISATION OF FUNCTIONING CONDITIONS

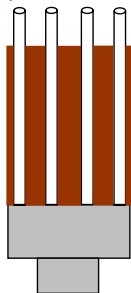


**EUROMBRA**

## CLASSIFICATION OF FOULING IN TERM INTENSITY AND REVERSIBILITY

- Scale of fouling observation and associated methodologies

Macro scale



- (i) Noticeable accumulation of sludge in the bundle  
Packing density/Turbulence  
Filtration conditions and sludge properties

→ Short term experiments :  
Screening effect by the fibre network  
Specific Residence Time Distribution,  
TMP evolution analysis and modelling

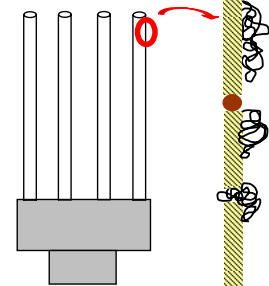
- Long term experiments :  
Analysis of fouling  
Biological activity influence  
and modelling

Micro scale



- (ii) "biofilm" development

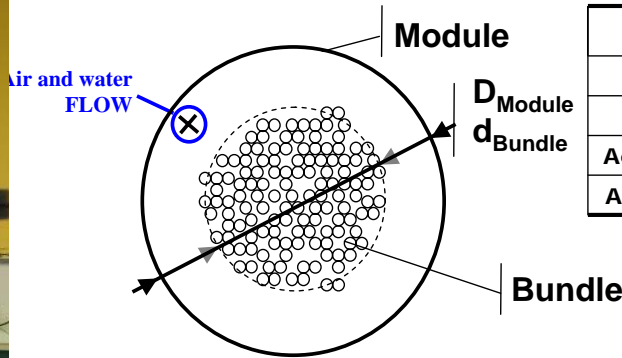
Nano scale



- (iii) irreversible sorption of soluble molecules inside the pores

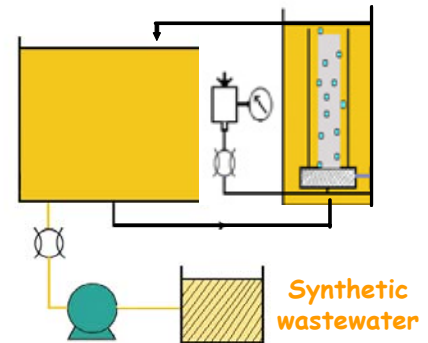
## I - Experimental set-up

- Membrane configuration : Side-stream Hollow Fibre bundle



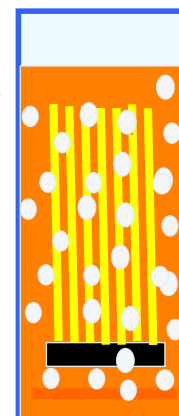
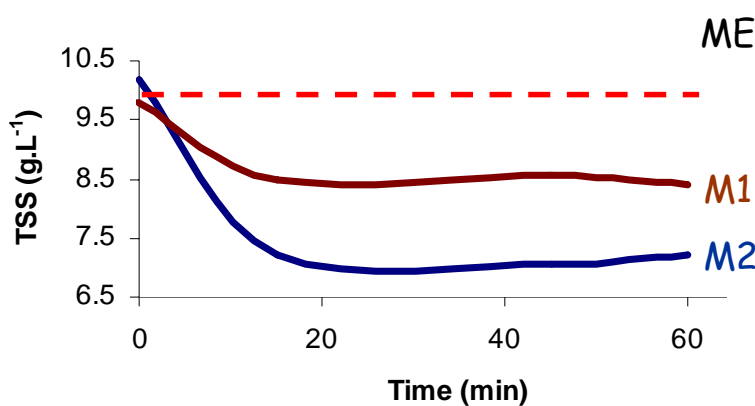
Aeration characteristics		
Aeration $L \cdot h^{-1}$	100	300
$SAD_m$ ( $m^3 \cdot m^2 \cdot h^{-1}$ )	0.45	1.36
Aeration rate $W \cdot m^{-2}$ membrane	0.55	1.66
Aeration rate $Wh \cdot m^{-3}$ permeate	18.5	160

Membrane characteristics : Puron®	
Module diameter (mm)	50
Fiber diameter	2.6
Membrane area ( $m^2$ )	0.22
Module porosity (%) $A_{fibres} / A_{Module}$	78
Bundle porosity (%) $A_{fibres} / A_{bundle}$	56
Packing density ( $m^2 \cdot m^{-3}$ )	320



## II - Short term - Screening effect

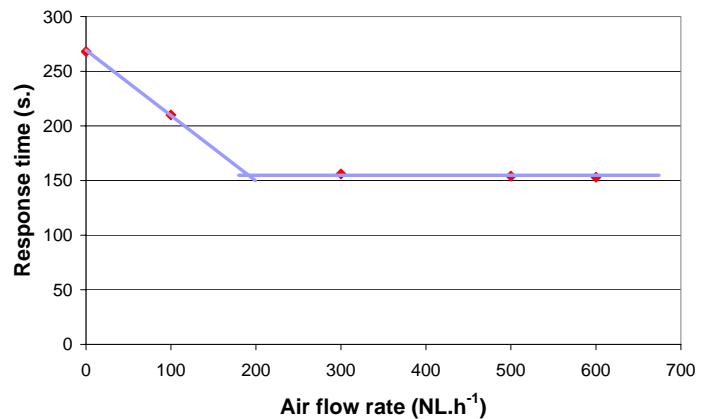
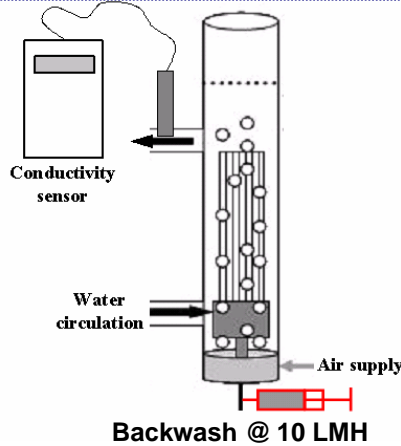
- SS monitoring during sludge Circulation in the filtration tank (without filtration)



Membrane bundle

- Detrimental effect of aeration within the first 15 minutes
- The membrane screening effect depends on
  - Packing density and local turbulence
  - fiber size and mobility

## II -Short term : Specific RTD = Mixing time



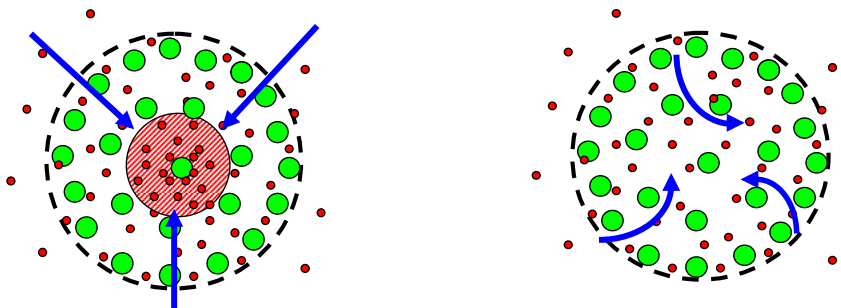
→ Stabilization of the response time shows a sufficient air bubbling effect to insure the macro mixing and the renewing of the water throughout the fiber bundle,

→ This observation is correlated with a visual observation of (i) air penetration inside the fiber bundle and (ii) individual fiber mobility

## II - Short term - Homogenous Filtration

→ At non sufficient aeration rate, some sludge accumulation appeared mainly in the central part of the bundle and induces a radial distribution of the filtration condition

→ In opposite at sufficient aeration rate the filtration remains homogenous on each fibre wherever its position inside the bundle



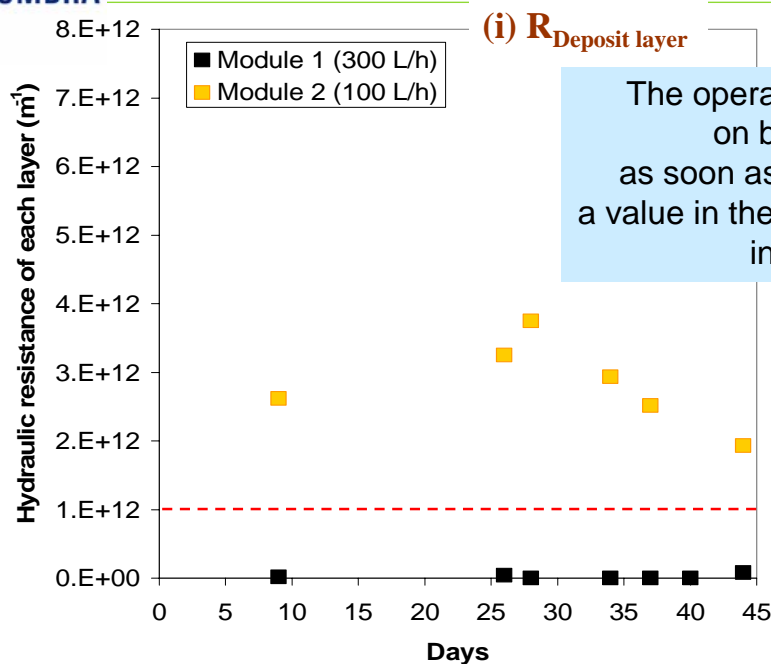
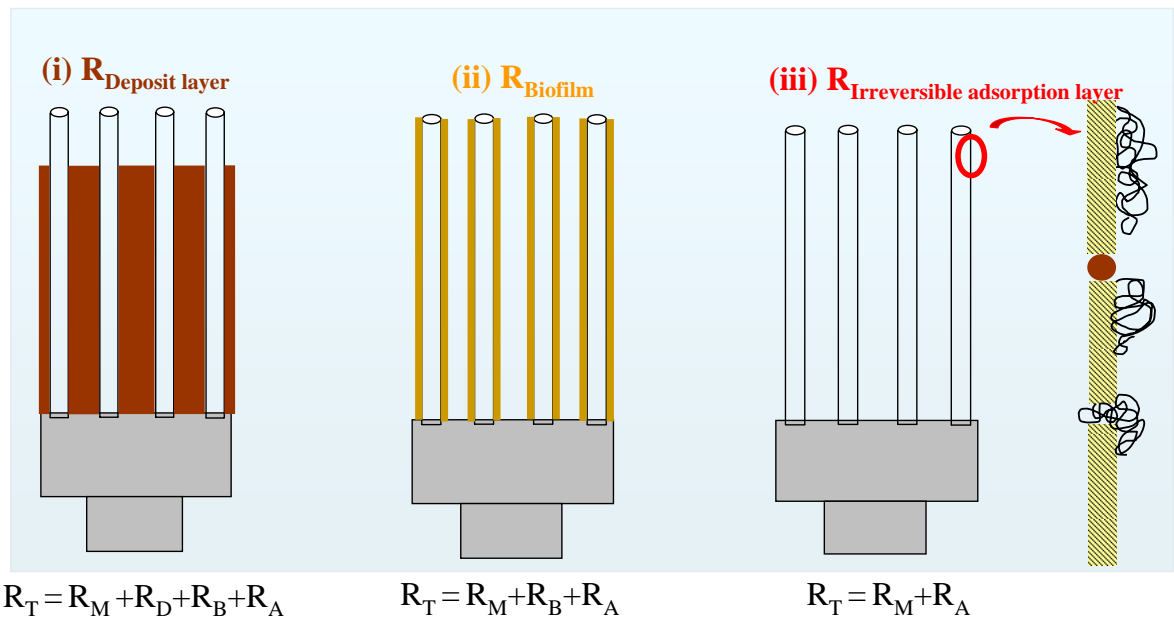
A **SPECIFIC MODELLING** (including dead end filtration law and the knowledge of (i)TMP evolution and (ii) specific sludge hydraulic resistance) allows the evaluation of the **SLUDGE ACCUMULATION RATE** inside the bundle = **OPTIMISATION OF HYDRAULIC CLEANING MANAGEMENT** (modification of air intensity, relaxation, backwashing)

$$PTM = PTM_0 + \mu \alpha C_{eq} \cdot J^2 \cdot t$$

$$m_{acc.} = \frac{m_{acc.}}{Filtration_{Time}} = A \cdot J_w \cdot C_{eq}$$

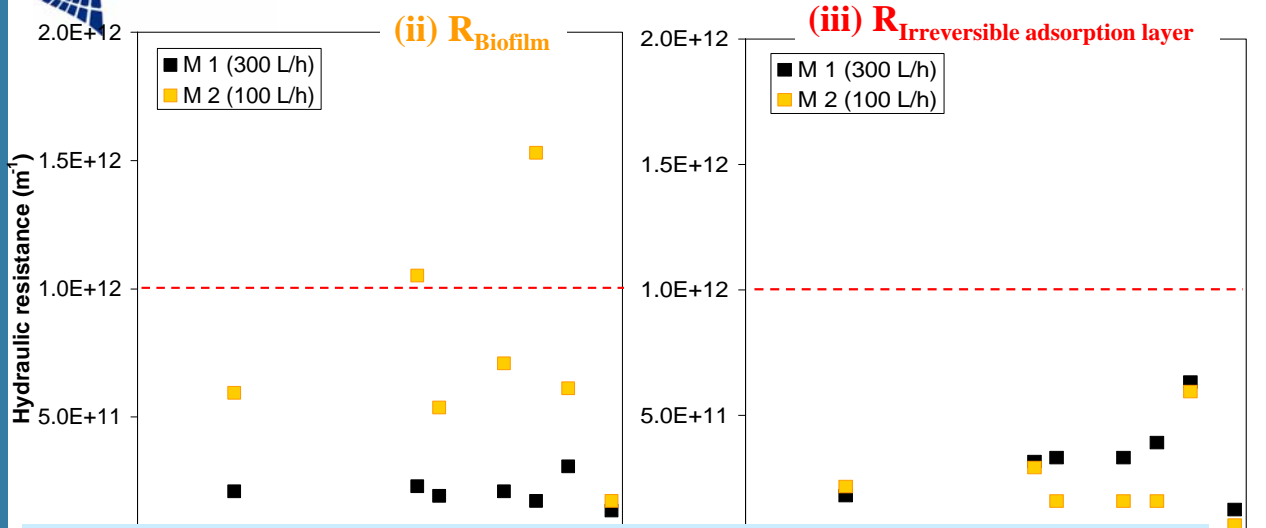


IV - long term experiment : Influence of the different scale up of fouling



IMPORTANCE OF AERATION ON SLUDGE ACCUMULATION

➔ At low aeration rate, the hydraulic resistance of deposit appears significant and much higher than in the most aerated bundle



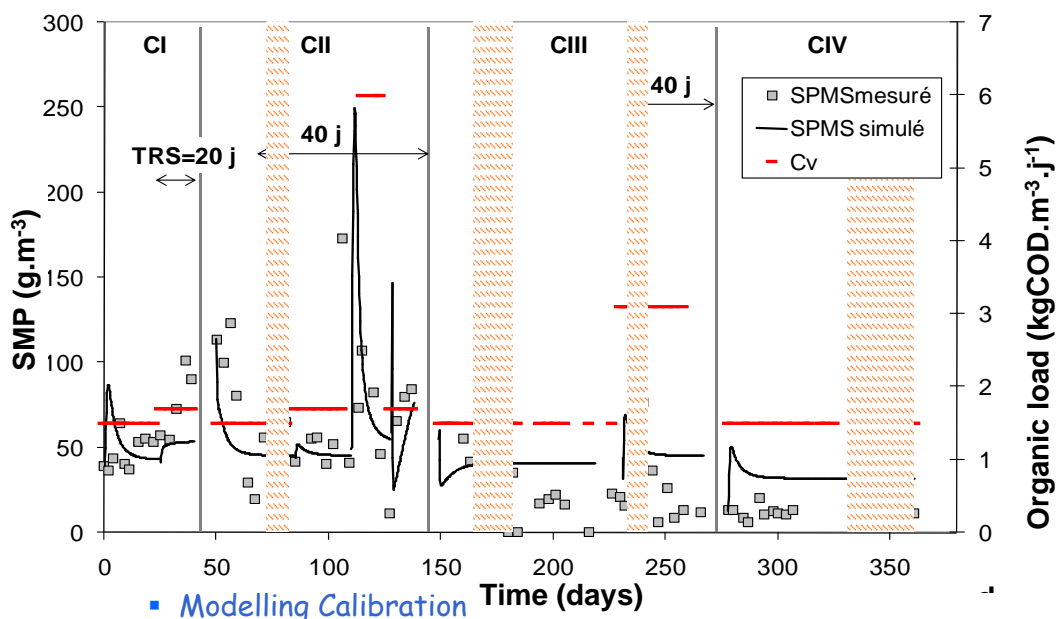
- IN BOTH CASES THE HYDRAULIC RESISTANCES DUE TO BIOFILM AND ADSORPTION REMAIN LARGELY LOWER THAN THE RESISTANCE OBSERVED IN THE PRESENCE OF SLUDGE ACCUMULATION INSIDE THE BUNDLE

- INTEREST TO AVOID SLUDGE ACCUMULATION

- INTEREST TO DEFINE PREDICTIVE TOOLS TO EVALUATE SMP PRODUCTION / ACCUMULATION

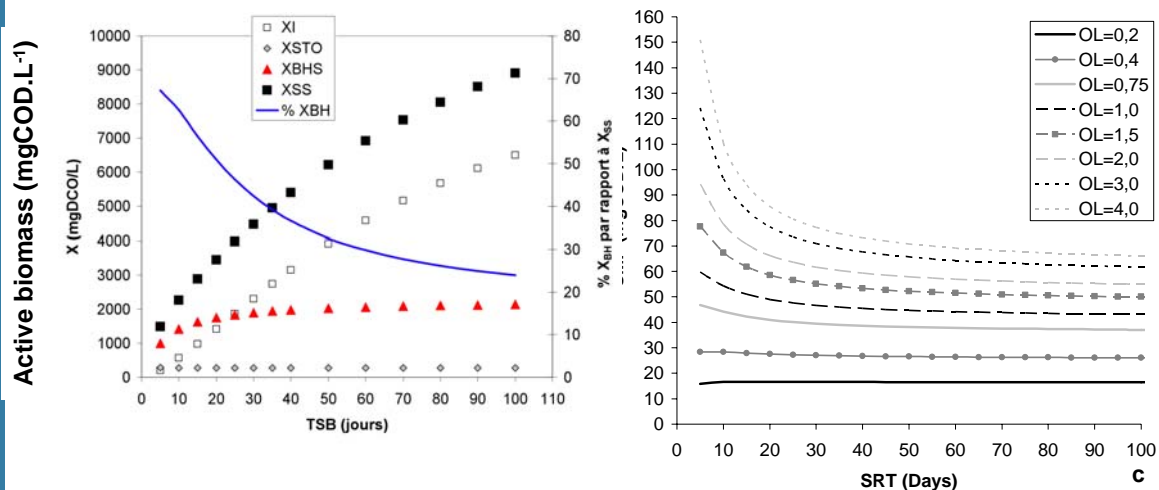
45

u



■ Modelling Calibration

▪ Simulation in steady state conditions with acetate as influent



- ➔ The organic load directly impacts active biomass and  $S_{SMP}$  concentrations,
- ➔ An adapted SRT allows the reduction of inert biomass and  $S_{SMP}$  concentrations,
- ➔ These simulations highlight the operating conditions that favor the control of TSS, active biomass and  $S_{SMP}$  production.

## V - Conclusion

- ➔ Short term experiment are useful to validate module configuration in regard with quick clogging (packing density and aeration impact).
- ➔ Results points out the importance of sludge accumulation on the total hydraulic resistance and the determining role of aeration to control its intensity.
- ➔ The analyses of irreversible fouling origin underline the negative role of soluble SMP.
- ➔ Introducing SMP production in ASM highlights the role of operating conditions (OL, HRT, SRT) and favors the identification of optimal functioning conditions

## Thank you for your attention and Acknowledgement

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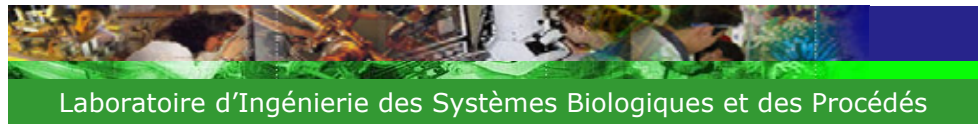
More info: [www.mbr-network.eu](http://www.mbr-network.eu)

## 13. NEW ADVANCES IN MBR AERATION AND FOULING CONTROL

*S. Pollet, B. Teychene, S. Khirani, M. Sperandio,  
C. Cabassud, C. Guigui*

# New advances in MBR aeration and fouling control

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INSA, CNRS, INRA, UMR 5504, UMR 792



Laboratoire d'Ingénierie des Systèmes Biologiques et des Procédés

## MBR process

KEY ISSUE= AERATION

Limiting phenomena

permeate

**Fouling** : adsorption, pore blocking, surface deposit

**Bundle clogging**

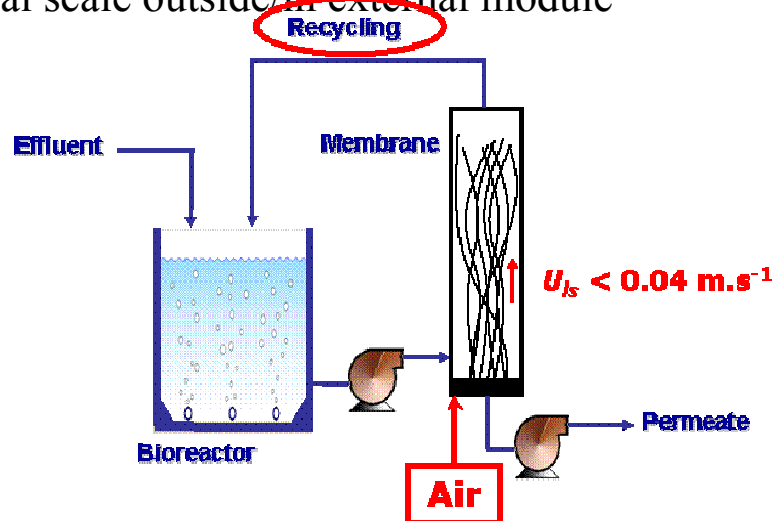
air air

What is the **role of aeration** on fouling and clogging ?

What is the influence of **bundle configuration** on fouling and clogging ?

# Objectives

- To propose a methodology/strategy for a scientifically based optimisation of the aeration process
- To present as an example some original results obtained with a semi-industrial scale outside/in external module



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## Scientific strategy

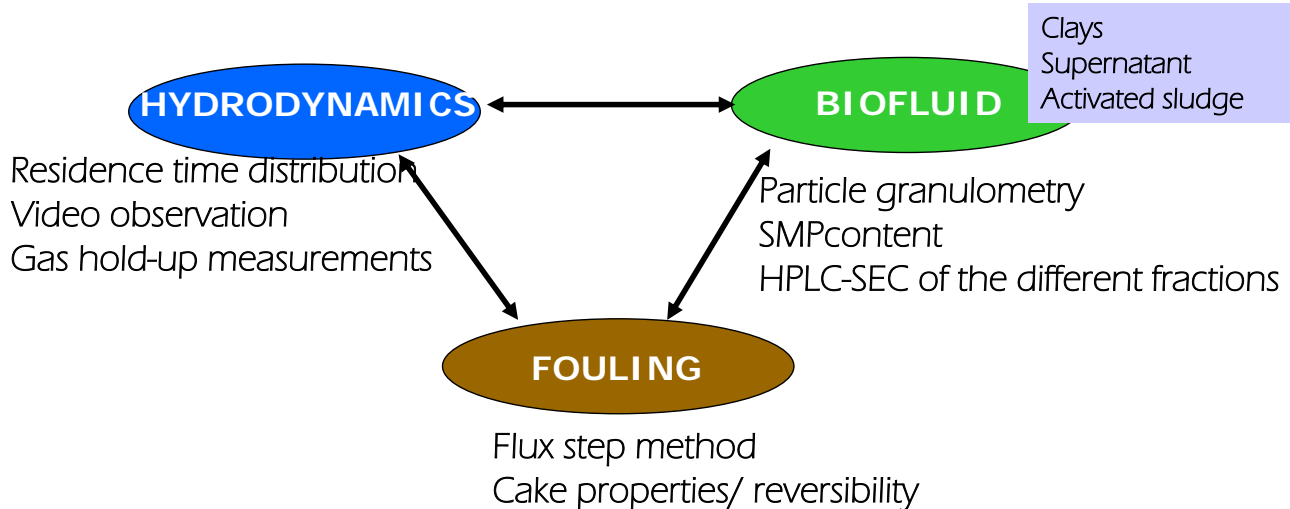
MEMBRANE

Configuration

OPERATING CONDITIONS

Aeration

Reproducible, comparable and time-saving methods

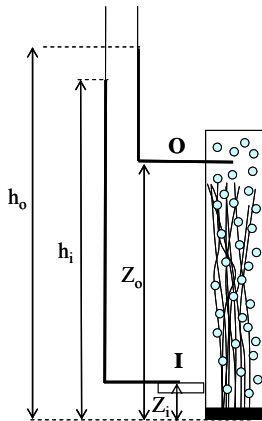


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## Methods to characterise hydrodynamic

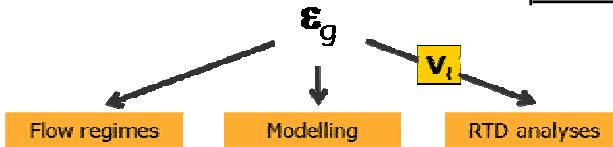
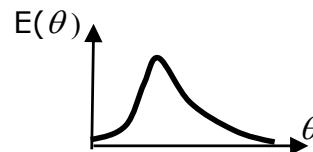
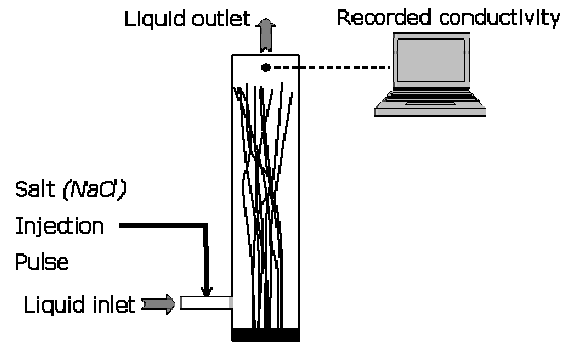
Water  
No filtration

### Gas hold-up measurements



$$\epsilon_g = \frac{V_g}{V_{free}} = \frac{h_o - h_i}{Z_o - Z_i} \rightarrow V_l = V_{free} - V_g$$

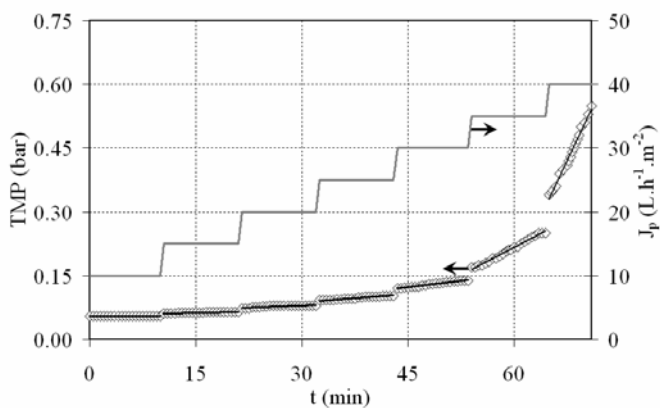
### Residence time distribution analyses



## Particle fouling

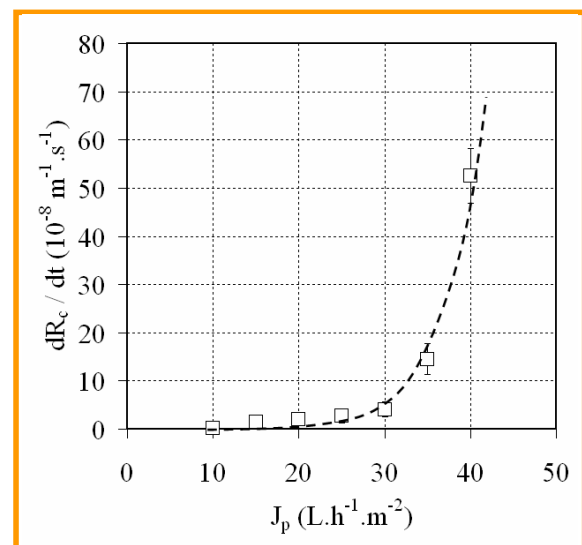
### Positive flux step method

Bentonite  
C = 0.65 g.L<sup>-1</sup>




$$\text{TMP} \rightarrow R_c = f(t)$$

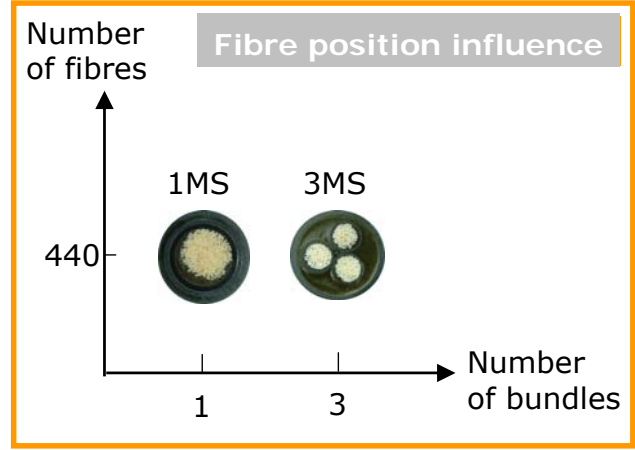
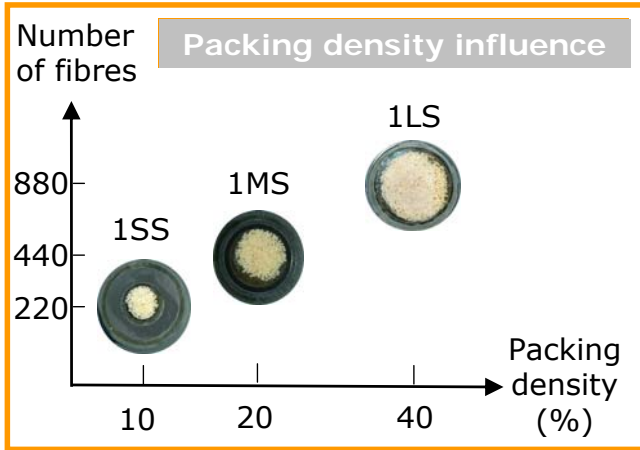
$$\frac{dR_c}{dt} = \frac{\Delta R_c}{\Delta t}$$








➔ A fingerprint of the fouling aptitude

### A set of HF modules


1MS  Medium size of bundle diameter

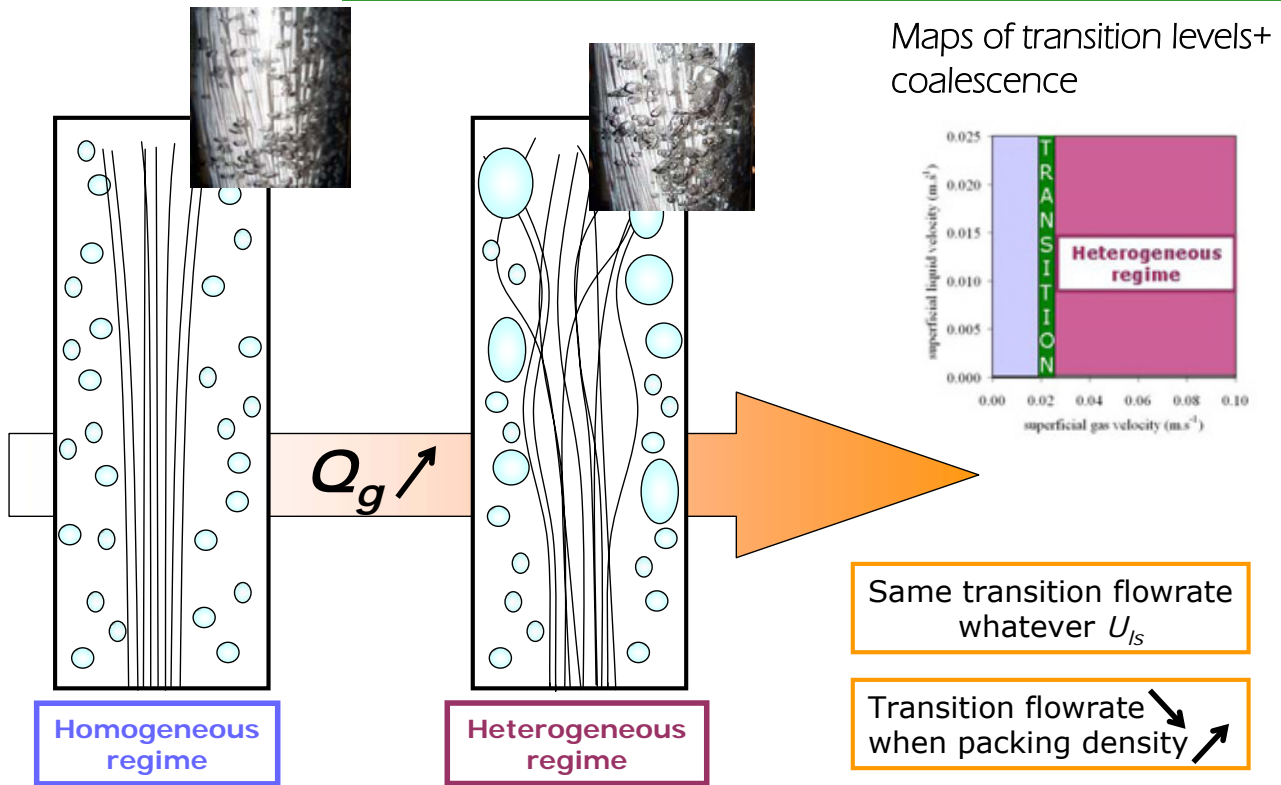


SAD<sub>m</sub> (Nm<sup>3</sup>.h<sup>-1</sup>.m<sup>-2</sup>) for every module.

Ug	Module				
	1SS	1MS	3MS	1LS	1LL
m.s <sup>-1</sup>					
0.038-0.154	0.6-2.4	0.26-1.0	0.26-1.0	0.10-0.38	0.23-0.92

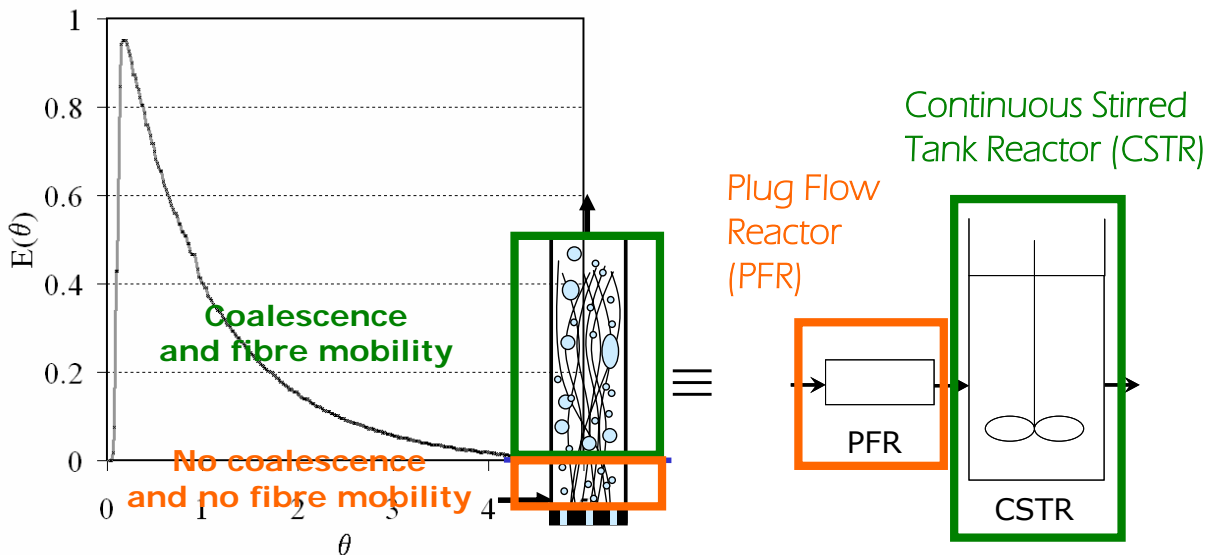
SAD<sub>p</sub> (Nm<sup>3</sup><sub>air</sub>.Nm<sup>-3</sup><sub>permate</sub>) for every module.

Ug	Module				
	1SS	1MS	3MS	1LS	1LL
m.s <sup>-1</sup>					
0.038-0.154	59-238	26-104	26-104	10-38	23-92

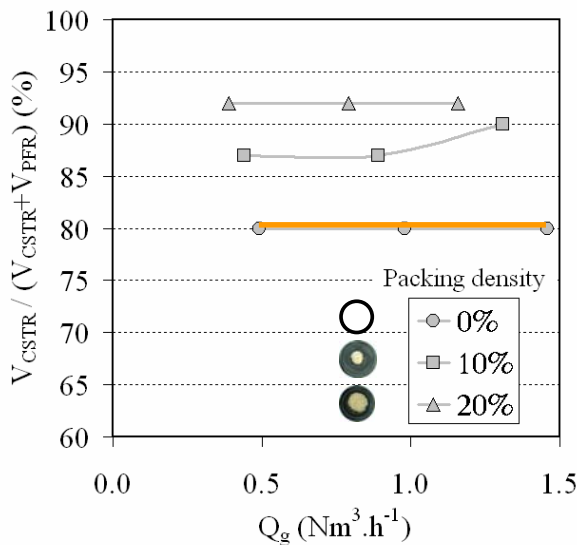
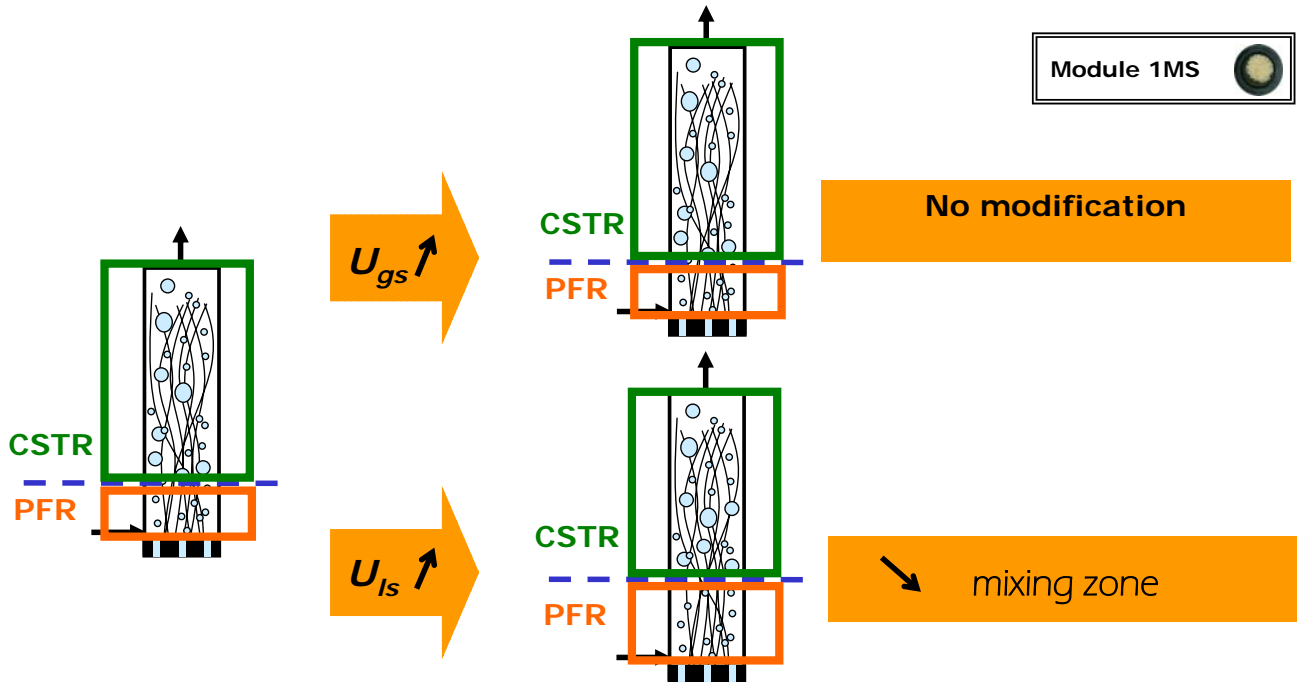


Hydrodynamic description of an aerated module

$U_{gs} = 0.038 \text{ m.s}^{-1}$   
Module 1MS



The relative proportion of the two zones is influenced by ....  
(for a given module)



Presence of fibres enhances mixing

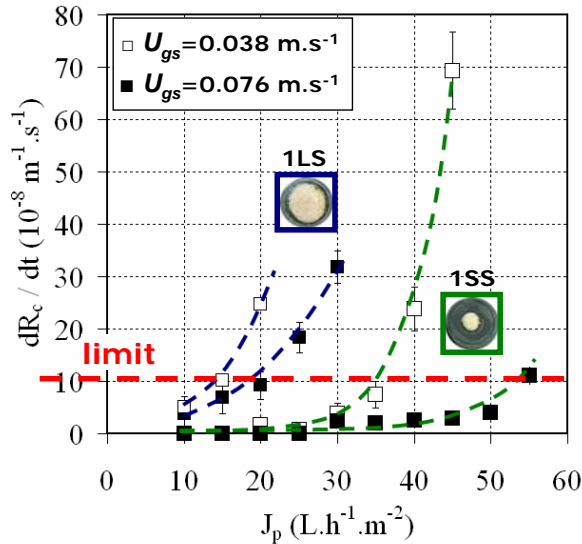
The proportion of the two zones is mainly affected by module design (packing density)

Can be influenced by the technology used to build the bundle

## Particle fouling

The choice of aeration conditions necessary to prevent particle fouling depends on.....

### MODULE PROPERTIES



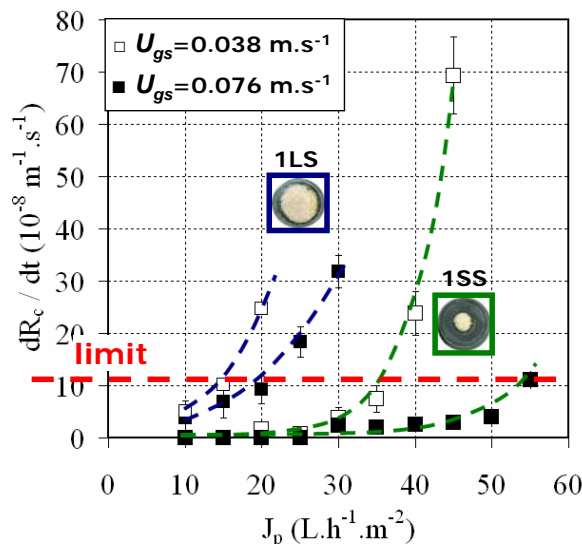
$J_p$ ( $\text{L} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ ) for $dR_c / dt \geq \text{limit}$		
$U_{gs}$	0.038	0.076
1SS	40	55
1LS	20	20

More compact module  
Higher number of fibers



Lower permeate flux for the same aeration

## Particle fouling



$J_p$ ( $\text{L} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ ) for $dR_c / dt \geq \text{limit}$		
$U_{gs}$	0.038	0.076
1SS	40	55
1LS	20	20

For a given module, ↑ aeration



Can be efficient ... or not

The required gas velocity depends on compacity

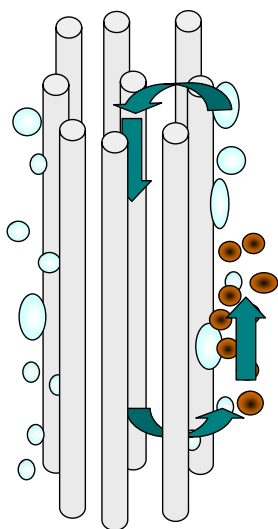
**Particle fouling**

Module 1MS 

$J_p$ (L.h <sup>-1</sup> .m <sup>-2</sup> ) for $dR_c / dt \geq$ limit			
$U_{ls}$ \ $U_{gs}$	0	0.019	0.038
0.013	25	30	35
0.025	25	30	35

The liquid velocity is not influent

**Gas Hold-up modelling thanks to a two-phase flow model**



An internal recirculation due to the gas flow = air-lift effect

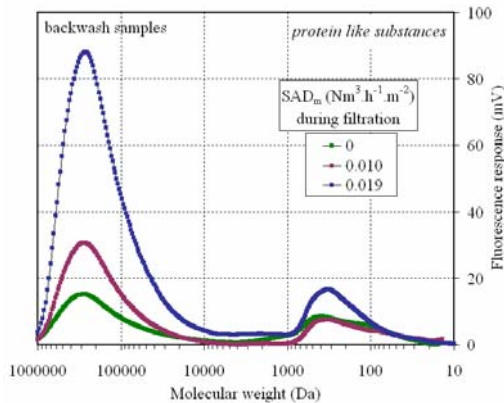
Internal recirculation can induce particles transport and clogging

More accumulation of particles in the bundle bottom, a non-mixing zone

Consequences on fouling can be >0 or <0  
 - bundle properties  
 - fibre ability to move (netting+ compacity)

# Fouling by a real activated sludge

## Analysis of backwash waters



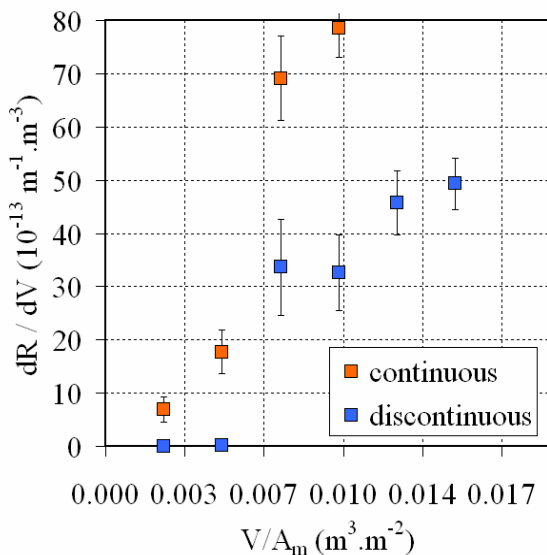
Sample	TOC mass (mg)		
	$U_{gs} = 0 \text{ m.s}^{-1}$	$U_{gs} = 0,038 \text{ m.s}^{-1}$	$U_{gs} 0,076 \text{ m.s}^{-1}$
Initial feed	130	139	139
Final feed	70	72	70
Backwash	22	23	42

Cake+clogging building and composition are affected by aeration conditions  
 Higher aeration → enhanced reversibility of limiting phenomena

## INTEREST OF SEQUENCED AERATION

(Same SADm=0.129m<sup>3</sup>/h/m<sup>2</sup>)

$U_{Is} = 0.013 \text{ m.s}^{-1}$   
 Module 1MS



- Discontinuous aeration limits TMP
- Discontinuous aeration limits fouling velocities
- TMP is similar before and after high aeration period

$R_c 10^{12} (\text{m}^{-1})$	7.2	0.6	92 %
$\alpha C 10^{13} (\text{m kg}^{-1})$	84.0	12.	85 %

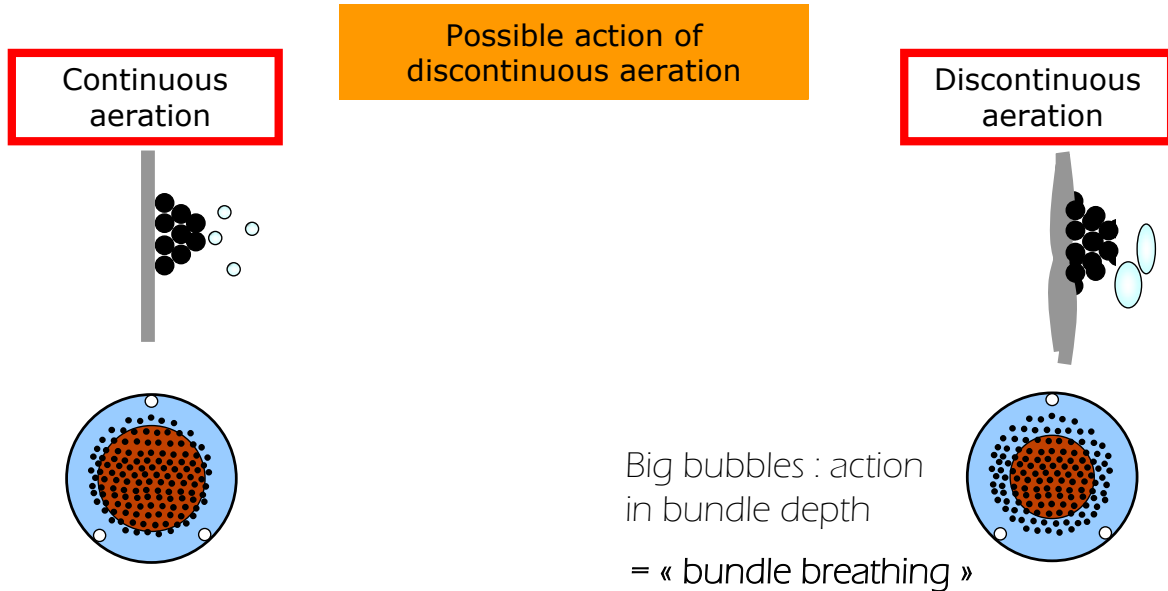
Differences in cake structure on fibre or bundle

$L_{p0}$ loss (%)	48	40	+ 8 %
$L_p$ recovery with a single backwash (%)	64	74	+ 10 %
Irreversible fouling (%)	28	16	43 %

Increase of reversibility

For the same aeration energy input

Discontinuous aeration (low level-high level) is better



## Conclusion

- Method that can be applied to other MBRs
- Module design: impact of compacity on aeration efficiency and on clogging
- Aeration conditions are to be optimised in relation with module design and manufacturing constraints ( fiber mobility)
- Discontinuous systems are favorable if they favor « breathing » of the bundle and thus clogging removal
- Aeration influences biofluid properties but also nature and reversibility of fouling

## Acknowledgments

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Contract No. 018480 - EUROMBRA

Duration: 01/10/05 - 30/09/08

EUROMBRA is part of the MBR-NETWORK Cluster



More info: [www.mbr-network.eu](http://www.mbr-network.eu)

# 14. SLUDGE FILTERABILITY AND DEWATERABILITY IN MUNICIPAL MBR

*G. Guglielmi, D. Chiarani, G. Andreottola*

MBR-Network  
Final workshop  
31<sup>st</sup> March – 1<sup>st</sup> April 2009  
Berlin

## Sludge filterability and dewaterability in a municipal MBR

G. Guglielmi, D. Chiarani, D.P. Saroj\*, G. Andreottola  
Università degli Studi di Trento, Italy  
\*UNESCO-IHE, Delft, The Netherlands



## Aims

- ▶ Investigate the characteristics of excess sludge from a large pilot scale plant in terms of:
  - dewaterability
  - filterability
  - settleability
  - statistical analysis
- ▶ 60 tests on a pilot-scale fixed-volume-chamber filter-press:
  - 6 polymers
  - 3 pressure values
  - different dosages
- ▶ Effect of sludge aerobic stabilisation on:
  - residual respiration rate
  - physical properties (SRF, CST)
  - biomass activity

## Experimental set-up (1/2)



[www.mbr-network.eu](http://www.mbr-network.eu)

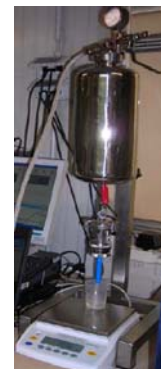
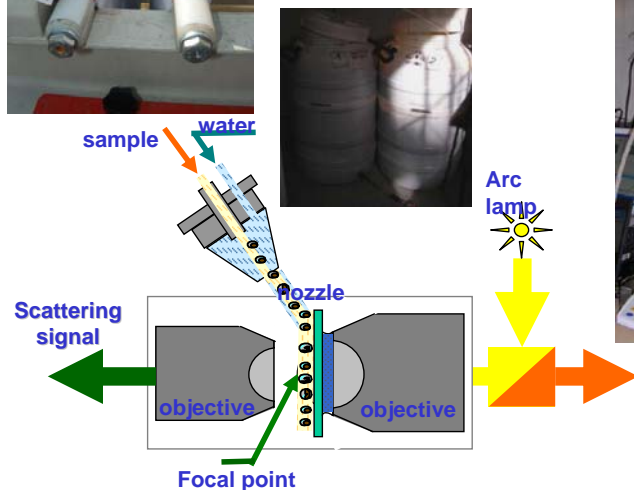
## Experimental set-up (2/2)



- ▶ Fixed-volume-chamber filter-press (2.1 L)
- ▶ Tests on surplus sludge from the pilot unit
- ▶ 6 commercial chemicals
- ▶ 7, 11 and 15 bar pressure
- ▶ 2 hours filtration
- ▶ Dosage: 5-25 g<sub>polymer</sub> kgTS<sup>-1</sup>
- ▶ 0.5% solution (w/w)



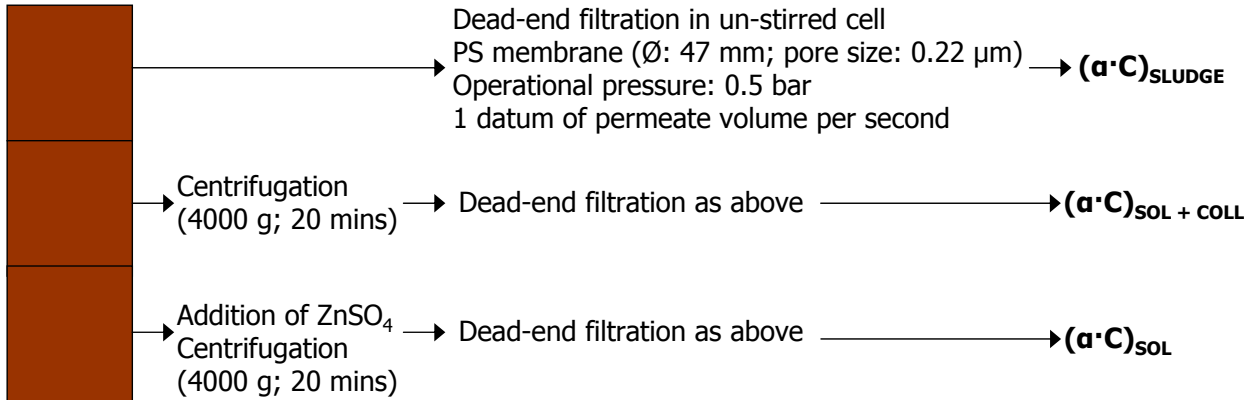
- ▶ 2 aerobic digesters (SRT<sub>1</sub> = 50 d; SRT<sub>2</sub> = 10 d)
- ▶ Biological properties (SOUR<sub>H</sub>, active VSS with flow-citometry)
- ▶ Physical properties (CST, SRF)
- ▶ DO ≥ 4 g m<sup>-3</sup>



[www.mbr-network.eu](http://www.mbr-network.eu)

# ( $\alpha$ -C) measurement

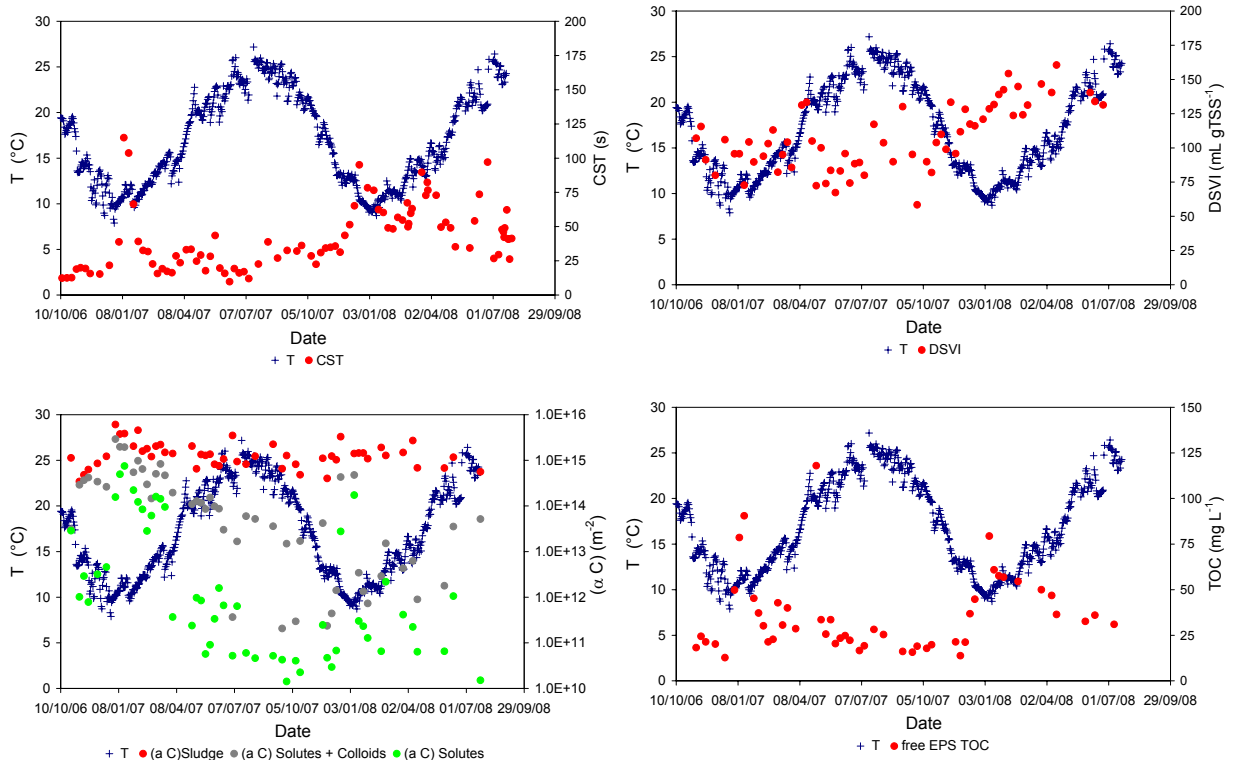
Sludge sample



Data elaboration according to Carman-Kozeny equation

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

# Results: T and CST vs time

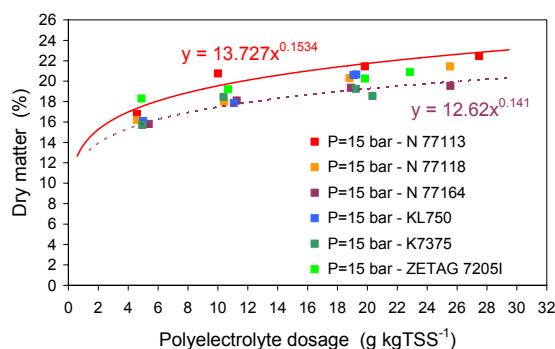
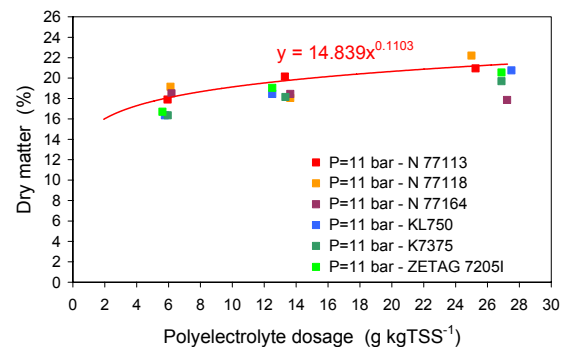
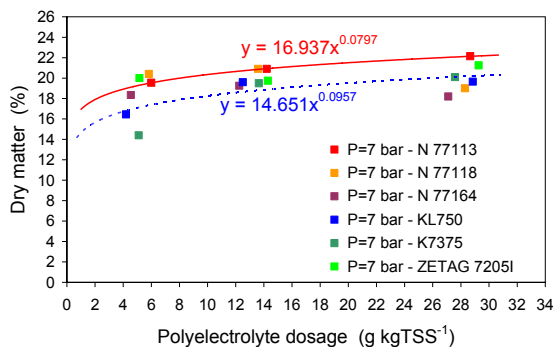


# Statistical analysis – Pearsons' coefficient

	TOC-EPSf	TOC-EPSb	CST	( $\alpha C$ ) <sub>sludge</sub>	( $\alpha C$ ) <sub>sol+coll</sub>	DSVI	T	MLSS
TOC-EPSf	1	0.207	0.656*	0.483*	0.670*	-0.295*	-0.237	0.266
TOC-EPS <sub>b</sub>	0.207	1	-0.169	-0.247	-0.124	0.082	0.455*	0.048
CST	0.656*	-0.169	1	0.536*	0.658*	0.437*	-0.436*	0.236
( $\alpha C$ ) <sub>sludge</sub>	0.483*	-0.247	0.536*	1	0.309*	-0.140	-0.435*	0.128
( $\alpha C$ ) <sub>sol+coll</sub>	0.670*	-0.124	0.658*	0.309*	1	-0.090	-0.580*	0.145
DSVI	-0.295*	0.082	0.437*	-0.140	-0.090	1	-0.332*	0.291*
T	-0.337*	0.455*	-0.436*	-0.435*	-0.580*	-0.332*	1	-0.238
MLSS	0.266	0.048	0.236	0.128	0.145	0.291*	-0.238	1

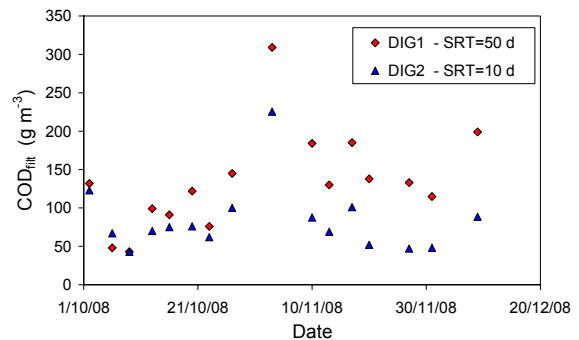
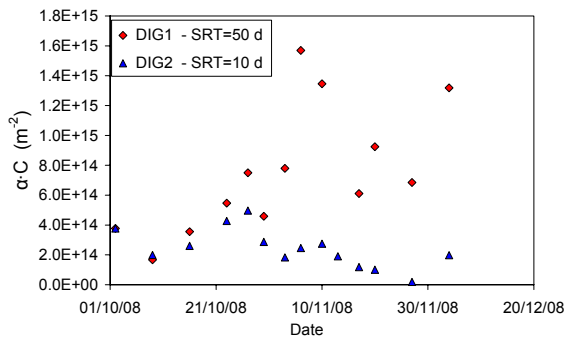
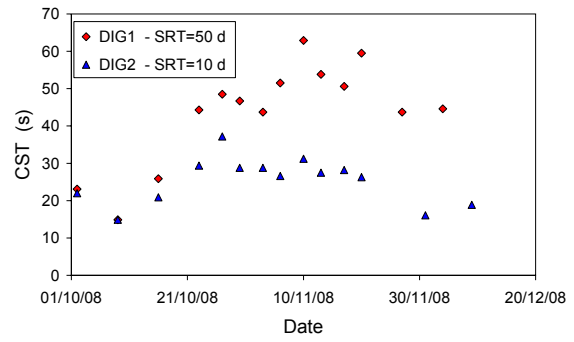
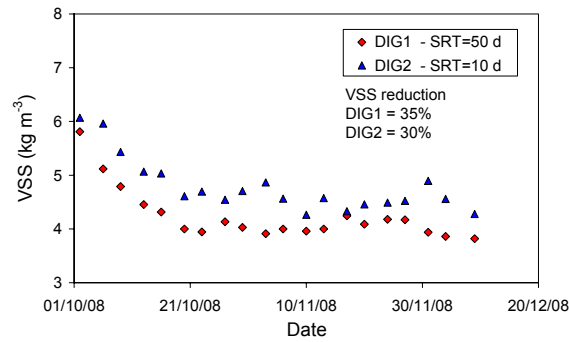
Significant correlation at 0.05 level (2-tailed)

# Filter-press tests: effect of polymer

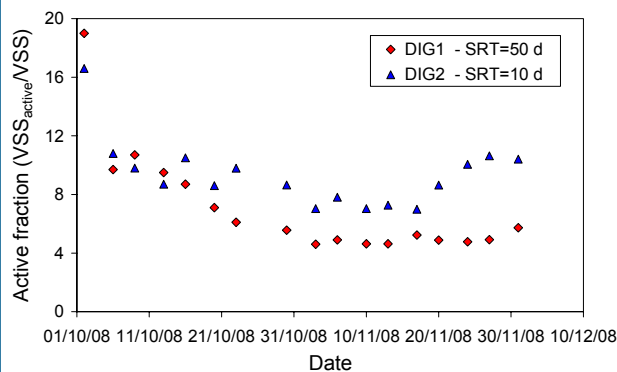
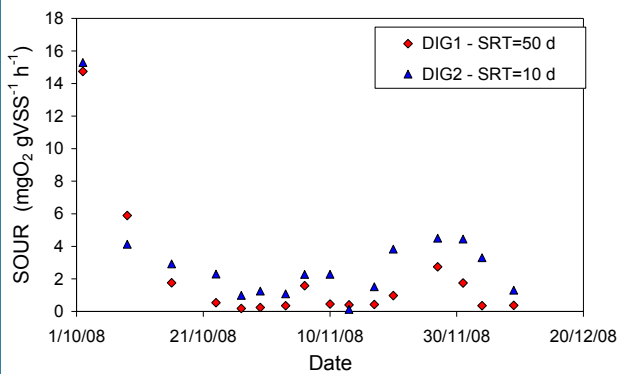


- ▶ DM % ranged between 16-24%
- ▶ Influence of pressure was negligible
- ▶ Best polymer: N7713
- ▶ Same behaviour of CAS sludge

# Aerobic digestion tests



# Aerobic digestion tests



- ▶ Only slight decrease of VSS
- ▶ High SRT negatively affects physical properties
- ▶ Indirect and direct measurements show a decrease of active heterotrophic biomass
- ▶ Other detrimental effects of long SRT

## Conclusions

- ▶ Effect of temperature on physical properties:
  - Temperature plays an important role on CST, ( $\alpha$ -C) and DSVI (negative correlation)
  - Temperature negatively correlates with organics in the liquid phase
- ▶ Pilot-scale dewatering tests:
  - weak impact of pressure
  - stronger impact of chemical
  - no relevant differences with CAS sludge
- ▶ Negative effects of higher SRT:
  - worse dewaterability and filterability
  - high energy costs for stabilisation (endogenous oxygen requirements)
  - only slight reduction of VSS

## Acknowledgement

EUROMBRA is a research project supported by the European Commission under the Sixth Framework Programme (Priority “Global Change and Ecosystems”)



Contract No. 018480 - EUROMBRA  
Duration: 01/10/05 - 30/09/08  
EUROMBRA is part of the MBR-NETWORK Cluster



More info: [www.mbr-network.eu](http://www.mbr-network.eu)

# 15. EFFECTS OF VARIOUS FLUX ENHANCERS IN MBR: LAB TESTS VS. PILOT SCALE OPERATION

*V. Iversen, S. Hermann, A. Drews, R. Mehrez, M.  
Ernst, M. Jekel, B. Lesjean, M. Kraume*

# Effects of various flux enhancers in MBR: lab tests vs. pilot scale operation

Vera Iversen\*, Stephanie Hermann\*, Anja Drews\*\*, Renata Mehrez\*\*\*,  
Mathias Ernst\*\*\*, Martin Jekel\*\*\*, Boris Lesjean\*\*\*\* and Matthias Kraume\*

\* Technische Universität Berlin, Chair of Chemical Engineering

\*\* University of Oxford, Dept. of Engineering Science

\*\*\* Technische Universität Berlin, Chair of Water Quality Control

\*\*\*\* Berlin Centre of Competence for Water



## Agenda

- ▶ Flux enhancers in MBR
  - Mechanisms
  - Literature review
  
- ▶ Pre-selection of flux enhancers
  - SMP-elimination
  - Biototoxicity
  - Filtration
  - Particle size distribution (PSD)
  
- ▶ TMP evolution in pilot plants
  
- ▶ Summary

# Mechanisms

## Flocculation

- cause colloids and other suspended particles in liquids to aggregate
- often multivalent cations
- metal salts, polymers and others

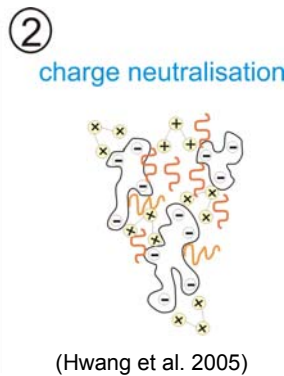
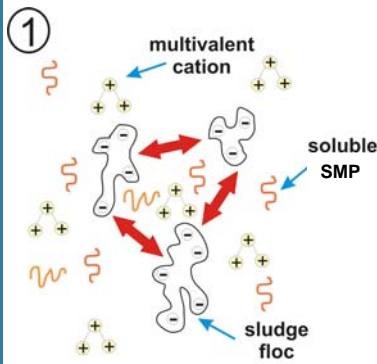
## Adsorption

- gas or liquid solute accumulates on the surface of a solid
- PAC (powdered activated carbon)

particular aggregation  
macroscopic flocs

combinations?  
other effects?

molecular aggregation  
microscopic flocs



- Elimination of SMP from supernatant
- Formation of larger flocs
- Change of supernatant viscosity?

Decrease of resistance

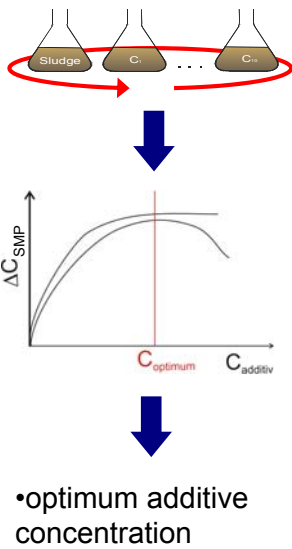
# Literature review – flux enhancement

Study	Additive	SMP	Permeability
Ji et al., 2008	aluminium sulphate, polymeric ferric sulphate chitosan		
Song et al. 2008	alum, ferric chloride		+
Remy et al., 2008	PAC		+
Munz et al., 2007		+	(+/-)
Yoon et al., 2006		+	+
Qiu, 2006		+	+
		+	+
		?	+/-
		?	
le Roux et al., 2006		+	+/-

Motivation: need of a conclusive study

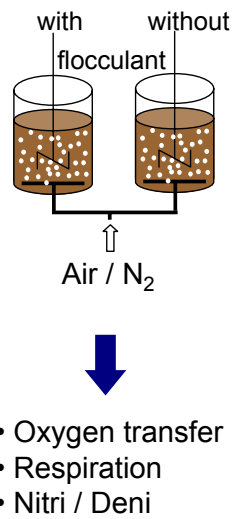
## Selection of flux enhancers ...

### Shaking flasks



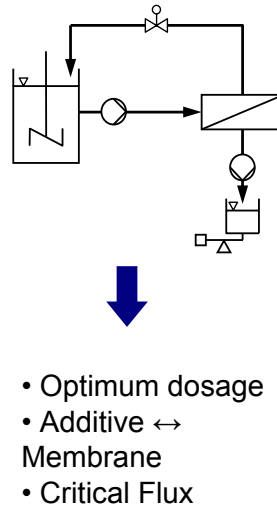
(Koseoglu et al., JMS, 2008)

### Biotoxicity



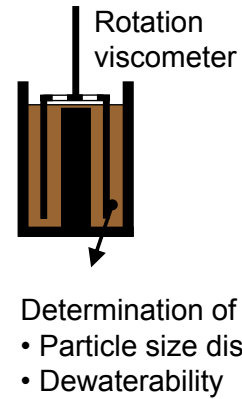
(Iversen et al., WST, 2007)

### Filterability



(Iversen et al., CIT, 2007  
Koseoglu et al., JMS, 2008)

### Particle size, Shear stability



(Iversen et al., MDIW, 2008)

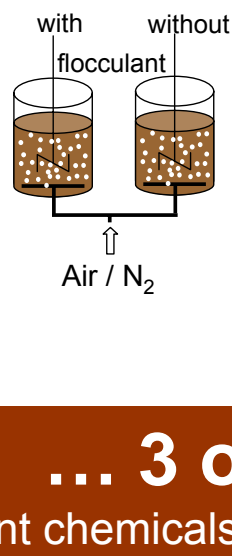
## Selection of flux enhancers ...

### Shaking flasks



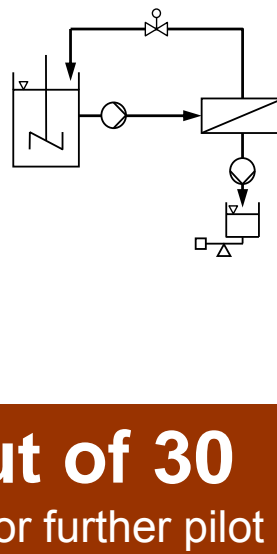
(Koseoglu et al., JMS, 2008)

### Biotoxicity



(Iversen et al., WST, 2007)

### Filterability



(Iversen et al., CIT, 2007  
Koseoglu et al., JMS, 2008)

### Particle size, Shear stability



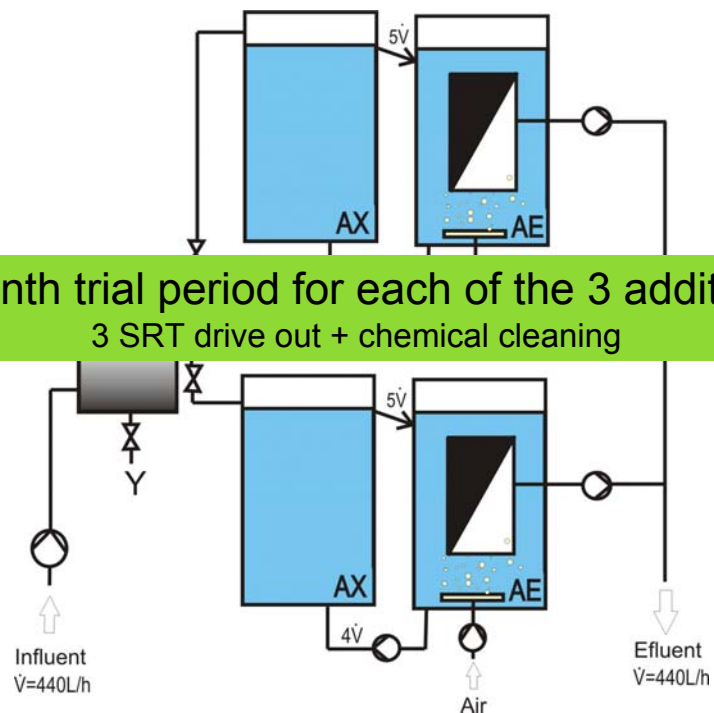
(Iversen et al., MDIW, 2008)

**... 3 out of 30**  
different chemicals for further pilot testing

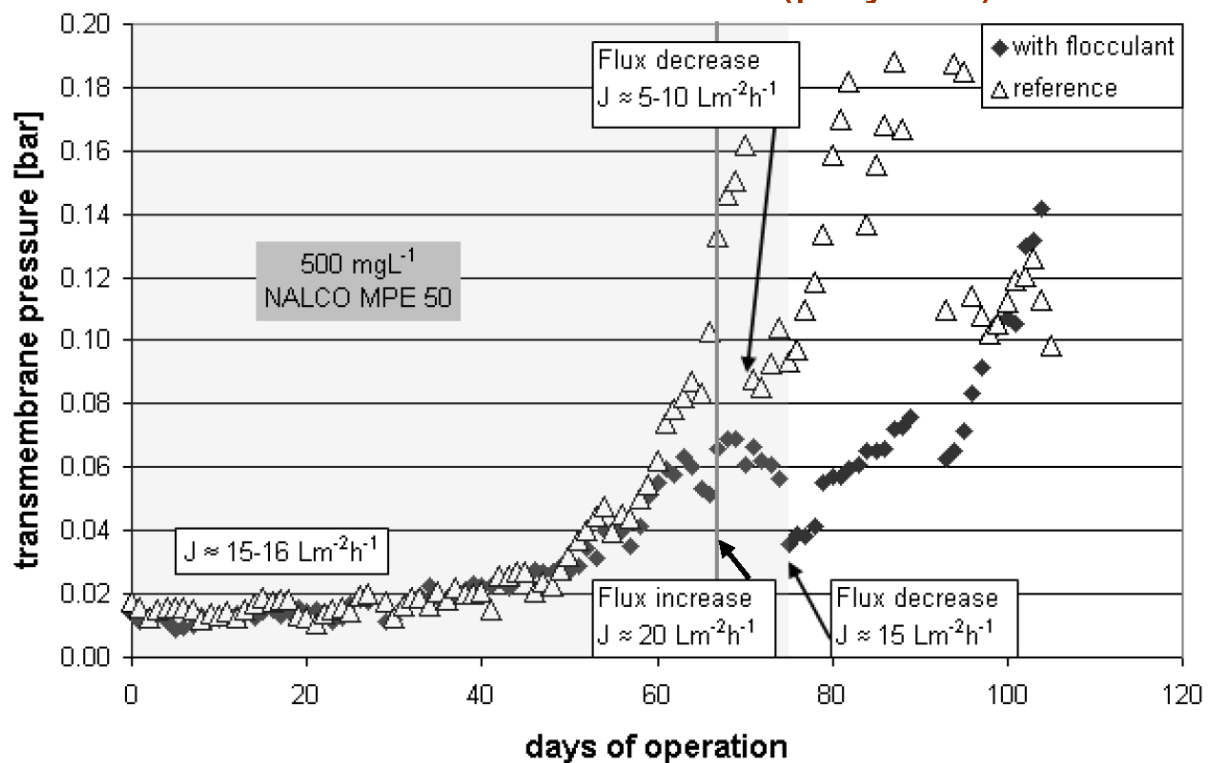
## Set-up of the parallel pilots

Volume = 1.6 m<sup>3</sup> each  
 HRT ≈ 7 h  
 SRT = 13 d  
 Membrane:  
 PVDF, 22 m<sup>2</sup>, 0.2 μm

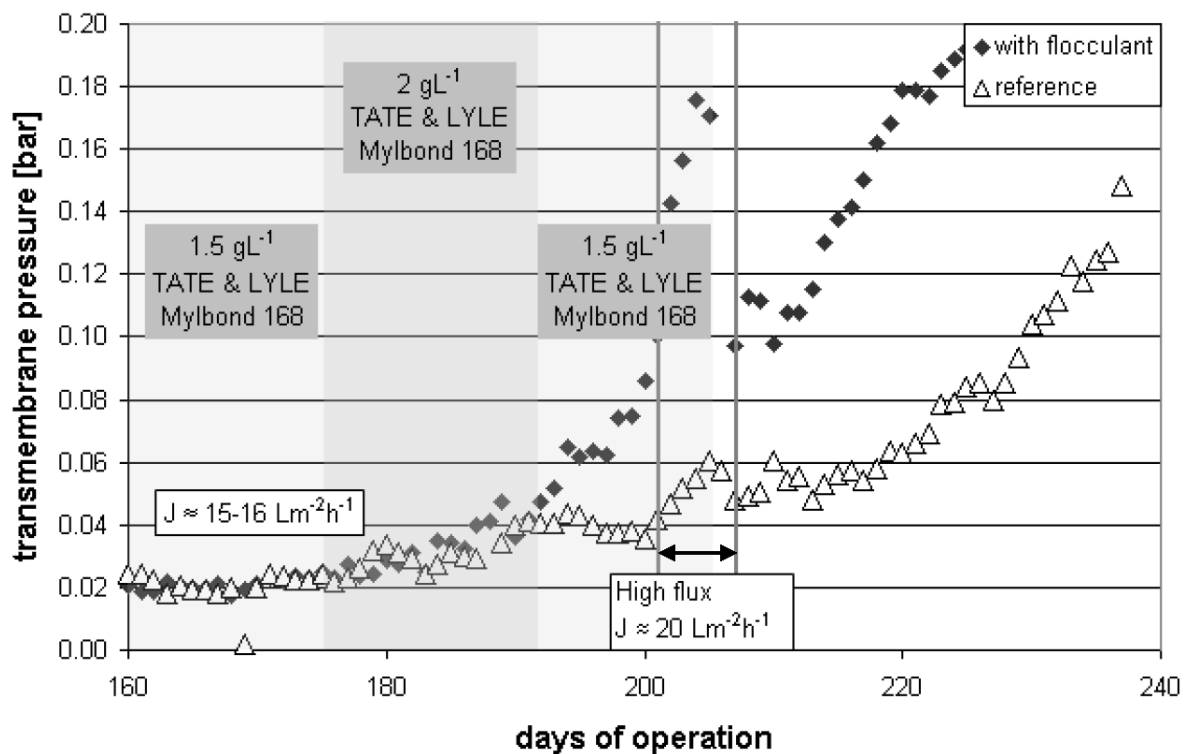
3 month trial period for each of the 3 additives,  
 3 SRT drive out + chemical cleaning



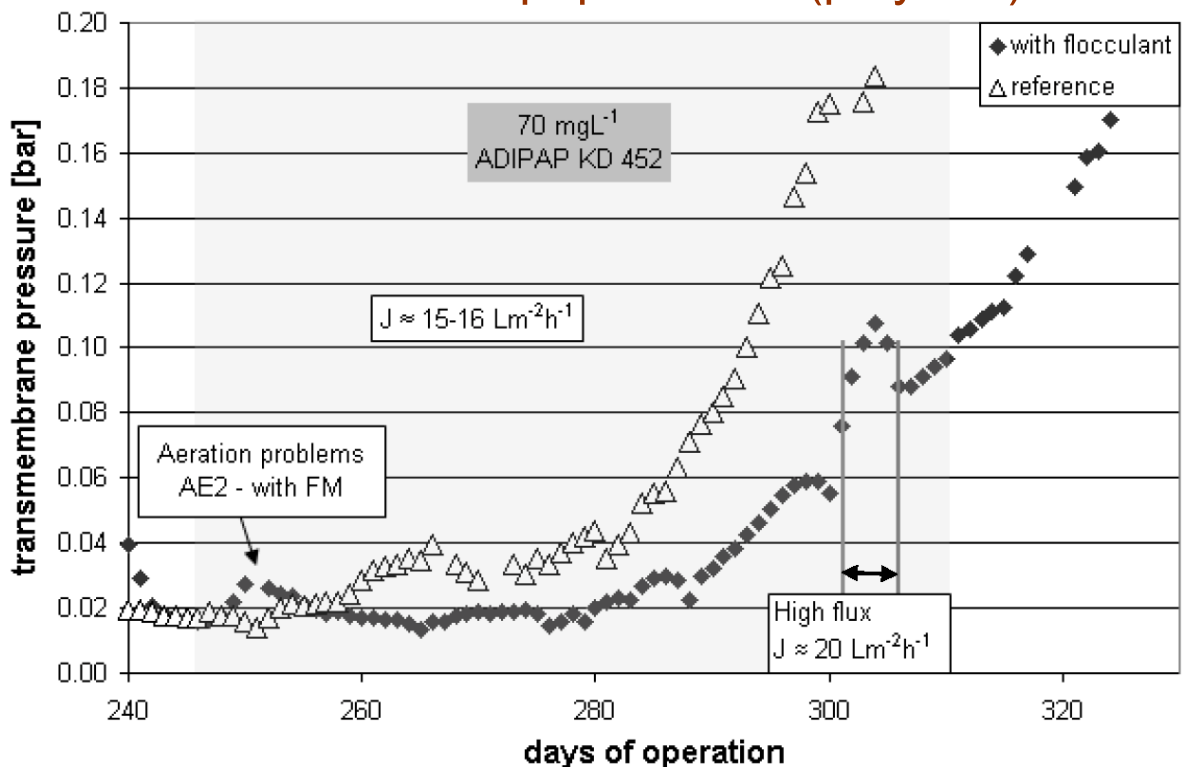
## Effect Nalco MPE 50 (polymer)



## Effect Tate & Lyle Mylbond 168 (starch)



## Effect Adipap KD 452 (polymer)



## Summary – 1

Substance	Product	C <sup>Add</sup> [mg L <sup>-1</sup> ]	SMP	k <sub>La</sub>	OUR	Nitri/ Deni	Particle size V	J <sub>crit</sub> test cell	Plant
<b>Metal salt</b>	<i>Magnasol 5108</i>	100	+	-	+	-	-	+	
	<i>Merck FeCl<sub>3</sub></i>	85	+	+	+	+	-	+	
<b>Chitosan</b>	<i>Chitosan 221</i>	200	++	-	-	+	++	-	
	<i>Chitosan 652</i>	250	+	+	-	+	+		
<b>Activated carbon</b>	<i>SA Super</i>	450	+	+	+	+	-		
	<i>Picahydro LP 27</i>	5000	+	-	-	-	-		
<b>Polymer</b>	<i>MPE-50</i>	500	++	++	+/-	+	+	++	+
	<i>MP H 30</i>	500	+						
	<i>MP L 30</i>	500	+	-	+	-	-	++	
	<i>Adifloc KD 451</i>	70	+	++	+	-	+		
	<i>Adifloc KD 452</i>	70	++	+	+	+	(+)	++	++
<b>Starch</b>	<i>Jaguar C162</i>	300		+	+	+	-		
	<i>Mylbond 168</i>	1500	+	++	+	+	-	+	--

## Summary – 2

- ▶ Selection of 3 additives out of 30
  - Good SMP-removal
  - Generally no biotoxic effects
  - Good flux enhancement in test cell trials
  - For the polymers: increase of particle size
  
- ▶ 2-3 month trials in pilot unit (2 polymers, 1 starch)
  - Enhancement for the polymers
  - No effects on elimination
  - Negative effect of starch (carbohydrate)
  - Starch „permeated“



**Need for large-scale validation!**  
**Especially polymers are effective for flux enhancement!**

## Acknowledgement

AMEDEUS is a research project supported  
by the European Commission  
under the Sixth Framework Programme  
(Priority “Global Change and Ecosystems”)



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More info: [www.mbr-network.eu](http://www.mbr-network.eu)

## 16. FOULING ORIGIN AND CLEANING STRATEGIES IN MBR

*A. Grelot, C. Machinal, A. Tazi-Pain, A. Grasmick.*

# Fouling origin and cleaning strategies in MBR

A. Grélot, C. Machinal, A. Tazi-Pain, A. Grasmick



Introduction

Materials & Methods

Results & Discussion

Conclusions & Perspectives

## Introduction

### Study context:

- Study within the framework of the European AMEDEUS project



### Partners:

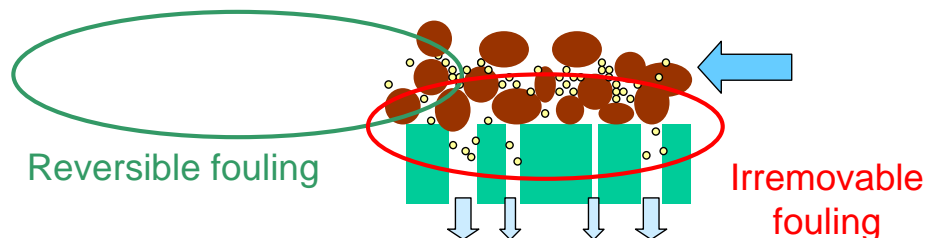
- Trials performed at Anjou Recherche
- Membrane developed by:
  - A3 Water Solutions
  - Polymem
  - inge
- Collaboration with the University of Montpellier



## Introduction

### Objective of the study:

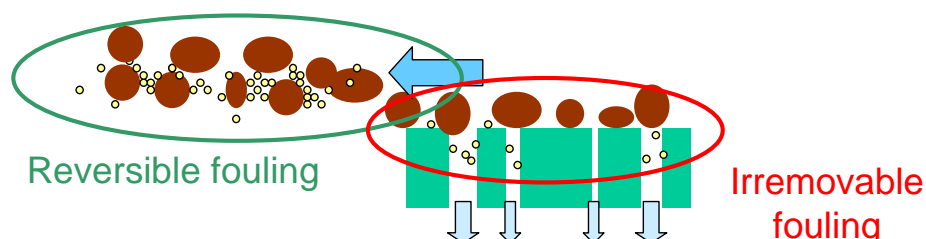
- Investigation of the fouling nature and origin of three different membranes provided by A3 Water Solutions, Polymem and Inge
  - ➔ Achieve a better control of the membrane fouling + reach lower operating costs
- Control of the **reversible fouling** (removable by physical means) at pilot-scale
- Analyse of the **irremovable fouling** and membrane regeneration with chemical cleanings



## Introduction

### Objective of the study:

- Investigation of the fouling nature and origin of three different membranes provided by A3 Water Solutions, Polymem and Inge
  - ➔ Achieve a better control of the membrane fouling + reach lower operating costs
- Control of the **reversible fouling** (removable by physical means) at pilot-scale
- Analyse of the **irremovable fouling** and membrane regeneration with chemical cleanings



## Content

▶ Material & Methods

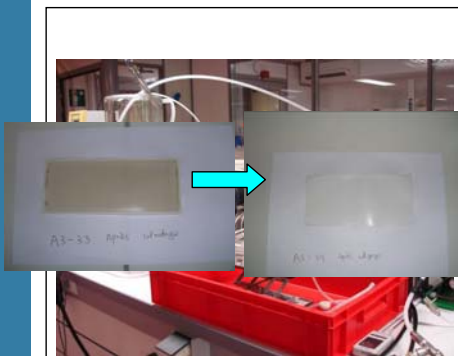
▶ Results:

- Control of the reversible fouling
- Analyse of the irremovable fouling of the membrane:
  - Resistance due to irremovable fouling part
  - Regeneration of the membrane with different cleaning product
  - Composition of the irremovable fouling part



▶ Conclusions & perspectives

## Irremovable fouling and cleaning: approach



1 -Fouling with sludge supernatant during 24h at lab-scale (P=0.2bar)



2 - Plates operated for 5 months in the MBR pilot plant



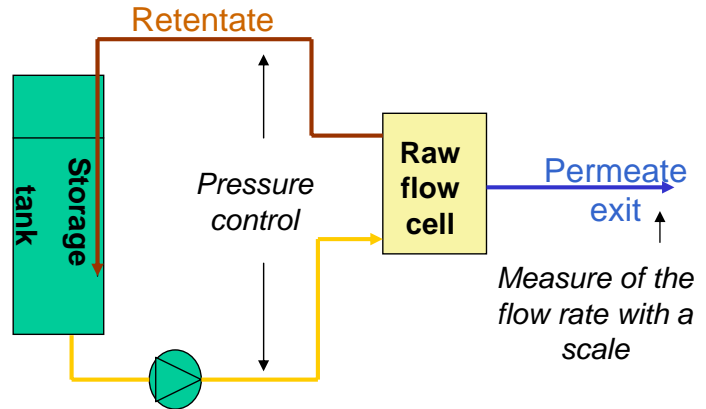
3- A3 Modules operated in MBR pilot plant

Same mixed liquor for each step:  
 $MLSS= 10g/l$ ;  $SRT= 25d$ ;  $F/M=0.13 \text{ kgCOD.kg MLSS}^{-1}.d^{-1}$

+ Analyse of the deposit on the fouled and cleaned membrane

## Permeability measurement

- ▶ Mini-Pilot built for the membrane permeability measurements
- ▶ A3 microfiltration PVDF membrane samples of 110 cm<sup>2</sup>



- ▶ Resistance due to the irremovable fouling:

$$R_{irr} = R_{after\ rinsing} - R_m$$




- ▶ Evaluation of the cleaning efficiency at lab-scale

## Cleaning products

Nature	Products
<b>REFERENCE</b>	Sodium hypochlorine ( $NaOCl$ )
<b>OXYDAZING AGENTS</b>	Caustic soda ( $NaOH$ )
	Hydrogen Peroxide ( $H_2O_2$ )
	Hydrogen Peroxide ( $H_2O_2$ ) at pH 11
	A3 Activor A 101 ( $KOH, NaOH, \dots$ )
<b>ENZYMES</b>	SERL
<b>ENZYMES + DETERGENTS</b>	Ultrasil 67/ 69 (ECOLAB)
	Filzym P (REALCO)
<b>ACIDS</b>	Hypochlorite acid ( $HCl$ )
	Citric acid ( $C_6H_8O_7$ )
	A3 activor AS 103 ( $HNO_3, H_3PO_4, \dots$ )




- ▶ Cleaning by soaking for 2h at room temperature

## Irreversible fouling part

Fouling protocols			
	Fouling with sludge supernatant	Fouling at pilot-scale (5 months)	Fouling at pilot-scale (12.5 months)
Rirr (x 10 <sup>12</sup> m <sup>-1</sup> )	0.23 ± 0.06 (26 samples)	0.20 ± 0.06 (17 samples)	0.25 ± 0.012 (7 samples)

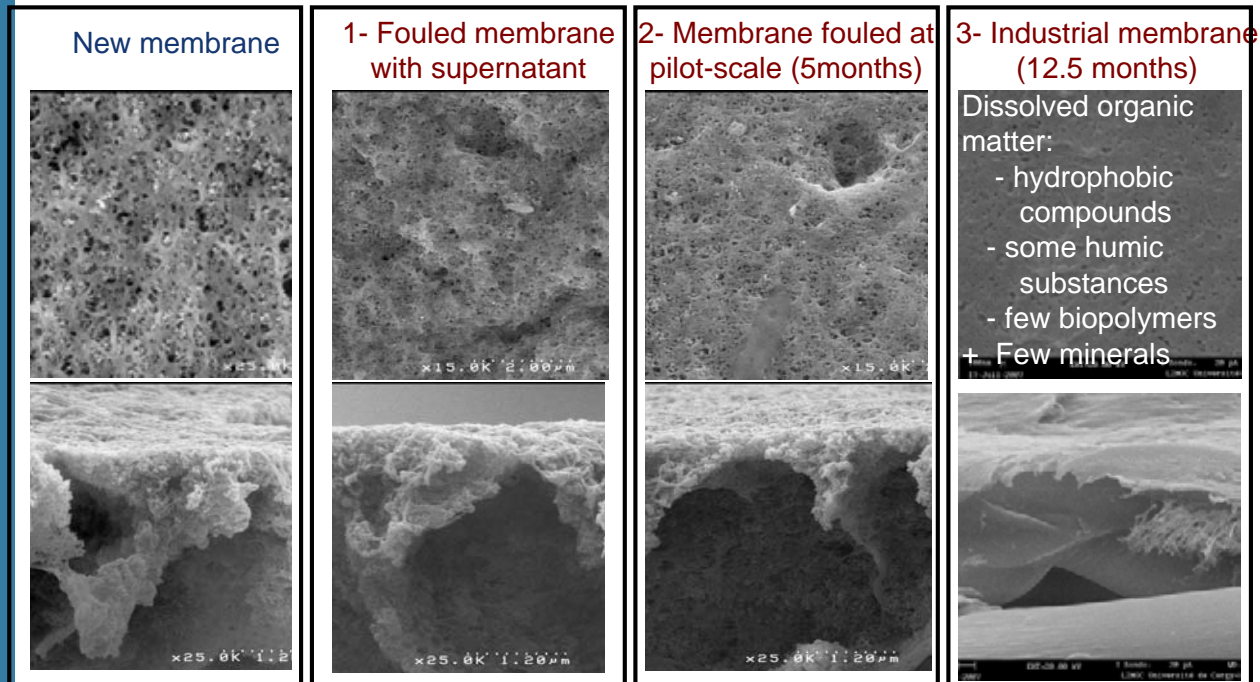
- ▶ Similar irremovable fouling part for the 3 fouling protocols
- ▶ Rirr > Rm (Rm= 0.10 x 10<sup>12</sup> m<sup>-1</sup>)

## Membrane regeneration with cleaning products

Fouling and cleaning protocols			
	<i>Cleaning at lab-scale</i>	<i>Cleaning at lab-scale</i>	<i>Cleaning in pilot-plant</i>
Efficient reagents	Chlorine 200–2000ppm Hydrogen peroxide Ultrasil 67/69 Filzym p	Chlorine 2000ppm Hydrogen peroxide	Chlorine 2000ppm
Least efficient reagents	NaOH	Chlorine 200ppm Ultrasil 67/69 Filzym p	Hydrogen peroxide

More chemical agents efficient on the membrane fouled with supernatant

## Fouling differences ?



Dissolved organic matter:

- hydrophobic compounds
- some humic substances
- few biopolymers

+ Few minerals

- ▶ Cleaning affected by the presence of an additional thin cake layer, presence of sludge

## Conclusions & Prospects

- ▶ Irremovable fouling:
  - $R_{irr} > R_m$
  - Development of a protocol at lab-scale to test foul membrane samples and test a wide variety of cleaning products
- ▶ Membrane regeneration:
  - Differences of the cleaning efficiency observed at the different scales because of a different fouling structure: full-scale tests remain essential
  - Chlorine was efficient for all tests
  - Other promising reagents: hydrogen peroxyde (longer soaking required ?)
  - Research of new cleaning reagents

## Publications

- Weinrich L. and Grélot A. (2007). *Evaluation of innovative operation concept for flat sheet MBR filtration system*. *Water Science and Technology* 57 (4), 613-620.
- Grélot A., Tazi-Pain A., Weinrich L., Lesjean B., Grasmick A. (2009). *Evaluation of a novel flat sheet MBR filtration system*. *Desalination* 236, 111-119
- Grélot A. Machinal C., Drouet K., Tazi-Pain A., Schrotter J.C, Grasmick A., Grinwis S. (2008). *In the search of alternative cleaning solutions for MBR plants*. *Water Science & Technology* 58(10), 2041-2049.
- Grélot A., Grelier P., Vincelet C., Bruess U., Grasmick A. (2008). Fouling characterisation of a PVDF membrane. *Membranes in Drinking Water Production and Wastewater Treatment Conference (MDIW08)*, Toulouse, 20-22 October 2008. (Submitted in 'Desalination')
- Grélot A., Grasmick A., Tazi-Pain A., Grelier P., Trouvé E., Lesjean B. (2009). Fouling control in a MBR pilot. Submitted in *Journal of Applied Membrane Science and Technology*.
- Grélot A., Busnot A., Grelier P., Tazi-Pain A., Heijnen M., Grasmick A. (2008). *The FiSh membrane: a new and appropriate configuration for MBR technologies*. *Membranes in Drinking Water Production and Wastewater Treatment Conference (MDIW08)*, Toulouse, 20-22 October 2008. (Submitted in 'Submitted in Desalination and Water Treatment')

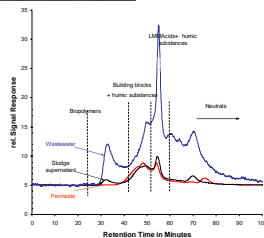
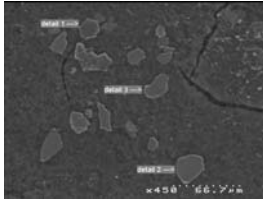
## Any questions



# Membrane characterisation and autopsy

## Investigation of different membrane samples:

- New membrane samples
- 4 membrane samples of the top module after one year operation (fouled membrane)
- 1 membrane samples of the bottom module after operation and cleaning with hydrogen peroxide (cleaned membrane)

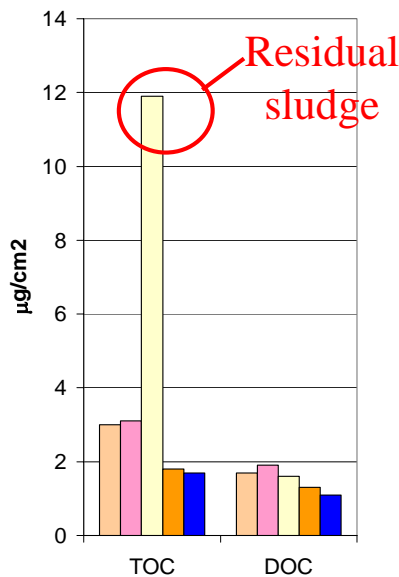


## Analyses and characterisations:

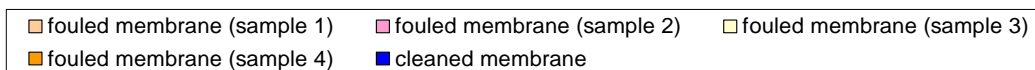
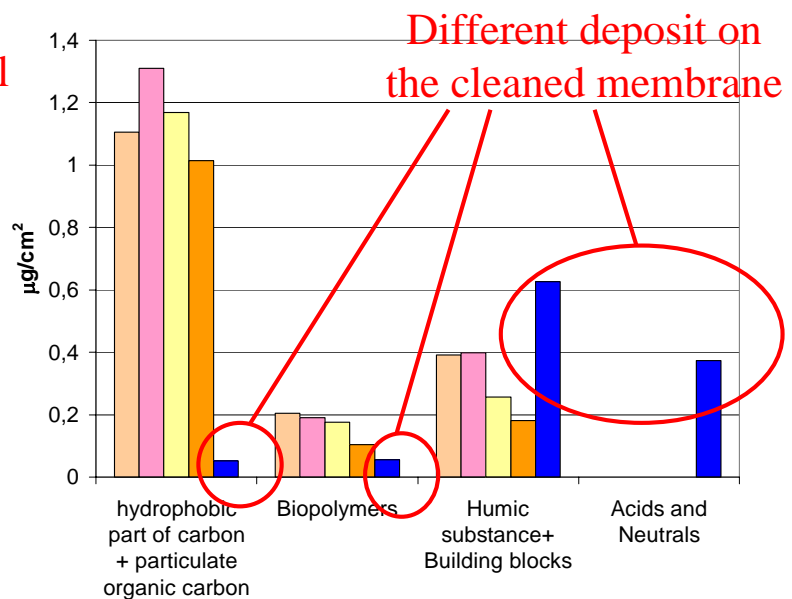
- Membrane permeability
- SEM pictures + EDAX
- Mineral elements by ICP
- Organic part : TOC and DOC
- Dissolved organic matter characterisation by SEC

# Membrane deposit of the membrane fouled in the pilot-plant: organic fraction

## Organic fraction

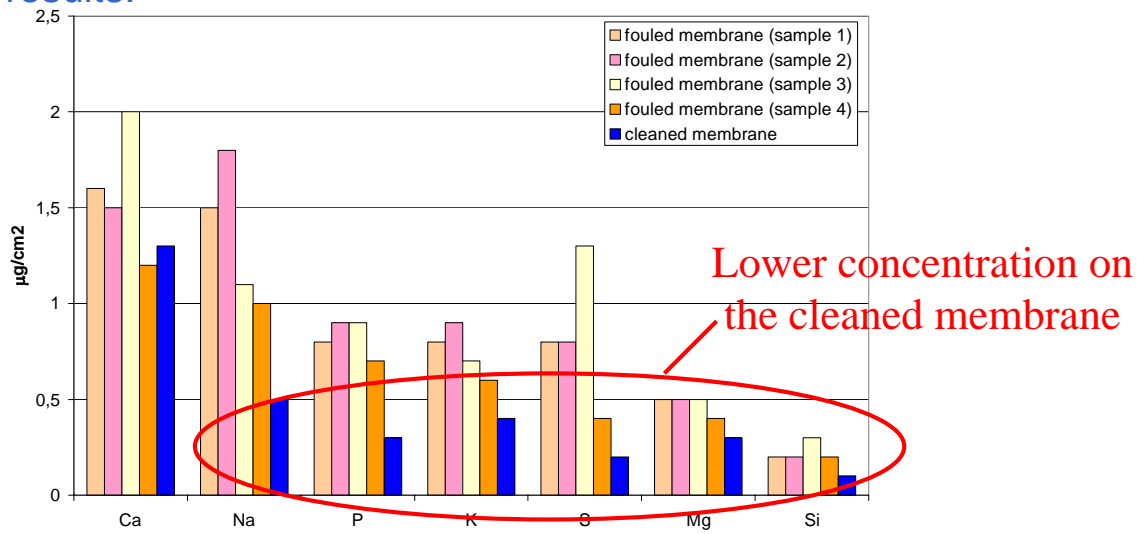


## Dissolved organic fraction composition



## Membrane deposit of the membrane fouled in the pilot-plant: mineral fraction

ICP results:



Elements detected by EDAX for all membranes:

- C, O, Cl from the membrane material
- Ca, Al, Fe, K, P, Si from the waste water

➔ Modification of the deposit after cleaning

# 17. OPTIMISED ELECTROSPUN NANOFIBERS FOR MUNICIPAL WASTEWATER TREATMENT BY TEXTILE BIOREACTORS (TBR)

*E. Fatarella, L. Lombardi, V. Iversen, S.  
Paulussen, U. Brüß, B. Lesjean, M. Kraume*

# Optimised Electrospun Nanofibers for Municipal Wastewater Treatment by Textile Bioreactor (TBR)

**Enrico Fatarella<sup>1</sup>; Luca Lombardi<sup>1</sup>; Vera Iversen<sup>2</sup>; Mathias Kraume<sup>2</sup>; S. Paulussen<sup>3</sup>; Ulrich Bruss<sup>4</sup>; Boris Leajeau<sup>5</sup>**

<sup>1</sup> Next Technology Tecnotessile, Prato (ITALY) – chemtech@tecnotex.it

<sup>2</sup> Technical University of Berlin (GERMANY)

<sup>3</sup> Flemish Institute for Technological Research, MoI (BELGIUM)

<sup>4</sup> A3 Water Solutions GmbH, Gelsenkirchen (GERMANY)

<sup>5</sup> Berlin Centre of Competence Water (GERMANY)



## NEXT Technology Tecnotessile

- ▶ Research Centre
- ▶ Location: Prato (Italy)



- ▶ Main Activities:
  - Design and development of applied research activities
  - Provision of services concerning chemical testing activities
  - Provision of consulting services for Quality System development
  - Provision of services of training courses for the textile sector

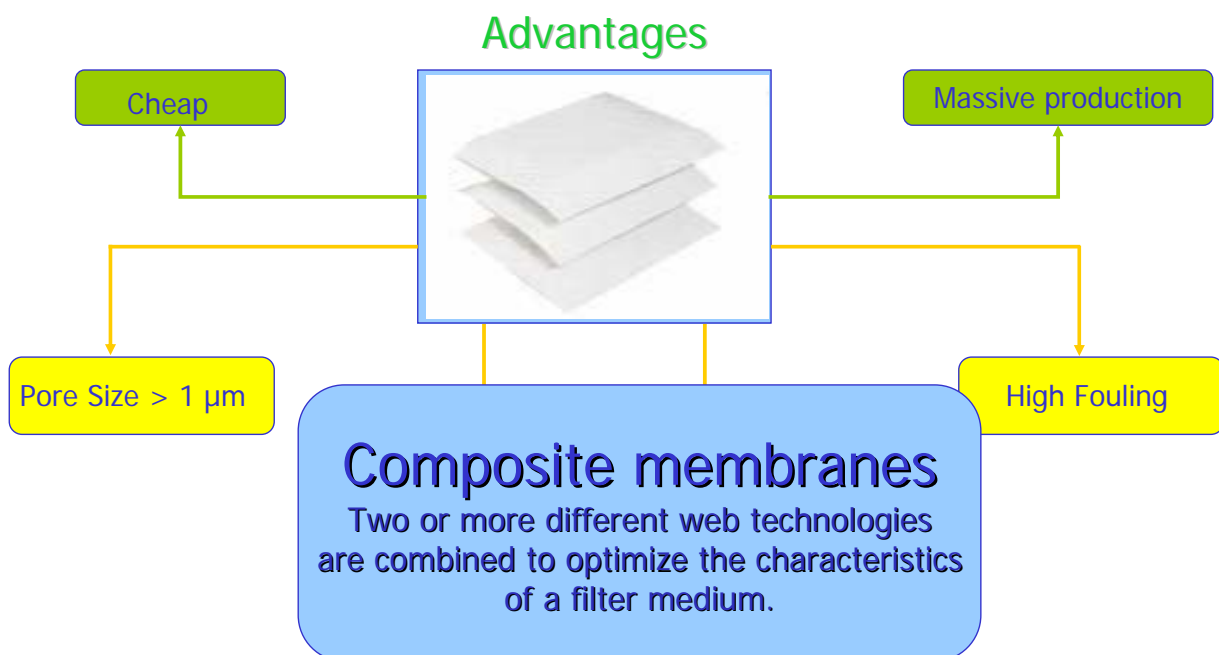
## Nonwoven Filters VS Conventional Membranes



- ▶ Lower Costs
- ▶ Lower Filtration Resistance
- ▶ Higher Flux
- ▶ Larger Pore Size
- ▶ Less Effective in the Removal of total Coliforms
- ▶ No Optimised Conditions are available

Chang et al, Desalination 202 (2007) 122-128

## Commercial Nonwoven Filter in MBR



## Composite textile membrane in MBR

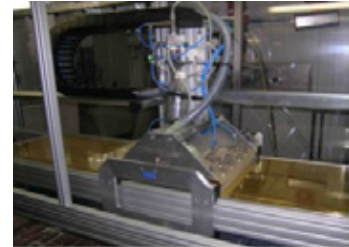
Nonwoven Support

Electrospun Nylon 6 nanofiber

Plasma Grafting



Tri-components composite  
Commercial nonwoven support



Most suitable conditions

- ▶ Nylon 6 concentration: 18%wt.
- ▶ Distance between the electrode: 9 cm
- ▶ Voltage: 30 kV
- ▶ Flow rate: 0.25 ml/min
- ▶ Syringe distance: 24.0 mm
- ▶ Drum speed: 0.23 m/min

Most suitable conditions

- ▶ High power input: 400 W
- ▶ Low discharge frequency: 1.5 kHz
- ▶ Treatment time: 6 passes at 4m/min

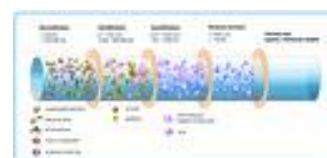
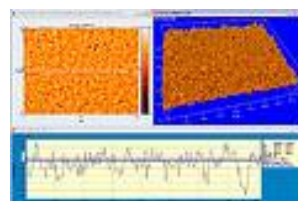
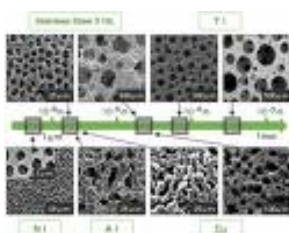
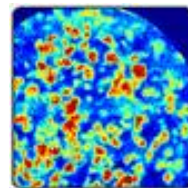
Selected precursor:

- ▶ Allylamine

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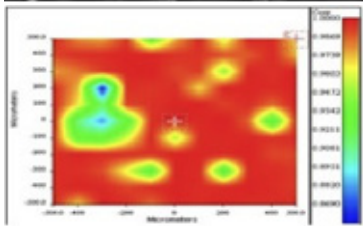
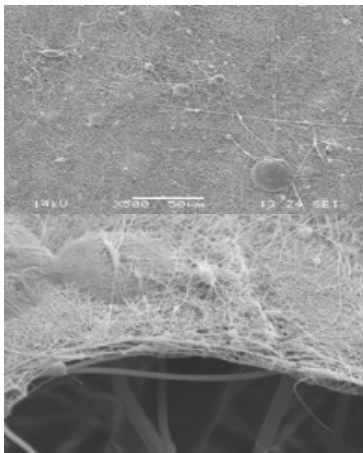
## Composite textile membrane Characterisation

- ▶ SEM analysis
- ▶ FT-IR analyses including Chemical Imaging
- ▶ Capillary flow porometry
- ▶ Tensile Strength measurement
- ▶ Topographic analyses performed with Altisurf equipment
- ▶ Contact angle measurements
- ▶ Ageing tests in alkali (pH 12) and acid (pH 1) solutions
- ▶ Biogrowth test
- ▶ Filterability test

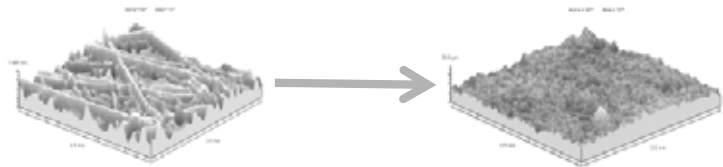


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## Composite textile membrane morphology



- ▶ Dense and homogeneous nanoweb  
Nanofiber diameter 200 -250 nm  
Width of the nanoweb 12 – 18 nm
- ▶ Good adhesion of the nanoweb onto textile support
- ▶ Homogeneous distribution of the Allylamine onto the nanoweb
- ▶ Reduction of the roughness to the value observed for the conventional membranes



## Composite textile membrane – DATA Sheet

Type	Novatexx2471	Composite	Conventional
Material	PP	Nylon 6	PVDF
Pore Size	10 $\mu\text{m}$	0.71 $\mu\text{m}$	0.29 $\mu\text{m}$
Thickness	0.18 mm	0.20 mm	0.20 mm
Tensile Strength <sup>°</sup>	15 MPa	15 MPa	21 MPa
Elongation	25%	26%	20%
Critical Flux	25 L/m <sup>3</sup> h	200 L/m <sup>3</sup> h	120 L/m <sup>3</sup> h
Biofilm Growth*	3.4 %wt.	0.9 %wt.	0.6 %wt.
Alkali resistance (pH = 10)			
Acid resistance (pH = 2)			

<sup>°</sup> standart test ASTM D638

\* After exposure to 0.5 g/L yeast solution at 35°C for 48 h

## Composite textile membrane – DATA Sheet

Type	Novatexx2413	Composite	Conventional
Material	PET	Nylon 6	PVDF
Pore Size	15 µm	0.85 µm	0.29 µm
Thickness	0.19 mm	0.21 mm	0.20 mm
Tensile Strength°	17 MPa	17 MPa	21 MPa
Elongation	25%	26%	20%
Critical Flux	25 L/m <sup>3</sup> h	115 L/m <sup>3</sup> h	120 L/m <sup>3</sup> h
Biofilm Growth*	4.4 %wt.	1.0 %wt.	0.6 %wt.
Alkali resistance (pH = 10)			
Acid resistance (pH = 2)			

° standart test ASTM D638

\* After exposure to 0.5 g/L yeast solution at 35°C for 48 h

## Composite textile membrane – Module



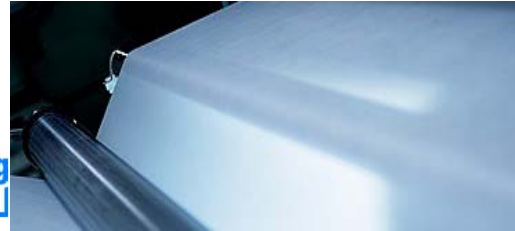
### TBR module

Number of Plate per unit	12
Membrane per plate	2
Membrane dimension	0.10 x 0.20 m <sup>2</sup>
Total filtration area	0.48 m <sup>2</sup>

## Industrial production of Composite Membrane

<b>Expenditure</b>	<b>Cost</b>	<b>Cost (€/m<sup>2</sup>)</b>
<i>Material</i>	-	0.024
Nylon 6	2.00 €/kg (UFI)	0.007
Formic Acid	0.65 €/kg (BASF)	0.017
<i>Support</i>	3.87 €/m <sup>2</sup> (Freudenberg)	3.870
<i>Investment Costs*</i>	600000 € (ELMARCO)	1.151
<i>Energy Costs</i>	0.13 €/kWh (ENEL)	0.044
<b>TOTAL</b>	-	<b>5.132</b>
<b>Conventional</b>		<b>14.00</b>

\* 5 year depreciation



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## Conclusions

- ▶ Nanocomposite membrane seems to be a good option for the production of cheap microfiltration membranes
- ▶ Good filtration performance seems to be ensured even if further investigations are needed
- ▶ A wide range of polymer can be used for the production of the nanofibrous layers – such as PC (already processed); PUR; Fluoropolymers ;PESO; etc.

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## Acknowledgement

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