LIFE DeNTreat

Decentralized innovative treatment of ammonium-rich urban wastewater

Lab scale activities

Simone Visigalli – Micol Bellucci

Politecnico di Milano

LIFE DeNTreat FINAL EVENT – web meeting

February 24th, 2021

The project has received funding from European Union´s LIFE Programme under Grant Agreement LIFE16 ENV/IT/000345

PN/Anammox process

- ✓ Low O_2 consumption
- ✓ No external carbon dosing
- ✓ Lower sludge production

PN - Partial nitritation:

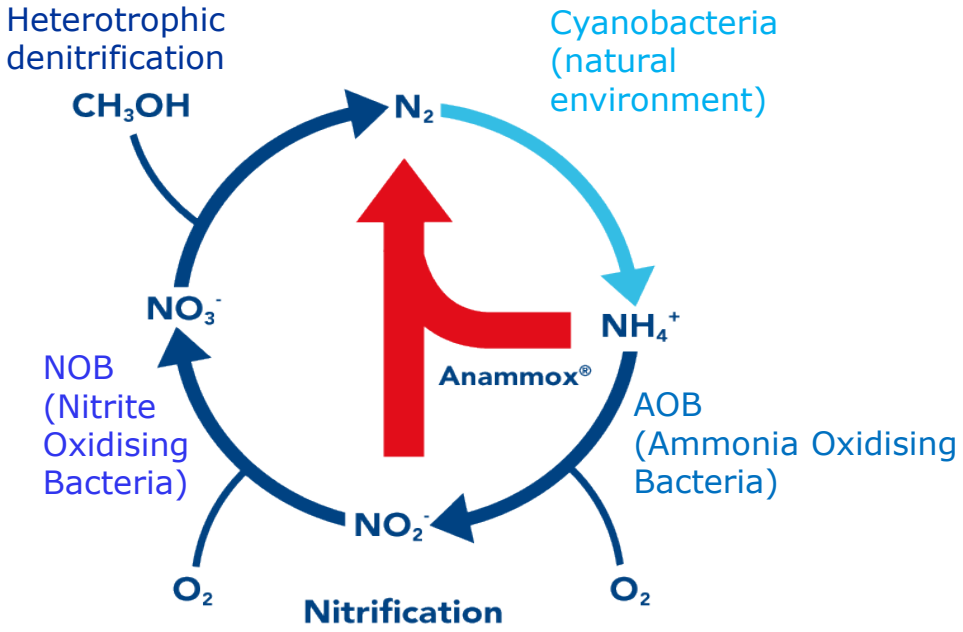
$$2NH_4^+ + 3O_2 \rightarrow 2NO_2^- + 4H^+ + 2H_2O$$

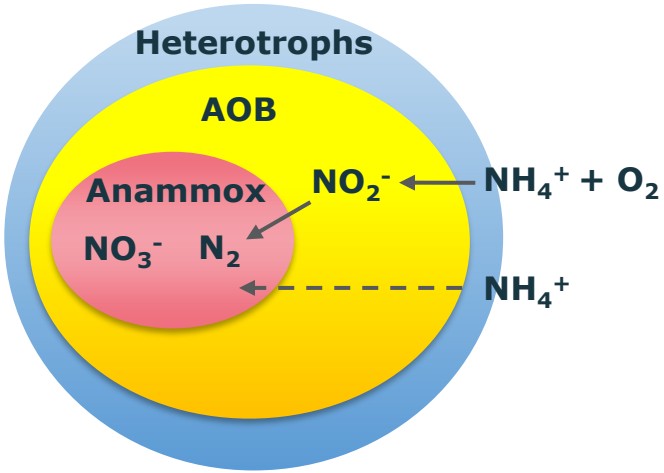
50% of N-NH_4^+ is converted

A - Anammox:

$$NH_4^+ + 1.32NO_2^- + 0.066HCO_3^- + 0.13H^+ \rightarrow 1.02N_2 + 0.26NO_3^- + 0.066CH_2O_{0.5}N_{0.15} + 2.03H_2O$$

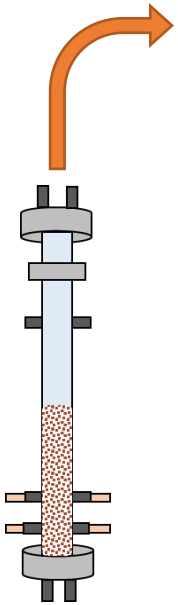
Max conversion efficiency = **88,8%**

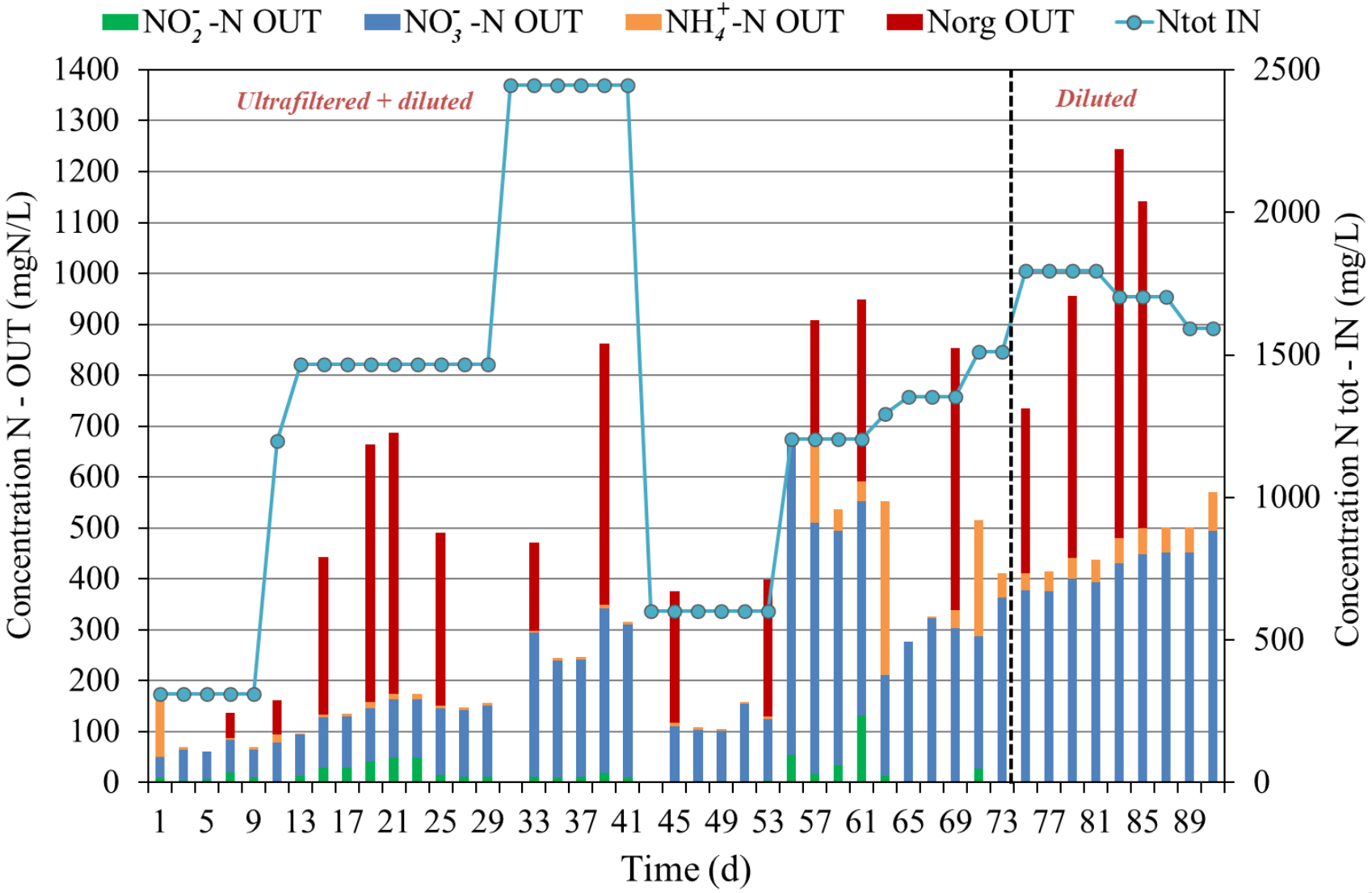




GRANULE STRUCTURE

Background from preliminary tests





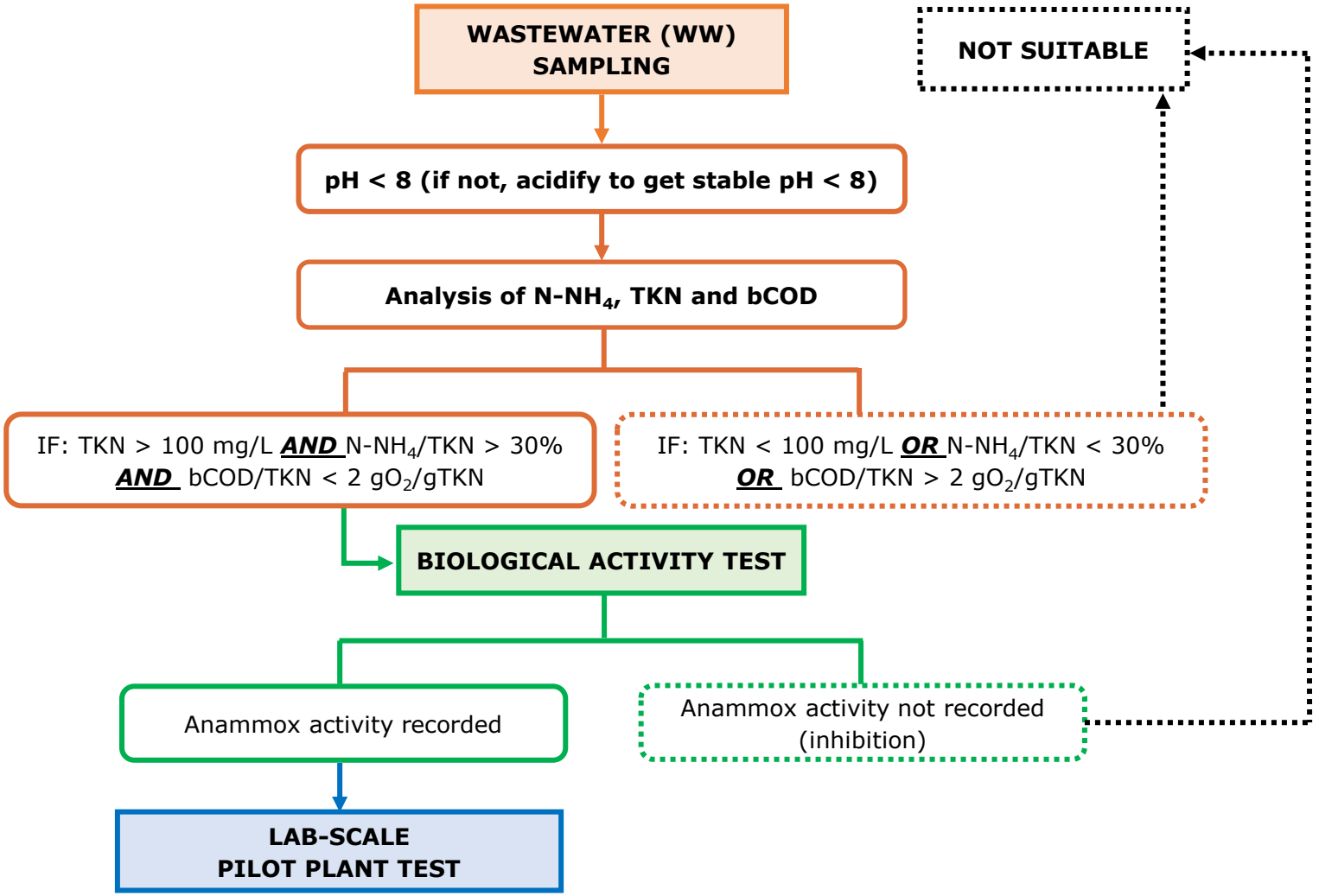
Background from preliminary tests

Concentrated DTP wastewaters

- ✘ Low removal rates if raw wastewater is **undiluted** (inhibiting compounds)
- ✘ Low removal rates if **bCOD / TKN > 2** in the wastewater
- ✘ Low removal rates if **NH_4^+-N / TKN < 30%** in the wastewater
- ✘ At low NH_4^+-N / TKN ratios, **urea conversion to NH_4^+-N** should be promoted
- ✘ **Ureolysis** is the kinetically limiting step: slow NH_4^+-N production → slow down of AOB activity and NOB may develop

- ✓ **Treatability confirmed on equalized effluents**

Criteria of WW suitability for PN/A



Wastewater samples

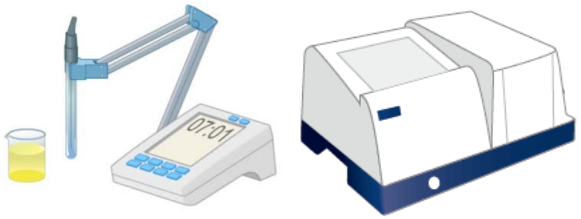
WW 1
Effluent from viscose process

WW 2
Final effluent

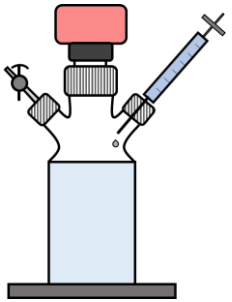
WW 3
Final effluent

WW 4
Final effluent (equalized)

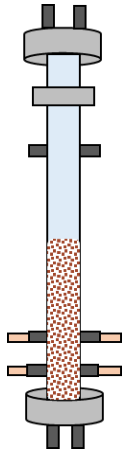
WW 5
Final effluent (equalized)



Physicochemical characterization



Manometric batch tests



Lab scale pilot tests

LIFE DeNTreat

Physicochemical characterization & manometric batch tests

Physicochemical characterization

	WW 1	WW 2	WW 3	WW 4	WW 5	
pH	8.8	8.9	9.2	9.2	7.9	< 8
Conductivity (mS/cm)	1.54	0.30	2.41	2.43	1.00	
COD (mg/L)	380	390	356	971	677	
TN (mg/L)	556	527	238	336	217	
TKN (mg/L)	551	526	224	335	216	> 100 mg/L
NH_4-N (mg/L)	41	41	165	17	182	
NO_3-N (mg/L)	4.99	1.36	13.8	1.16	0.78	
NO_2-N (mg/L)	0	0	0.13	0.27	0	
NH_4-N/TKN (%)	7.4	7.8	73.7	5.1	84.3	> 30%
COD/TKN (gO_2/gTKN)	0.69	0.74	1.59	2.90	3.13	
PO_4-P (mg/L)	97.2	2.5	4.4	4.2	5.8	
TSS (mg/L)	70.0	33.3	NA	200.0	186.7	
VSS (mg/L)	26.7	33.3	NA	NA	170.0	

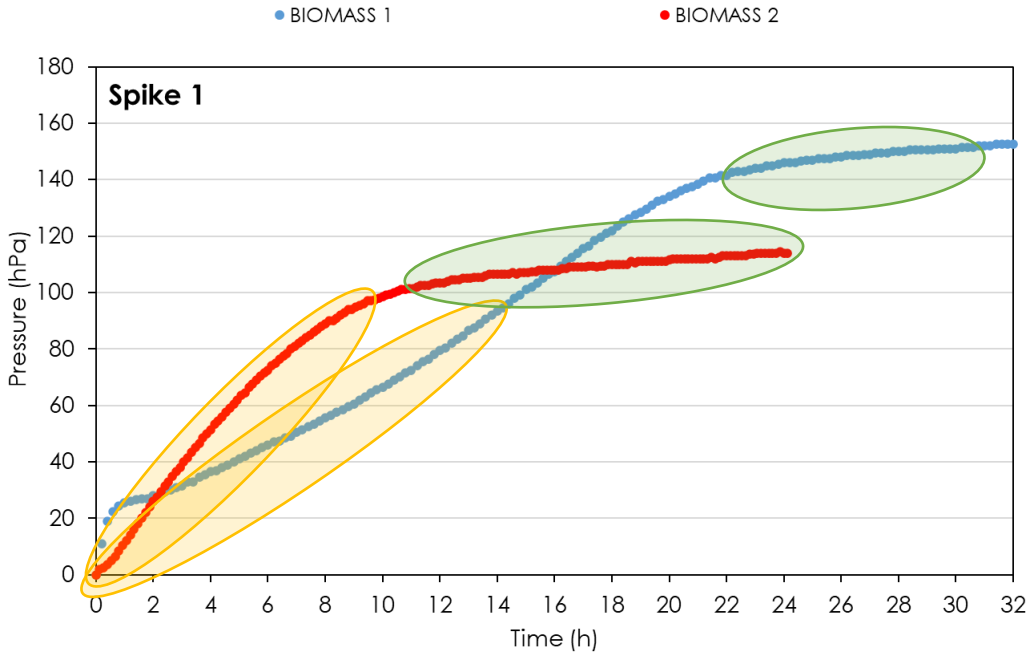
Manometric batch tests

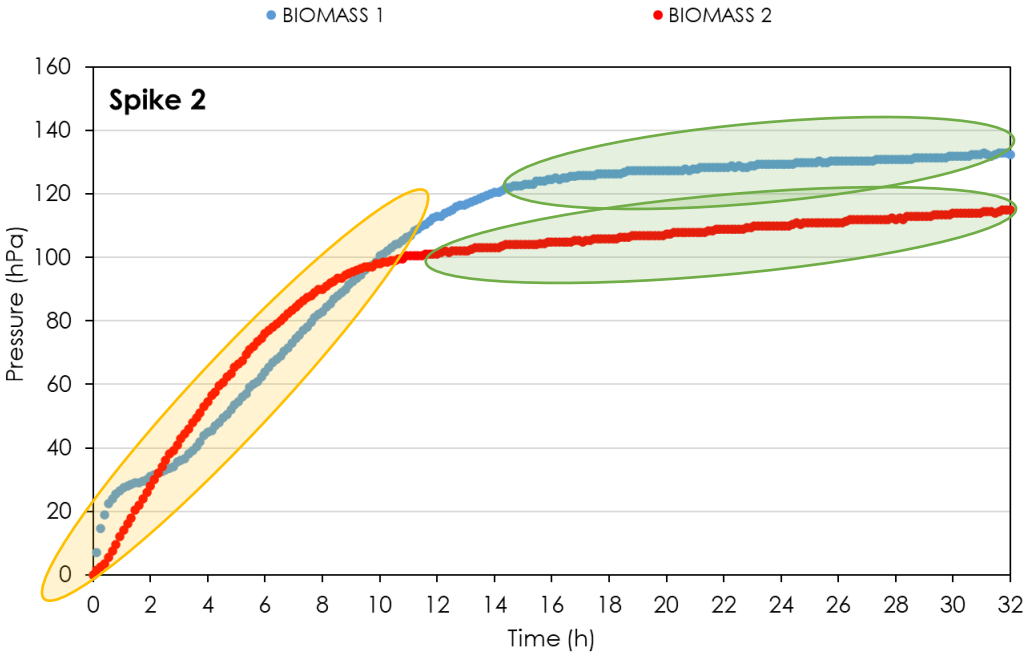
Activity test evaluates the capability and the rate at which the Anammox granules are removing NH_4 and NO_2 from the solution by measuring the pressure increase due to the production of N_2.

DO = 0,0 ppm

The activity test in presence of a wastewater allows evaluating the ability of Anammox biomass to operate in presence of that specific substrate.

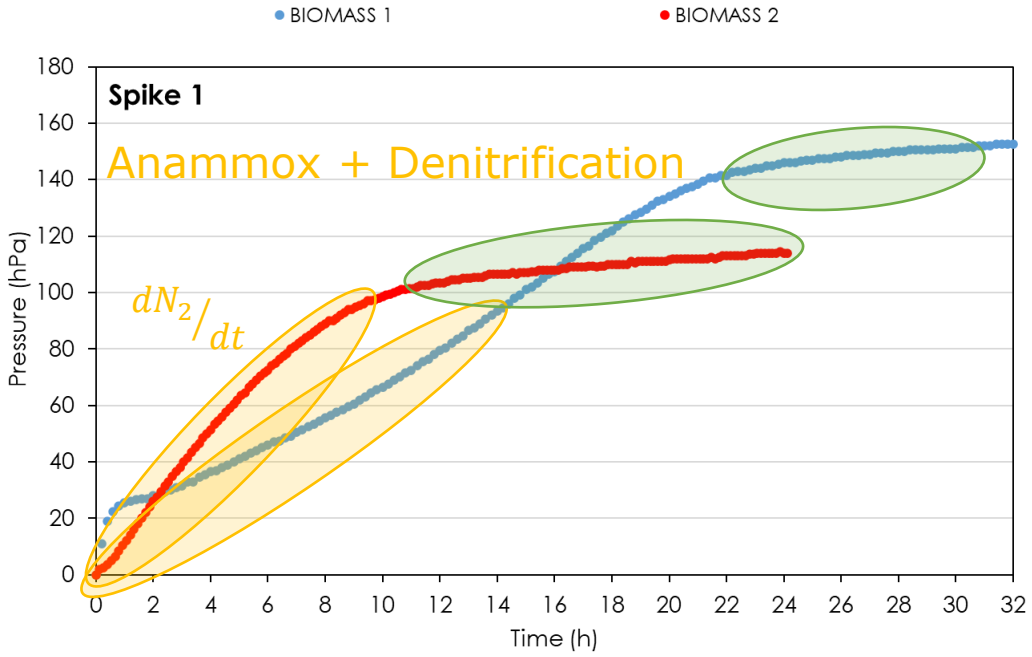
Manometric batch tests

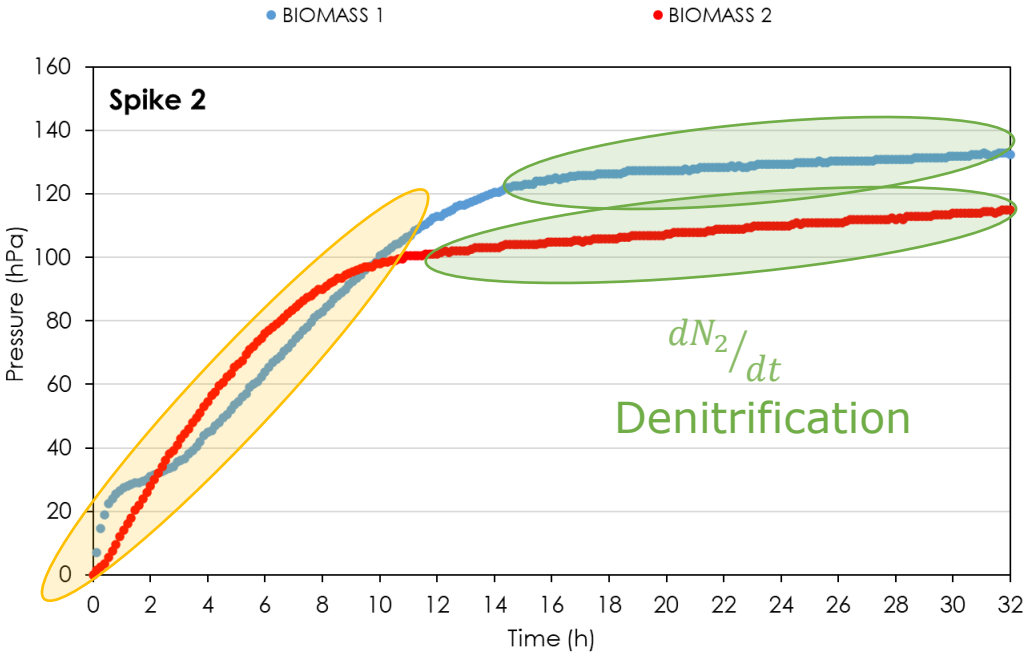




The **steepest** part of the curve is known to be addressable to the activity of both Anammox and denitrification, while the **less steep** part can be addressed to the denitrification activity only. At the net of the contribution of denitrifiers, it is possible to assess the sole activity of Anammox biomass in controlled conditions.

Manometric batch tests



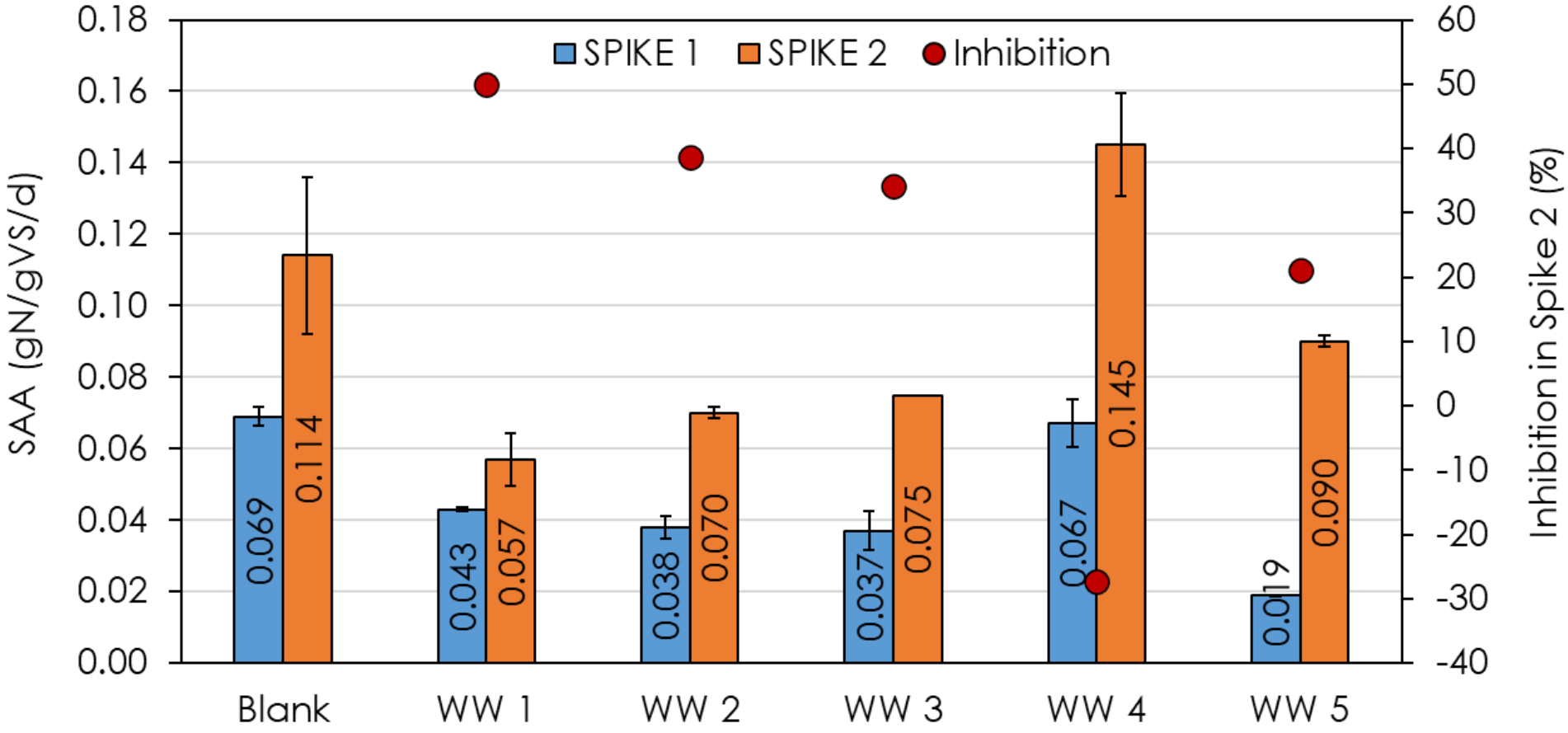


$$n_{N_2}(t) = \frac{P(t) \times V_{HS}}{R \times T}$$

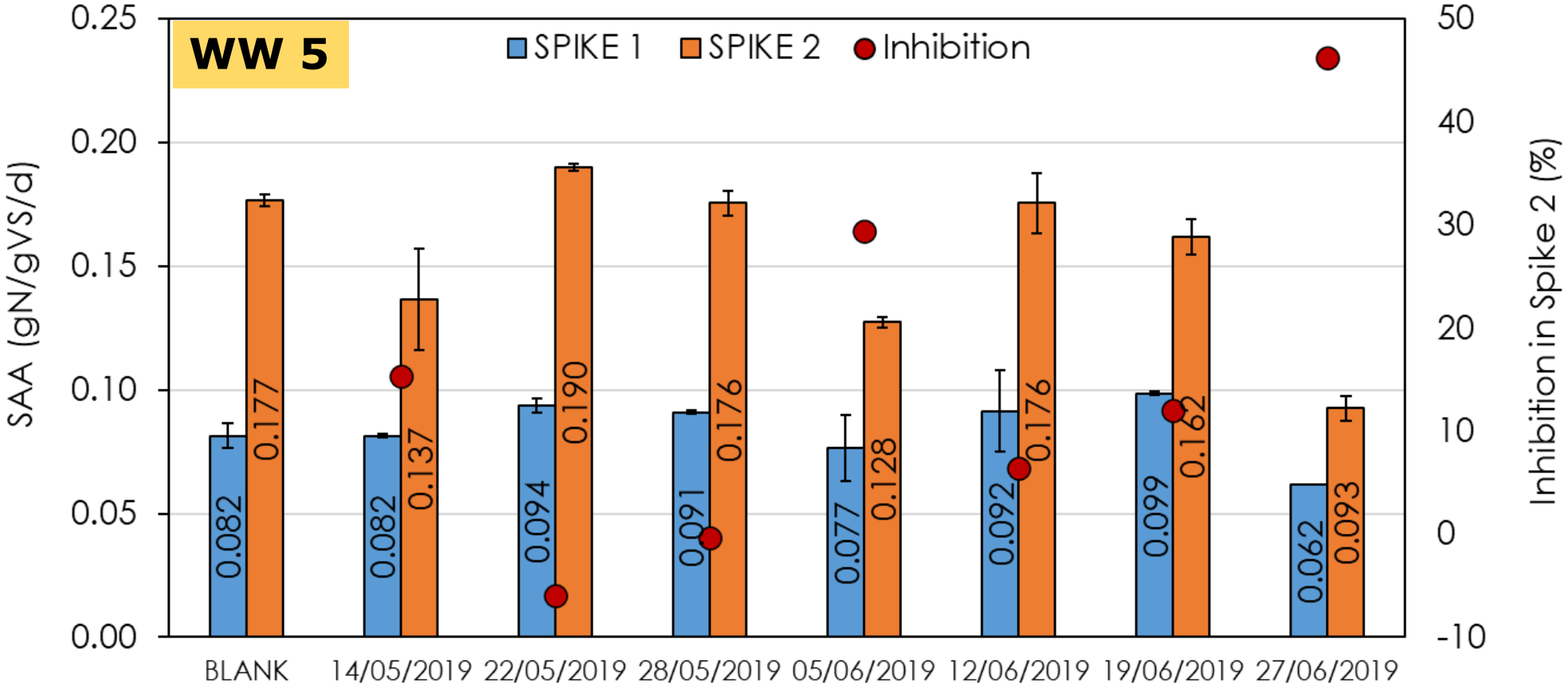
$$\left(\frac{dN_2}{dt}\right)_{AMX} = \left(\frac{dN_2}{dt}\right) - \left(\frac{dN_2}{dt}\right)$$

$$SAA = \left(\frac{\left(\frac{dN_2}{dt}\right)_{AMX} \times 1.32 \times 14}{1.02} \times \frac{1}{gVS} \times \frac{24\ h}{d}\right) + \left(\frac{\left(\frac{dN_2}{dt}\right)_{AMX} \times 1 \times 14}{1.02} \times \frac{1}{gVS} \times \frac{24\ h}{d}\right)$$

Manometric batch tests



Manometric batch tests



Manometric batch tests – Main results

- **Initial adaptation** of the biomass to the wastewater **is needed** to increase the SAA.
- **Inhibition** was always **lower than 50%** with respect to the synthetic solution.
- Some wastewaters may show a **higher SAA than the synthetic solution**.
- **WW 4**, in spite of a high concentration of organic nitrogen, could reach a SAA of 0,145 gN/gVS/d.
- The **quality of the discharge** from the DTP companies **varies in time**: mild to strong inhibiting components could be present in some of the discharges that can significantly reduce the SAA.

Lab scale pilot tests

Wastewater samples

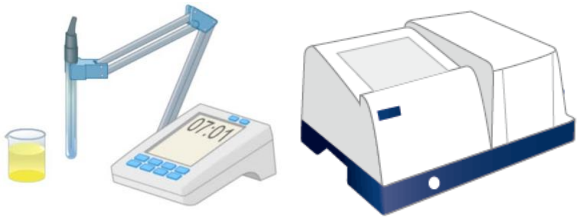
WW 1
Effluent from viscose process

WW 2
Final effluent

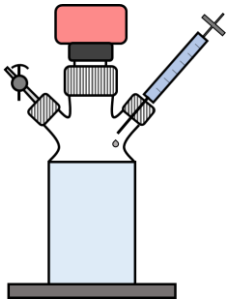
WW 3
Final effluent

WW 4
Final effluent (equalized)

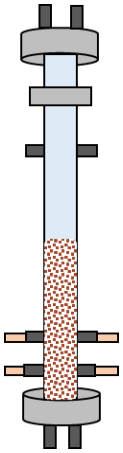
WW 5
Final effluent (equalized)



Physicochemical characterization



Manometric batch tests



Lab scale pilot tests

Wastewater samples

WW 1
Effluent from viscose process

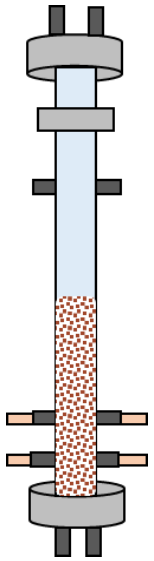
WW 2
Final effluent

WW 3
Final effluent

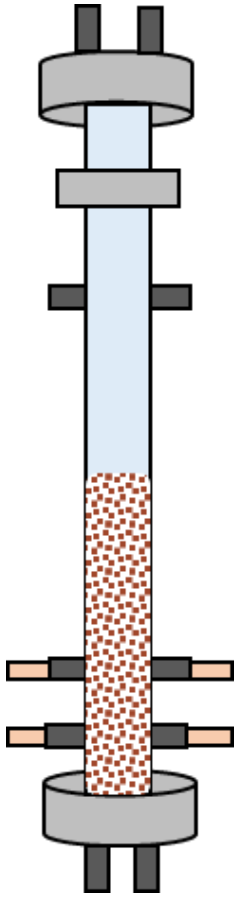
WW 4
Final effluent (equalized)

WW 5
Final effluent (equalized)

Quick clinic tests (7 days)



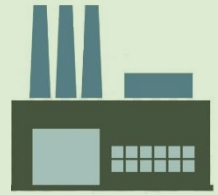
Wastewater samples



Phase 1 → 145 days
Investigation of the treatability of WW 5 (inhibitory effects)

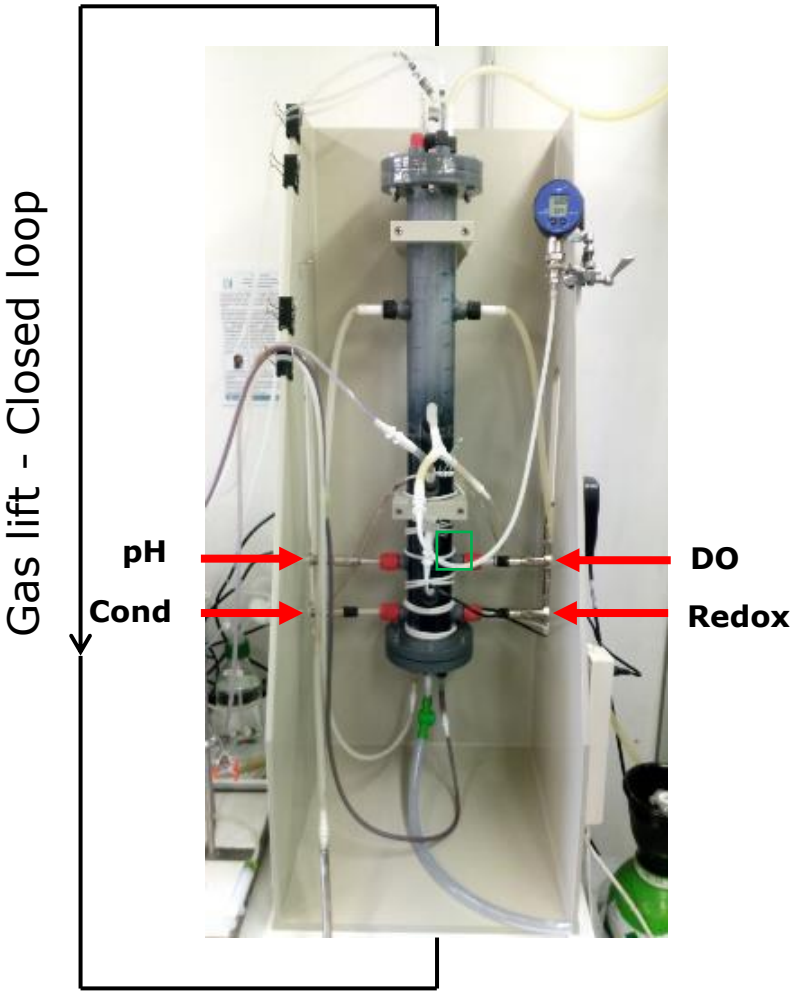
Phase 2 → 175 days
Optimization of the operating conditions to increase NLR, NRR and N-removal efficiency

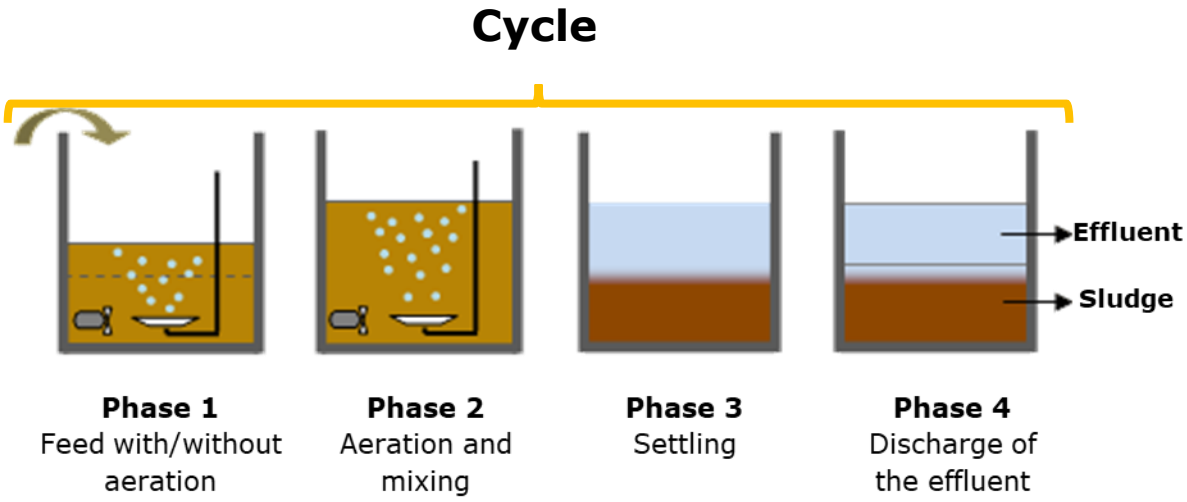
Steady state conditions?



WW 5
Final effluent (equalized)

The lab-scale pilot





Temperature: 31 - 33 °C

Working volume: 2 L

Volume exchanged per cycle: 0.5 L

Biomass concentration: 8 gVS/L

Fixed operating conditions

Cycle duration: 3-6 hours

pH: 7.2 - 7.9

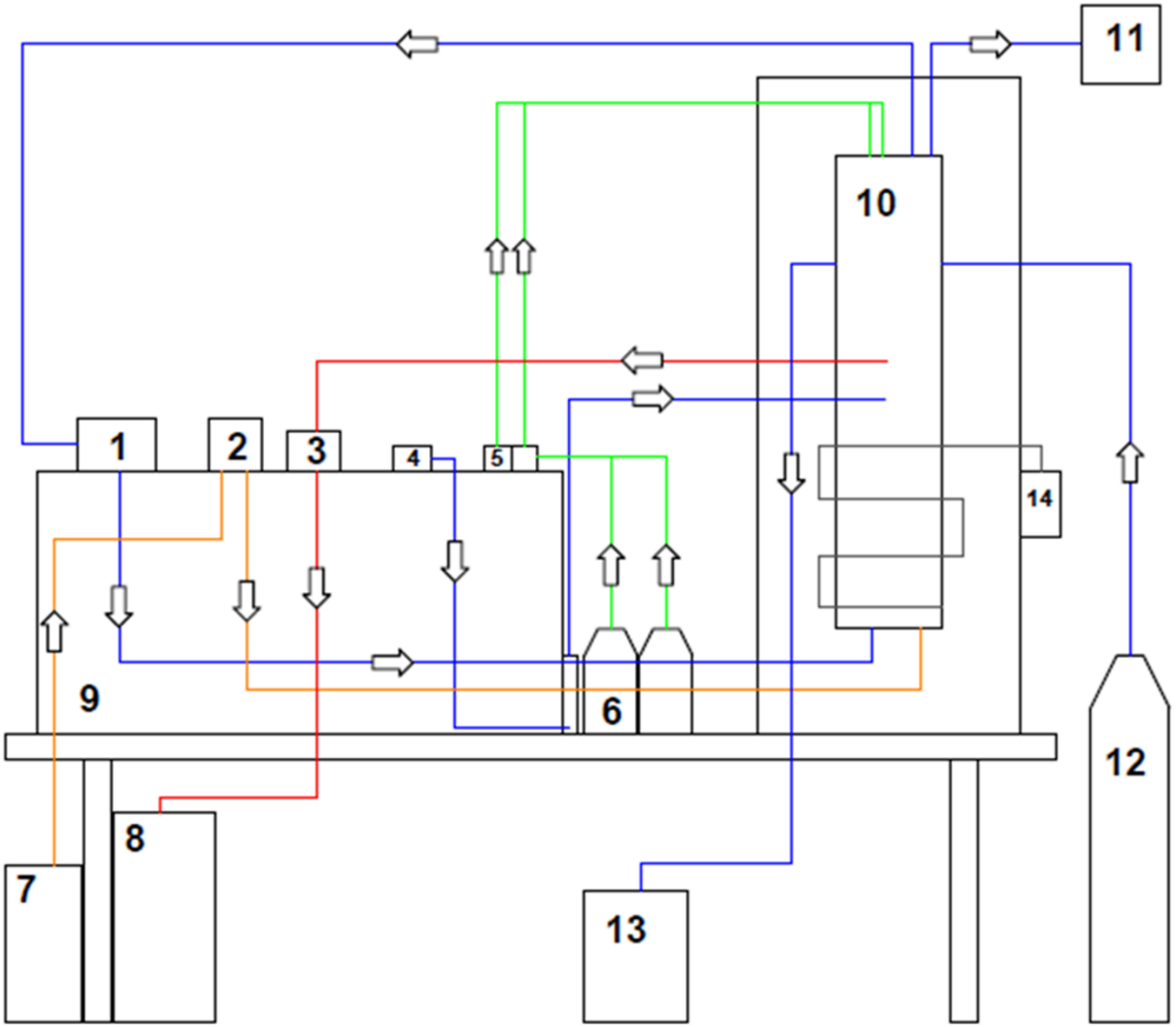
DO: 0.2 – 1.0 ppm

Variable operating conditions

The lab-scale pilot

1: Air recirculation compressor
2: Feed pump
3: Discharge pump
4: Air compressor
5: Acid-base dosing pumps
6: Acid-base tanks
7: Feed tank
8: Discharge tank
9: PLC
10: SBR reactor
11: Gas bag
12: Azomix tank
13: Overpressure drain tank
14: Thermostat

Gas
Feed
Discharge
Acid-base



Quick clinic test - WW 1

Cycle duration: 6 - 8 hours **pH: 7.2 - 7.6** **DO: 0.2 - 0.4 ppm**

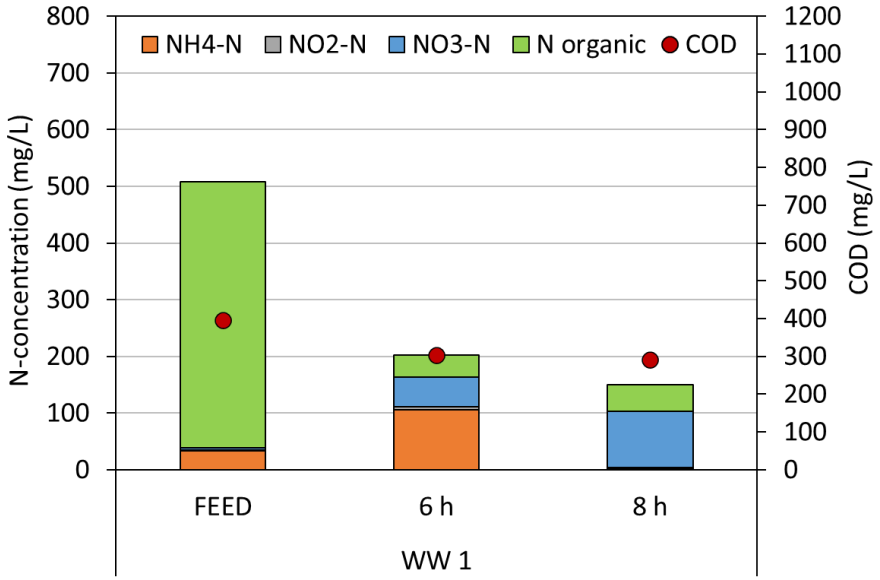
		NO_2-N	NO_3-N	NH_4-N	Organic N	TOT N	COD
INFLUENT	50% WW 1	8.3	6.0	77.2	261	352	274
	100% WW 1	**0.9**	**4.9**	**33.4**	**469**	**508**	**395**
EFFLUENT	50% WW 1 (6h)	1.9	27.9	138	82	250	267
	100% WW 1 (6h)	**4.7**	**52.6**	**106**	**39**	**202**	**304**
	100% WW 1 (8h)	**1.3**	**98.5**	**2.7**	**48**	**151**	**291**

50% WW 1 (6h): N-removal efficiency up to 28%

100% WW 1 (6h): N-removal efficiency up to 60%

100% WW 1 (8h): N-removal efficiency up to 70%

A 10% fraction of organic N is not ammonified and looks like it is unbiodegradable



Quick clinic test - WW 2

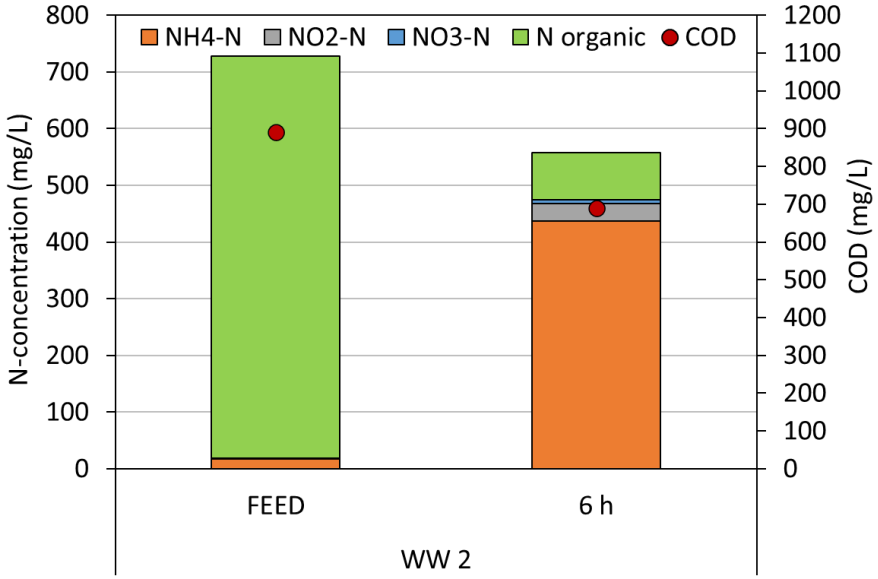
Cycle duration: 6 hours **pH: 7.2 - 7.6** **DO: 0.2 - 0.4 ppm**

		NO_2-N	NO_3-N	NH_4-N	Organic N	TOT N	COD
INFLUENT	50% WW 2	0.50	4.11	103.75	293.64	402.00	487.50
	100% WW 2	**0.00**	**1.34**	**17.50**	**709.16**	**728.00**	**891.00**
EFFLUENT	50% WW 2	0.75	19.46	91.55	31.74	143.50	278.00
	100% WW 2	**30.06**	**7.37**	**437.50**	**83.08**	**558.00**	**690.50**

50% WW 2: N-removal efficiency up to 64%

100% WW 2: N-removal efficiency up to 23%

- Inhibitory action of free ammonia
- Time not sufficient for the conversion of N-NH_4 to N-NO_2



Quick clinic test - WW 3

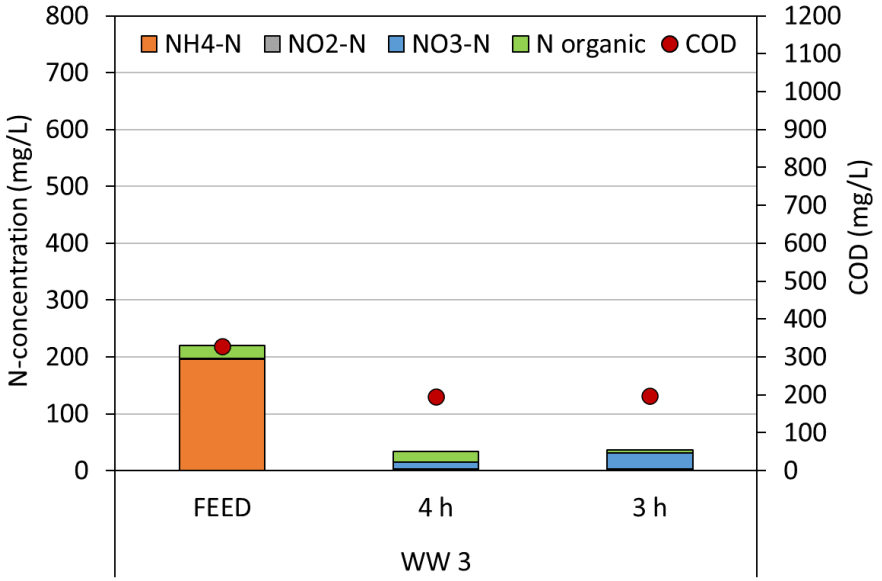
Cycle duration: 4 - 3 hours **pH: 7.6 - 7.8** **DO: 0.4 - 0.7 ppm**

		NO_2-N	NO_3-N	NH_4-N	Organic N	TOT N	COD
INFLUENT	100% WW 3	0.0	0.8	196.0	23.2	220.0	329
EFFLUENT	100% WW 3 (4h)	2.2	12.5	0.5	18.2	33.4	196
	100% WW 3 (3h)	2.7	27.8	0.5	5.2	36.2	198

100% WW 3 (4h): N-removal efficiency up to 84.8%

100% WW 3 (3h): N-removal efficiency up to 83.5%

- No adaptation of the biomass needed;
- N-NH_4^+ was completely removed;
- Loading may be increased further.



Quick clinic test - WW 4

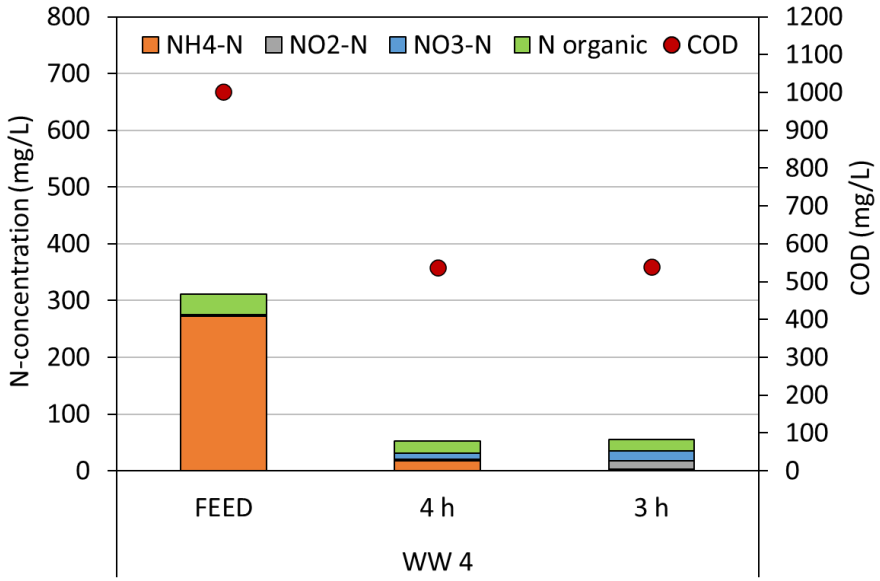
Cycle duration: 4 - 3 hours **pH: 7.6 - 7.8** **DO: 0.4 - 0.7 ppm**

		NO_2-N	NO_3-N	NH_4-N	Organic N	TOT N	COD
INFLUENT	100% WW 4	0.0	2.7	273.0	35.3	311.0	1001
EFFLUENT	100% WW 4 (4h)	3.1	10.7	17.1	21.7	52.6	537
	100% WW 4 (3h)	15.6	16.3	2.7	20.5	55.1	539

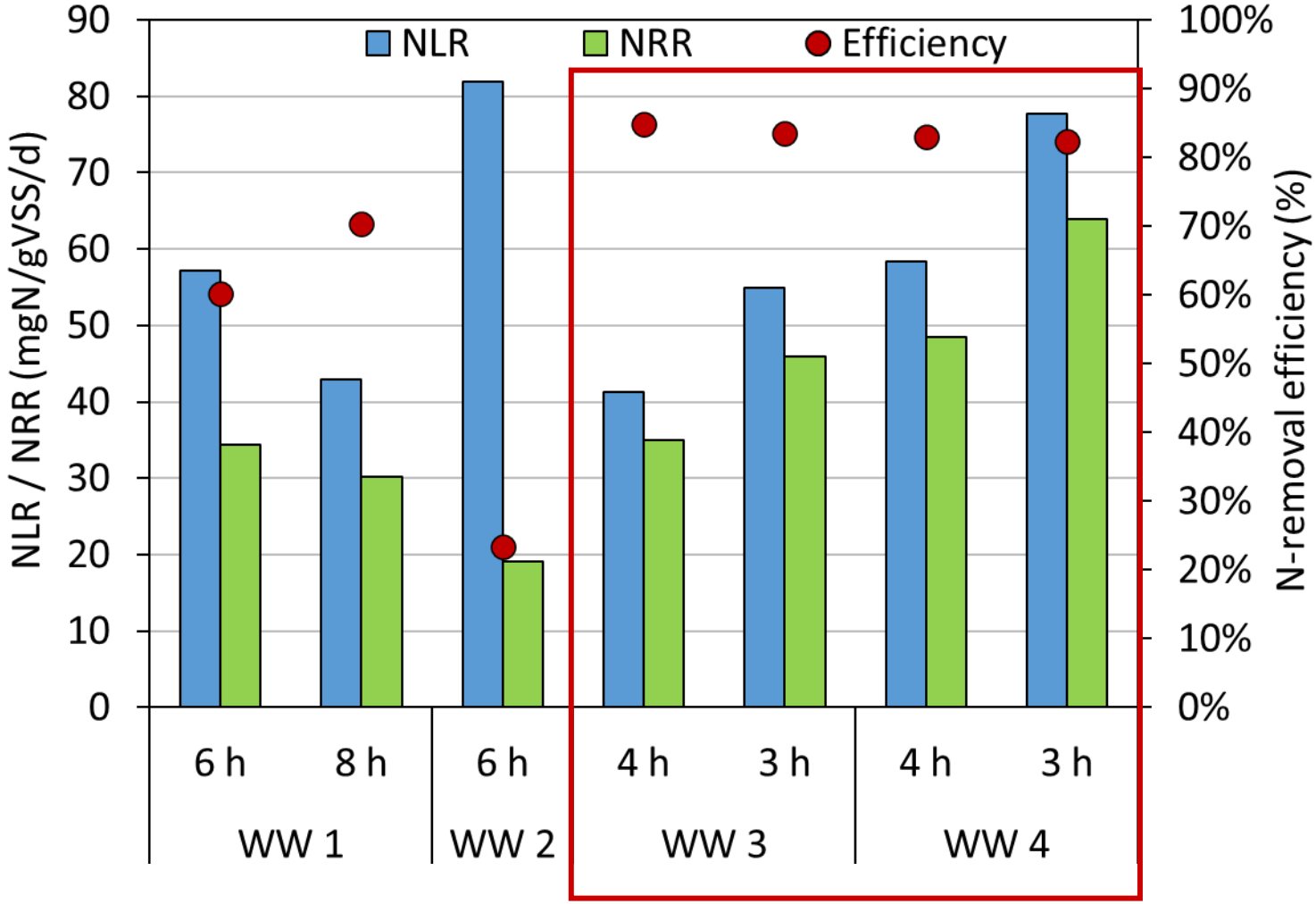
100% WW 4 (4h): N-removal efficiency up to 83.1%

100% WW 4 (3h): N-removal efficiency up to 82.3%

- No adaptation of the biomass needed;
- N-NH_4^+ was completely removed;
- Loading may be increased further.



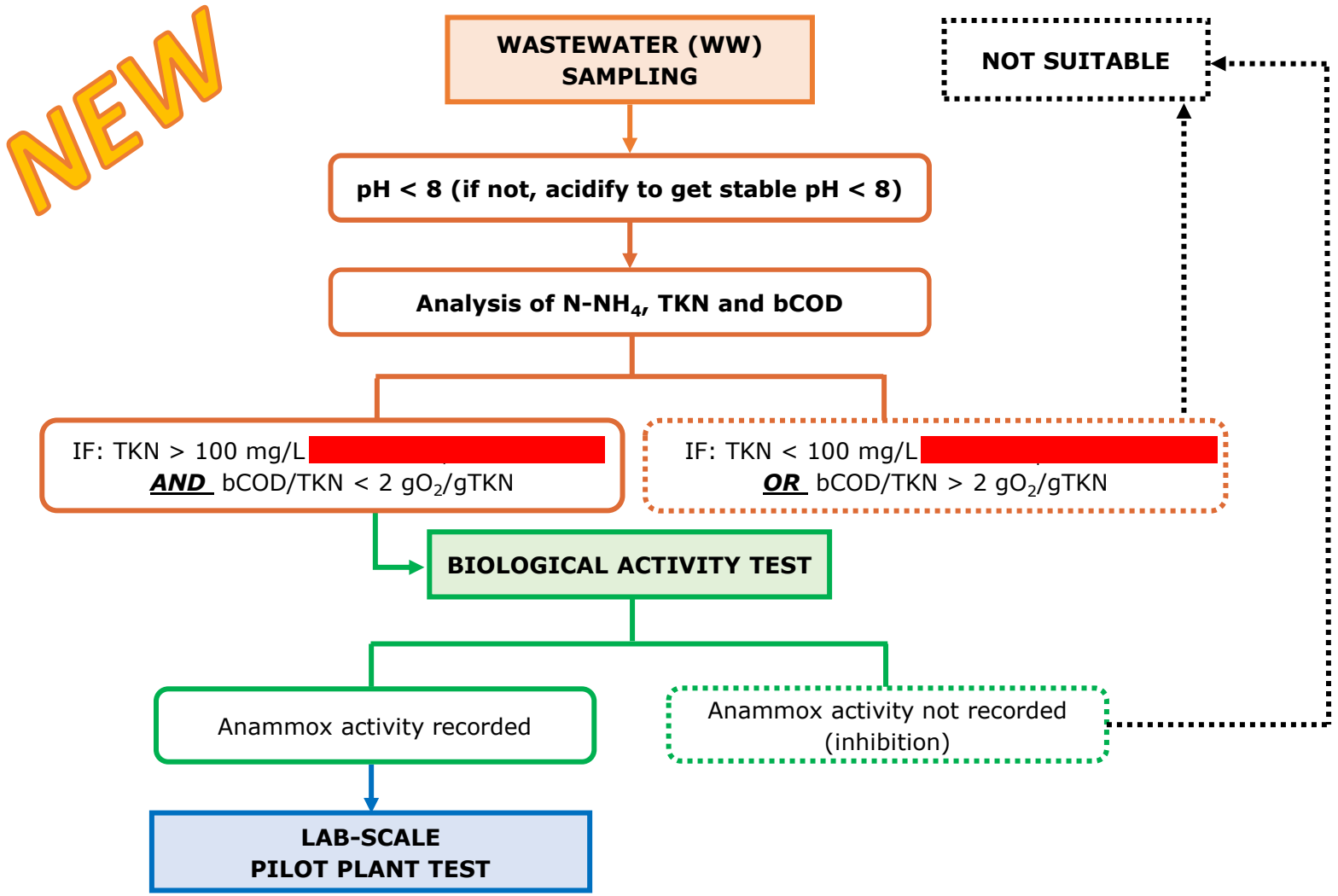
Quick clinic tests - Summary



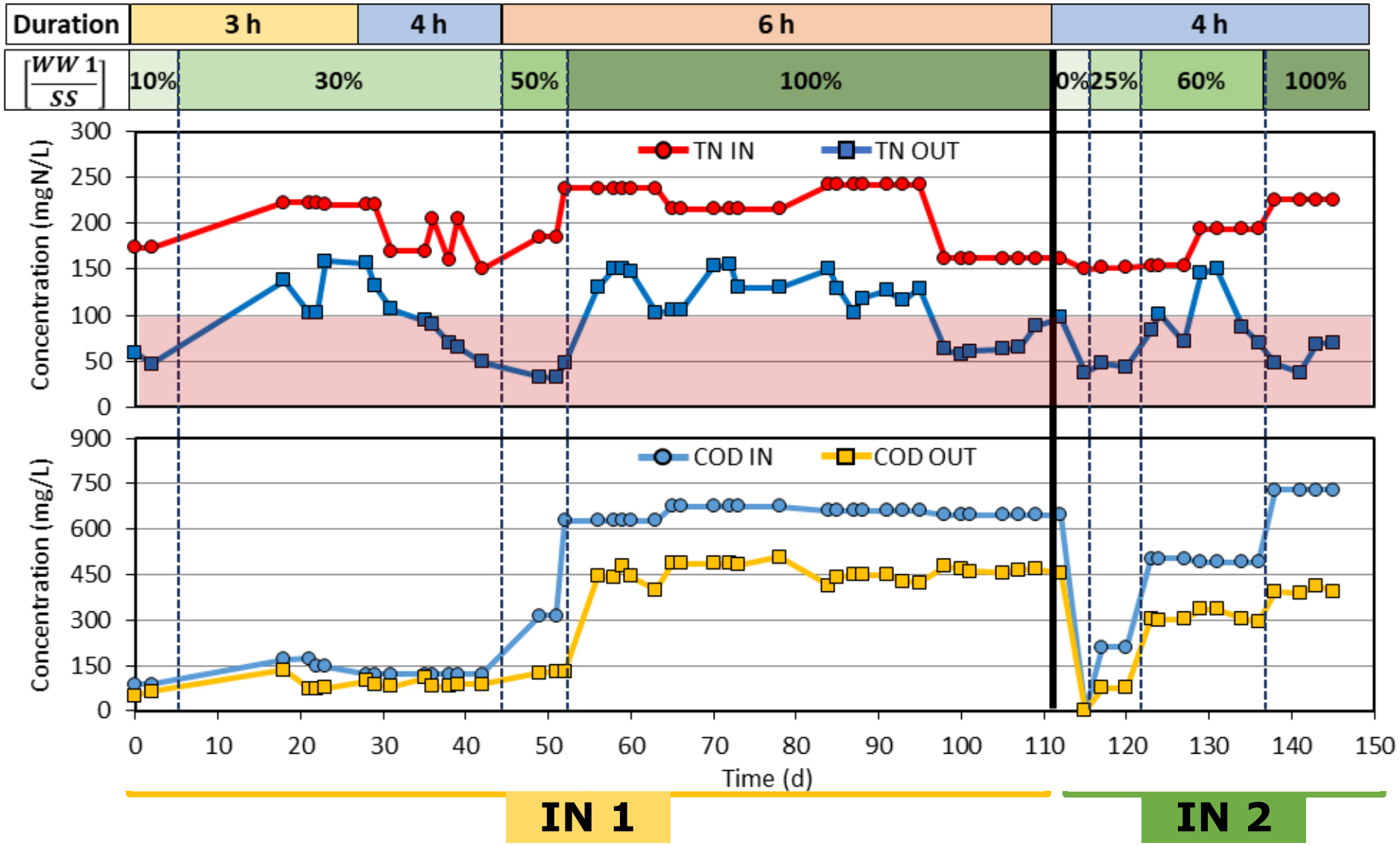
Quick clinic tests – Main results

- For **WW 1** and **WW 2** a large portion of the **organic nitrogen was ammonified**: probably some bacterial populations (*Nitrosococcus spp* and *Nitrosomonas ureae*) convert urea into ammonium which is then removed via PN/A.
- No dilution with synthetic solutions was needed for the quick clinic tests in the lab pilot plant for **WW 3** and **WW 4**.
- Both **WW 3** and **WW 4** showed a N-removal efficiency higher than 80% → no substantial difference in N-removal has been found by reducing the cycles duration from 4 to 3 hours.
- **NLR** values were in the range 41-55 mgN/gVSS/d for **WW 3** and 58-78 mgN/gVSS/d for **WW 4** and may have been increased further.
- The condition "**NH_4-N / TKN > 30%**" is excessively conservative, as the process starts even with very low ratios.
- An efficient **pH control** is needed to avoid excessive accumulation of toxic free ammonia.

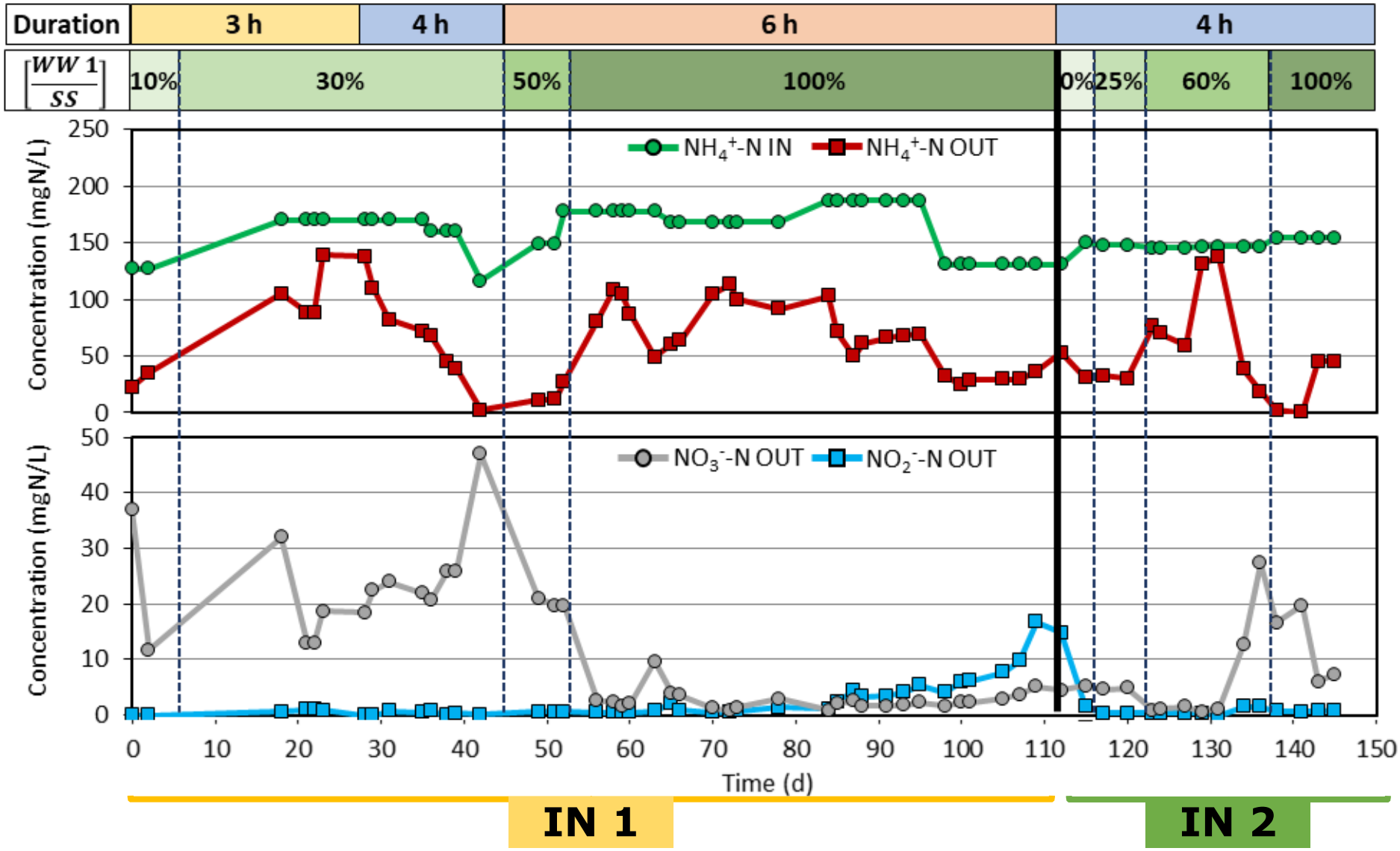
Criteria of WW suitability for PN/A



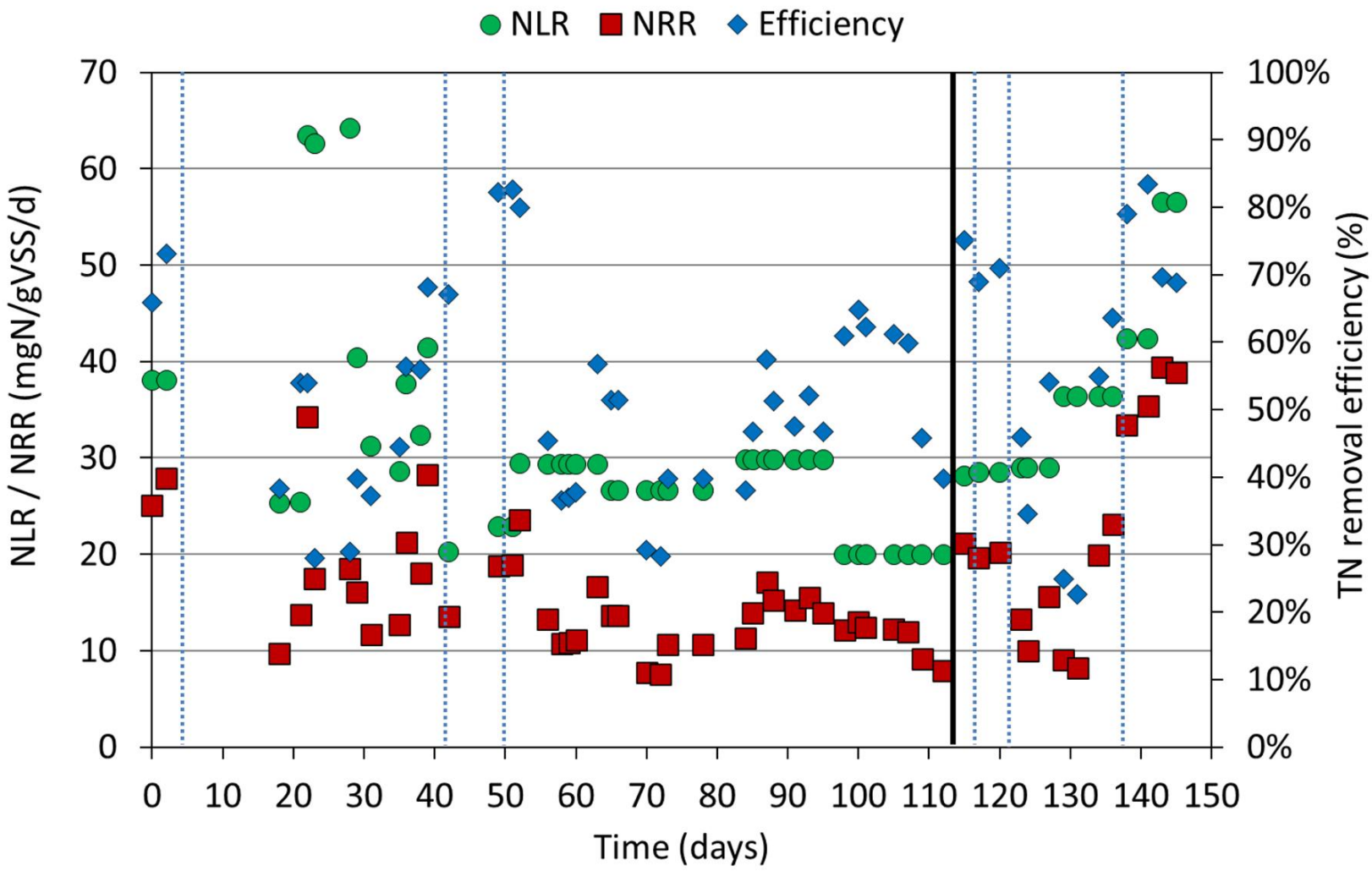
WW 5 – Phase 1


Duration
3 h
4 h
6 h
4 h
[WW 1 / SS]
10%
30%
50%
100%
0%
25%
60%
100%
TN IN
TN OUT
Concentration (mgN/L)
COD IN
COD OUT
Concentration (mg/L)
Time (d)
IN 1
IN 2

WW 5 – Phase 1

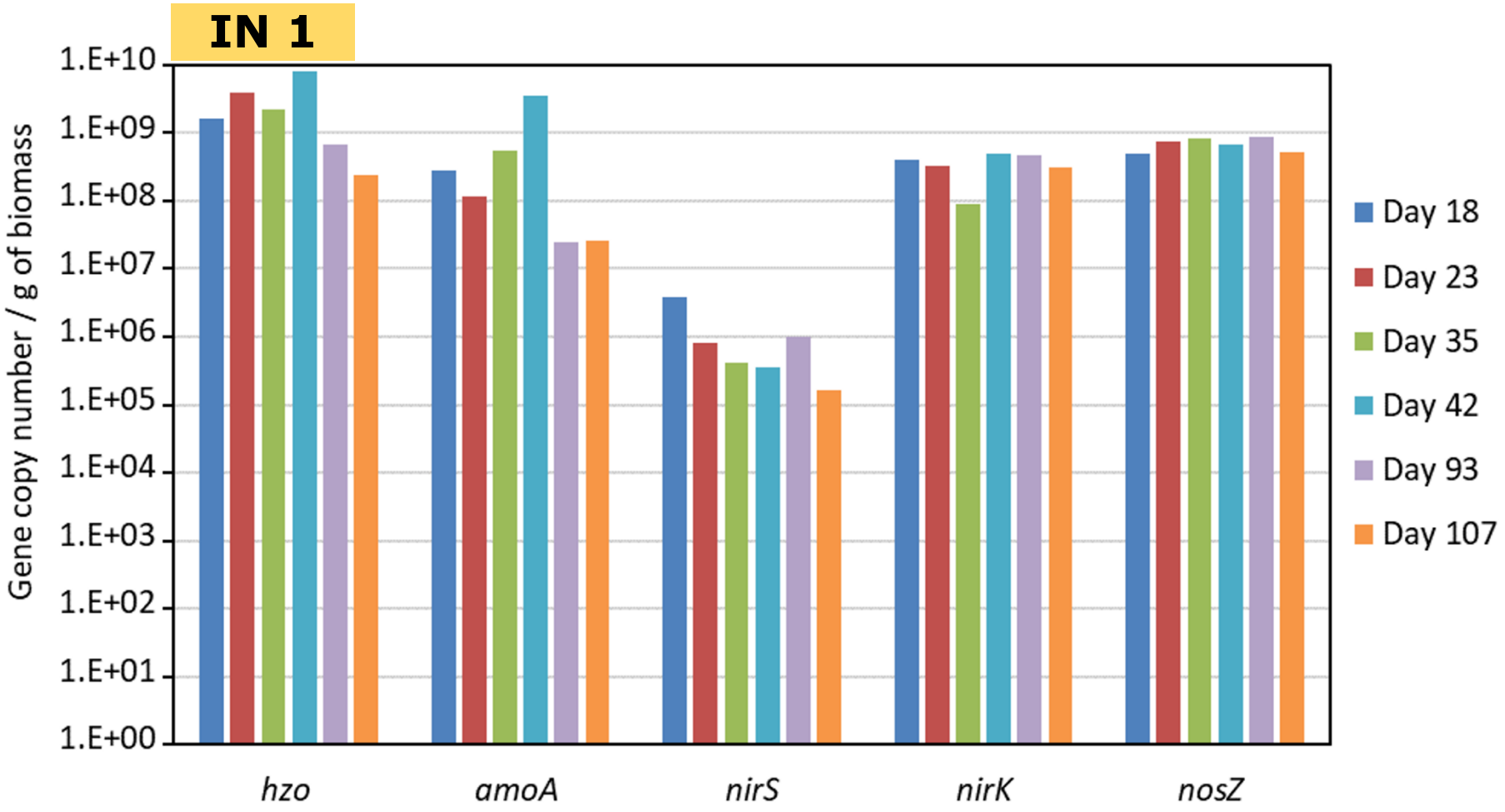

Duration
3 h
4 h
6 h
4 h
[WW 1 / SS]
10%
30%
50%
100%
0%
25%
60%
100%
Concentration (mgN/L)
NH4+-N IN
NH4+-N OUT
NO3--N OUT
NO2--N OUT
Time (d)
IN 1
IN 2

WW 5 – Phase 1



NLR_{avg} = 32.0 mgN/gVSS/d **NRR_{avg} = 16.7 mgN/gVSS/d** **NRE_{avg} = 52.3%**

WW 5 – Phase 1

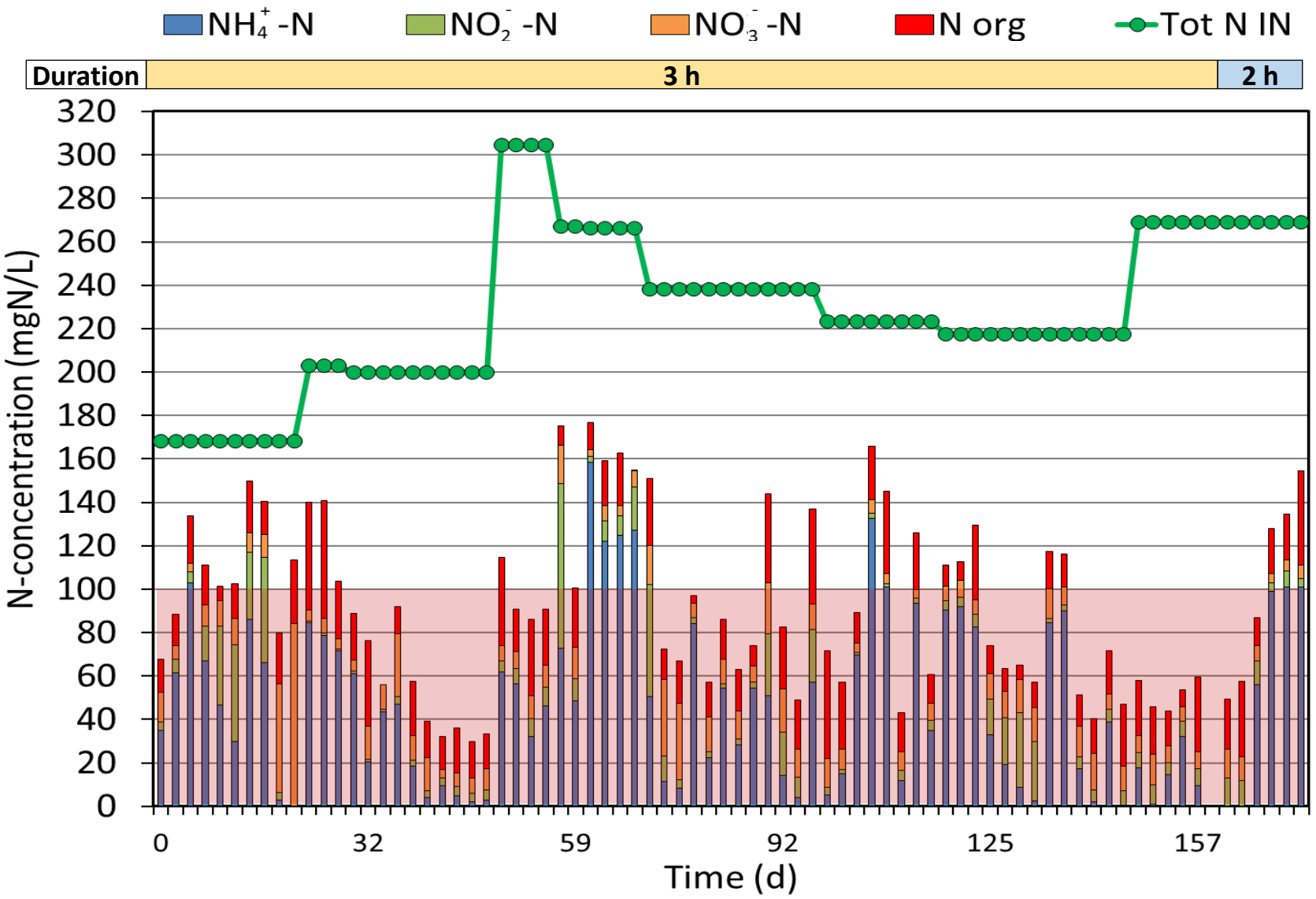

IN 1
Gene copy number / g of biomass
1.E+10
1.E+09
1.E+08
1.E+07
1.E+06
1.E+05
1.E+04
1.E+03
1.E+02
1.E+01
1.E+00
hzo
amoA
nirS
nirK
nosZ
Day 18
Day 23
Day 35
Day 42
Day 93
Day 107

WW 5 – Phase 1 - Main results

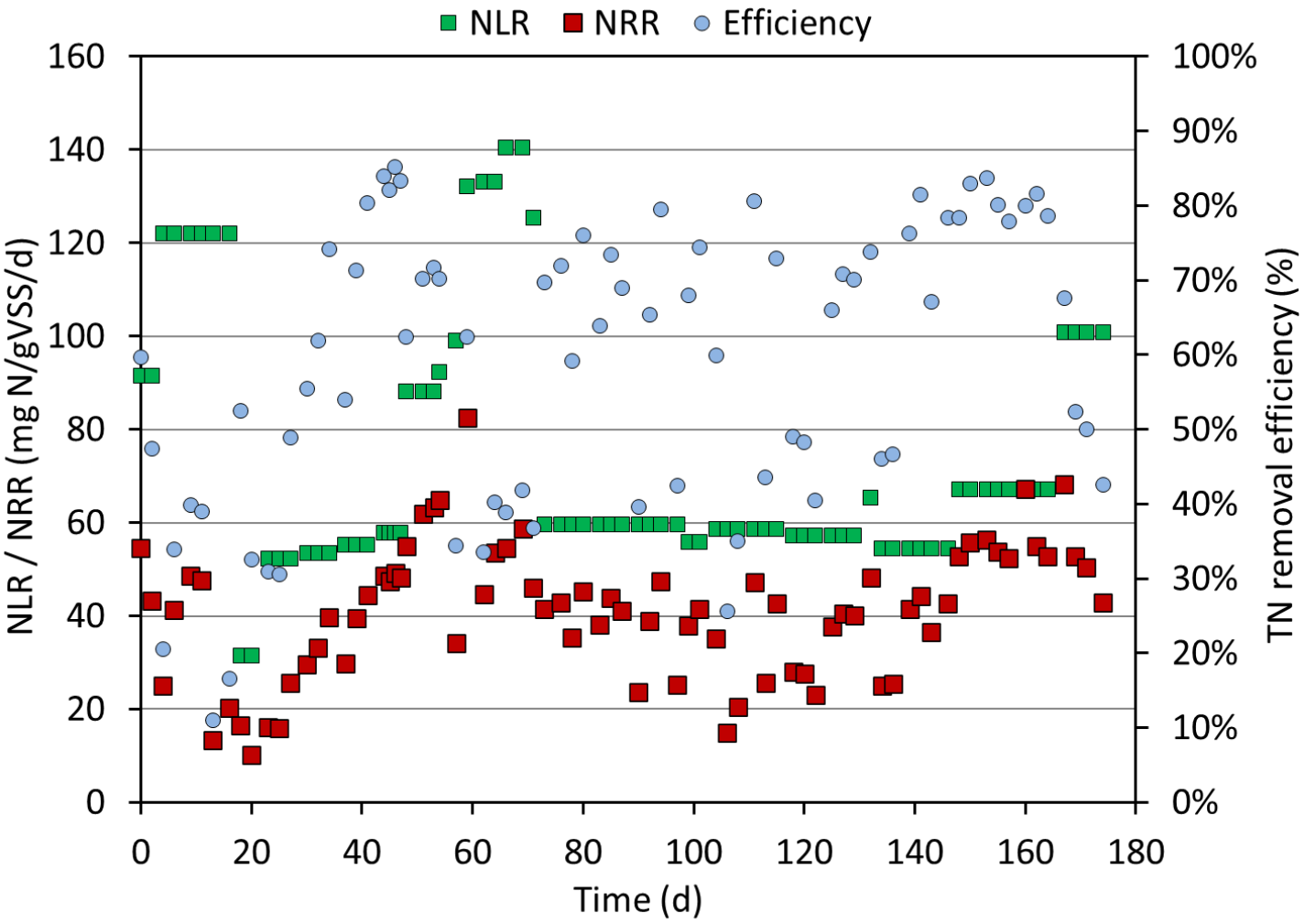
- ✓ **Treatability of WW 5 confirmed**
- ✓ **NRE** up to 85%
- ✓ **Ureolysis** of the ammonifiable organic nitrogen occurs spontaneously (AOBs convert urea to NH_4-N)

- ✘ **Adaptation** of the biomass is needed to achieve NRE > 60%: **requires dilution** at the initial stages
- ✘ High **variability of wastewater's characteristics** slows down the biomass adaptation
- ✘ Possible **long-term inhibitory effects** of DTP wastewater on bacteria consortia

WW 5 – Phase 2


NH_4^+-N
NO_2^--N
NO_3^--N
N org
Tot N IN
Duration
3 h
2 h
N-concentration (mgN/L)
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
0
32
59
92
125
157
Time (d)

WW 5 – Phase 2



NLR_{avg} = 73.7 mgN/gVSS/d **NRR_{avg} = 41.3 mgN/gVSS/d** **NRE_{avg} = 59.0%**

WW 5 – Phase 2 – Main results

- ✓ **Treatability of WW 5 confirmed**
- ✓ With a proper control of DO and pH, **NLR** and **NRR** more than **doubled** with respect to Phase 1
- ✓ With a low variability of the influent characteristics, **long-term inhibitory effects were not observed**

- ✘ **Steady state conditions were not achieved**
- ✘ A **strict control of the process** (T, DO, pH, duration of the phases…) is mandatory to avoid short-term inhibition

Thank you

Simone Visigalli
Micol Bellucci

Piazza Leonardo da Vinci, 32 – Milano (ITA)

simone.visigalli@polimi.it
micol.bellucci@polimi.it

info@life-dentreat.eu

The project has received funding from European Union´s LIFE Programme under Grant Agreement LIFE16 ENV/IT/000345