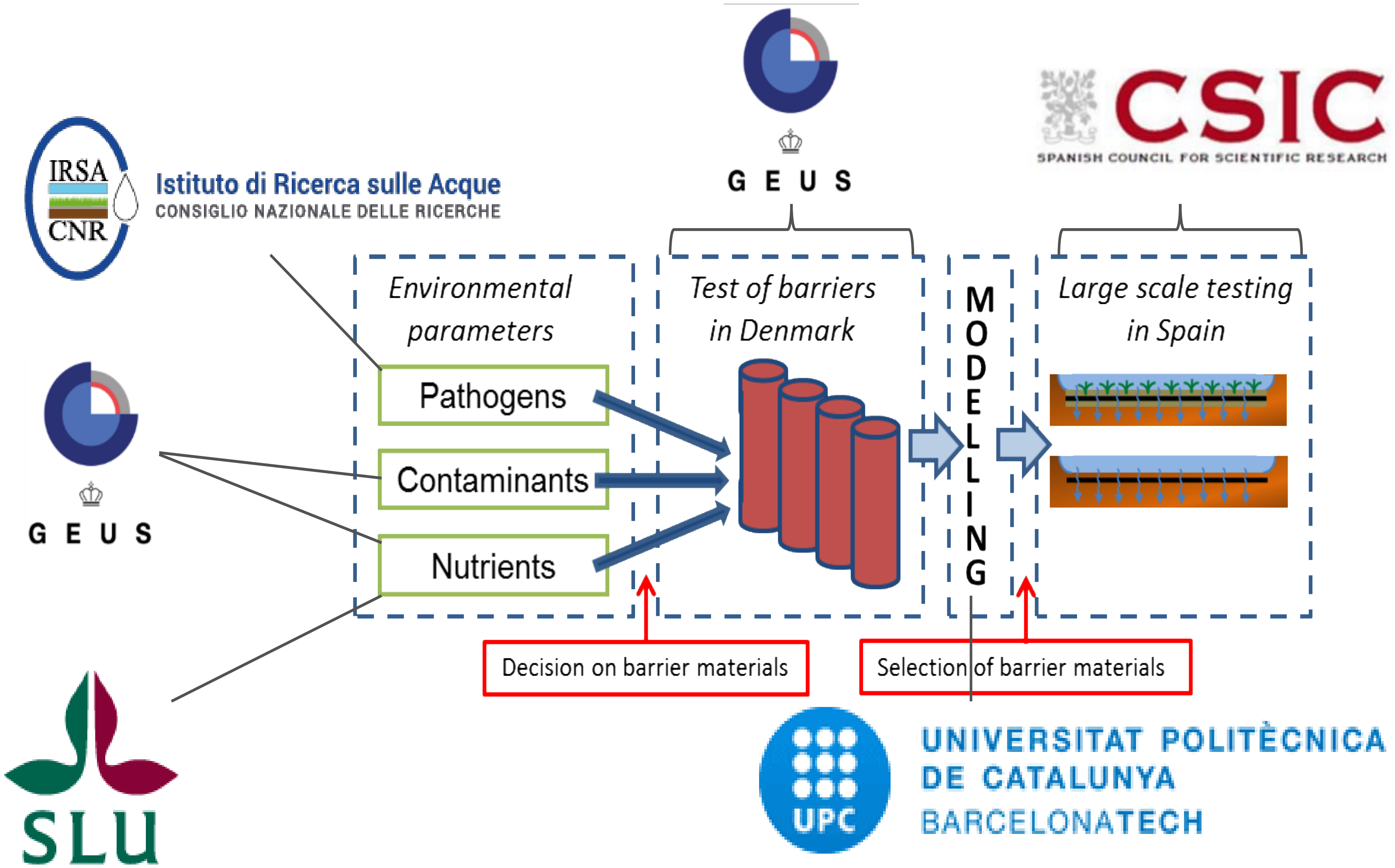
An overview of the ACWAPUR project

Accelerating quality water improvement with horizontal reactive barriers during artificial recharge of aquifers

ACWAPUR project
acwapur.eu

Objective: To develop innovative techniques, tools and management guidelines to avoid recharging pathogenic organisms, inorganic nutrients and organic pollutants to aquifers during the recharge process.

How? Reactive permeable barriers, composed of layers of organic matter that promote the organic pollutants adsorption and facilitate the creation of different redox conditions, to accelerate the processes of anaerobic degradation.



The concept

- Previously tested on “Sant Vicenç dels Horts” site, 5000 m2 experimental recharge basin. There we learned some lessons:

    - Plants growing over the basin surface delaying clogging

    - Different reactive barrier composition (%organic matter and sediments, clay)

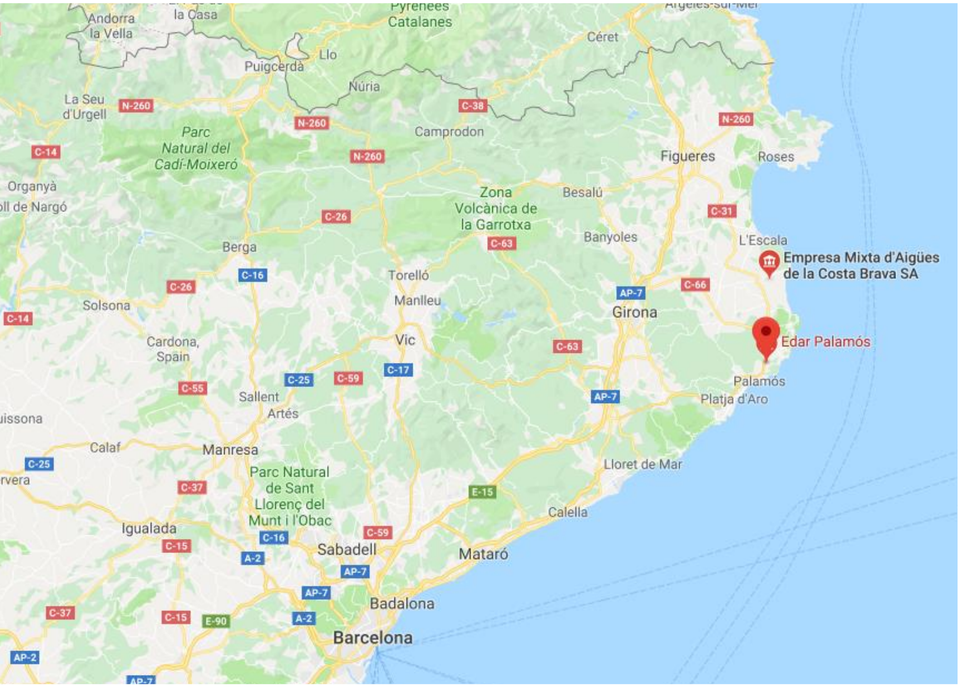
    - Aquifer heterogeneities

- So, ACWAPUR went for new experiments in a known site.
- And then…

If anything can go wrong, it will

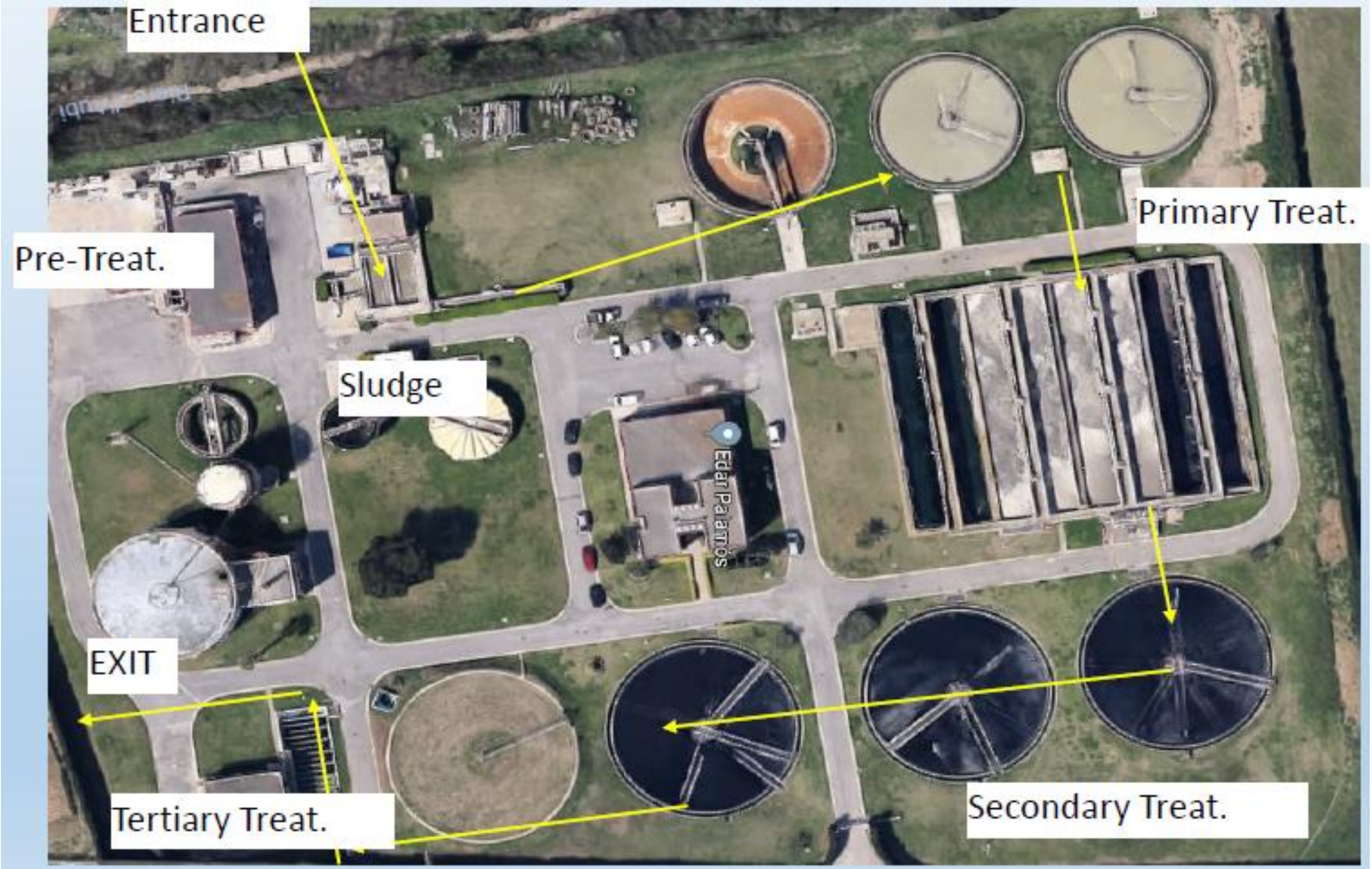
- Murphy was a hell of an optimist
- Before Day 0 everything went wrong. The initial site was dismantled by ACA and we had to rethink the project from scratch
- Then, the column experiments had all kind of problems that you can imagine (and some more you cannot), and they were delayed.
- So, the full project had huge delays, and the consequence is that now, with the project already finished, we are still performing experiments, getting results, modelling,.. ; and, in short, having fun. Publications will follow. One of them is supposed to have more authors than people dying in any random episode of GOT.

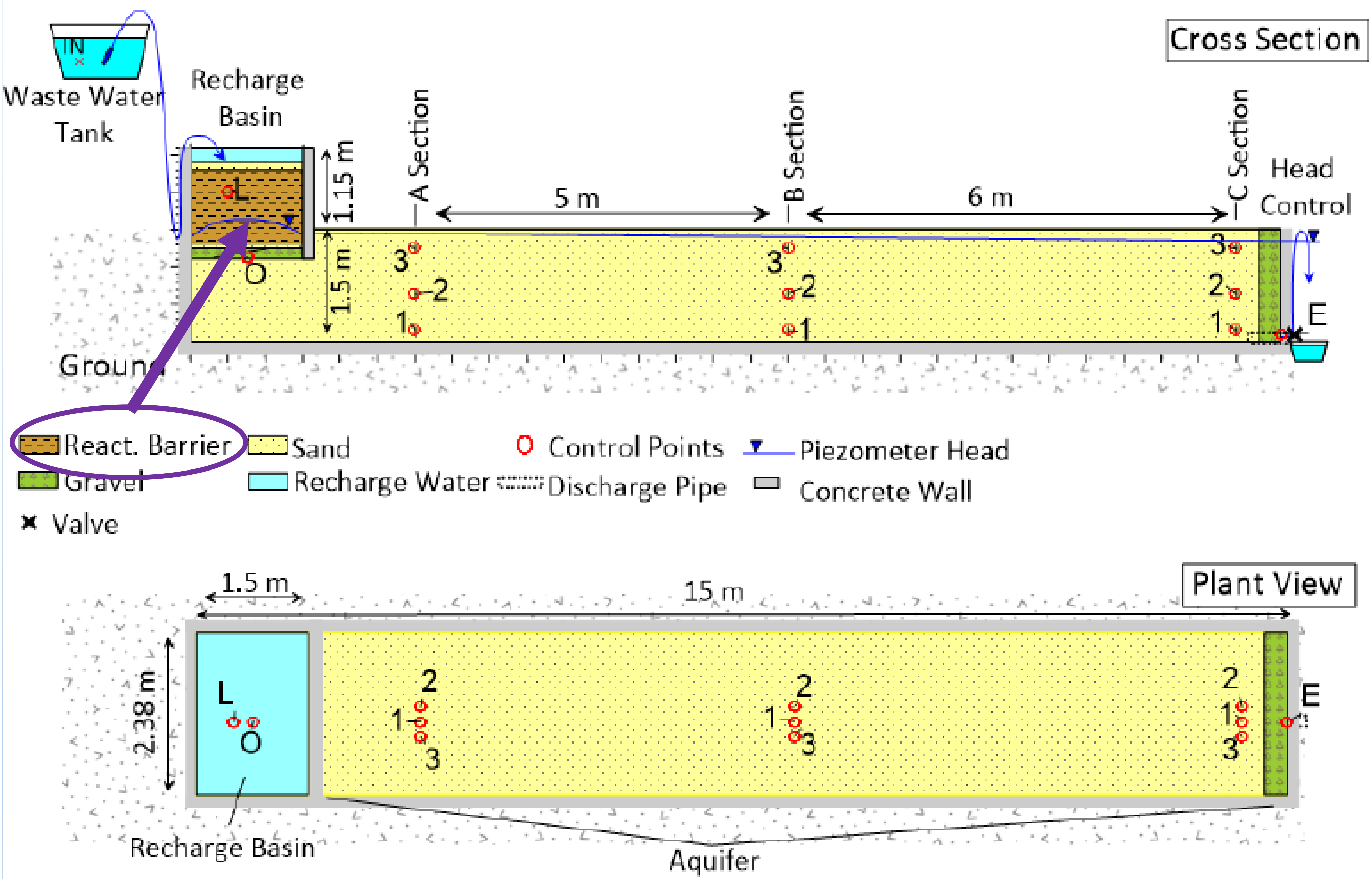
The study area



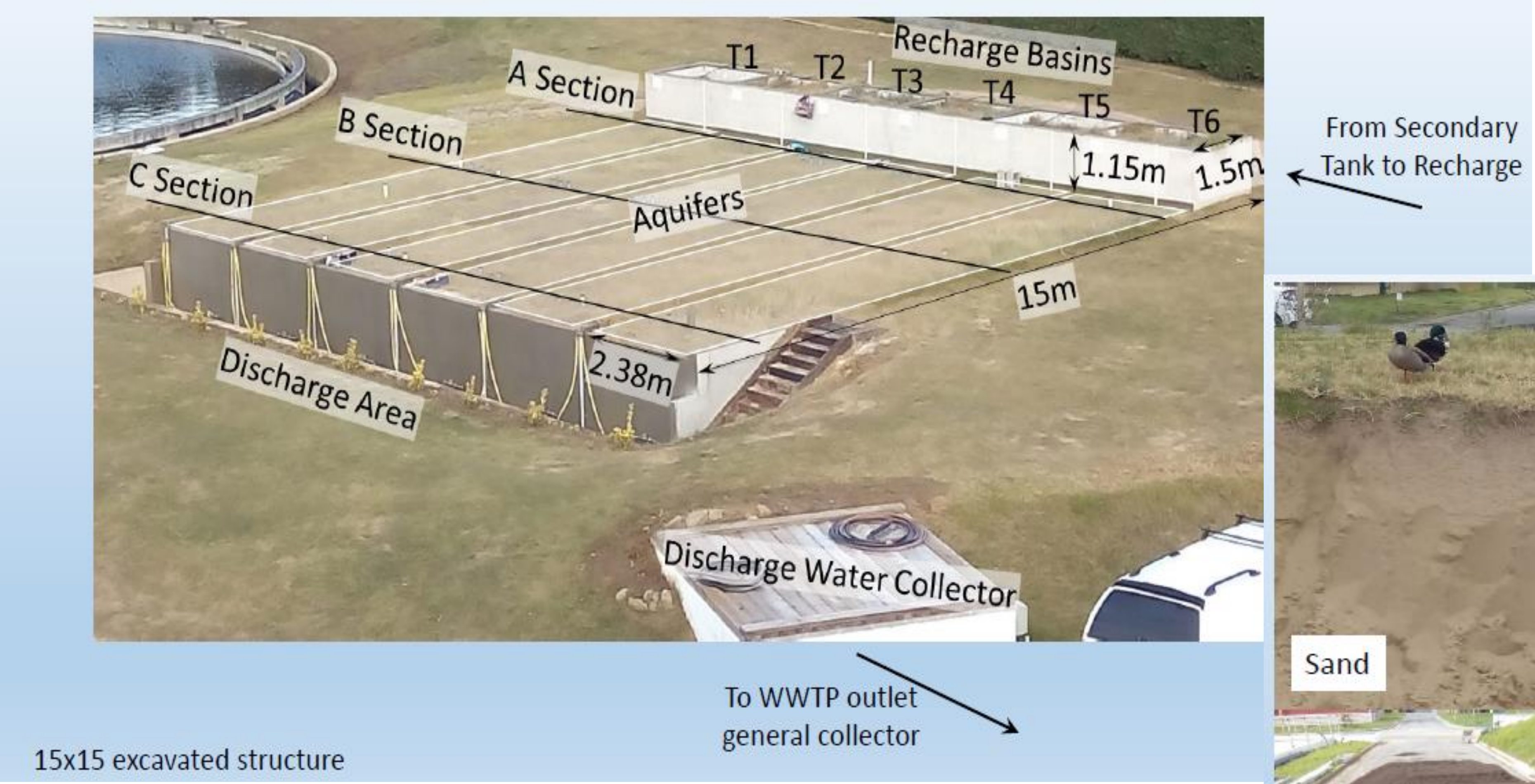
The ACWAPUR SITE

But we are humble hydrogeologists, and we just work here





Cross Section
Waste Water Tank
IN
Recharge Basin
1.15 m
1.5 m
L
O
A Section
B Section
C Section
5 m
6 m
Head Control
3
2
1
E
Ground
React. Barrier
Gravel
Valve
Sand
Recharge Water
Control Points
Discharge Pipe
Piezometer Head
Concrete Wall
Plant View
1.5 m
15 m
2.38 m
Recharge Basin
Aquifer



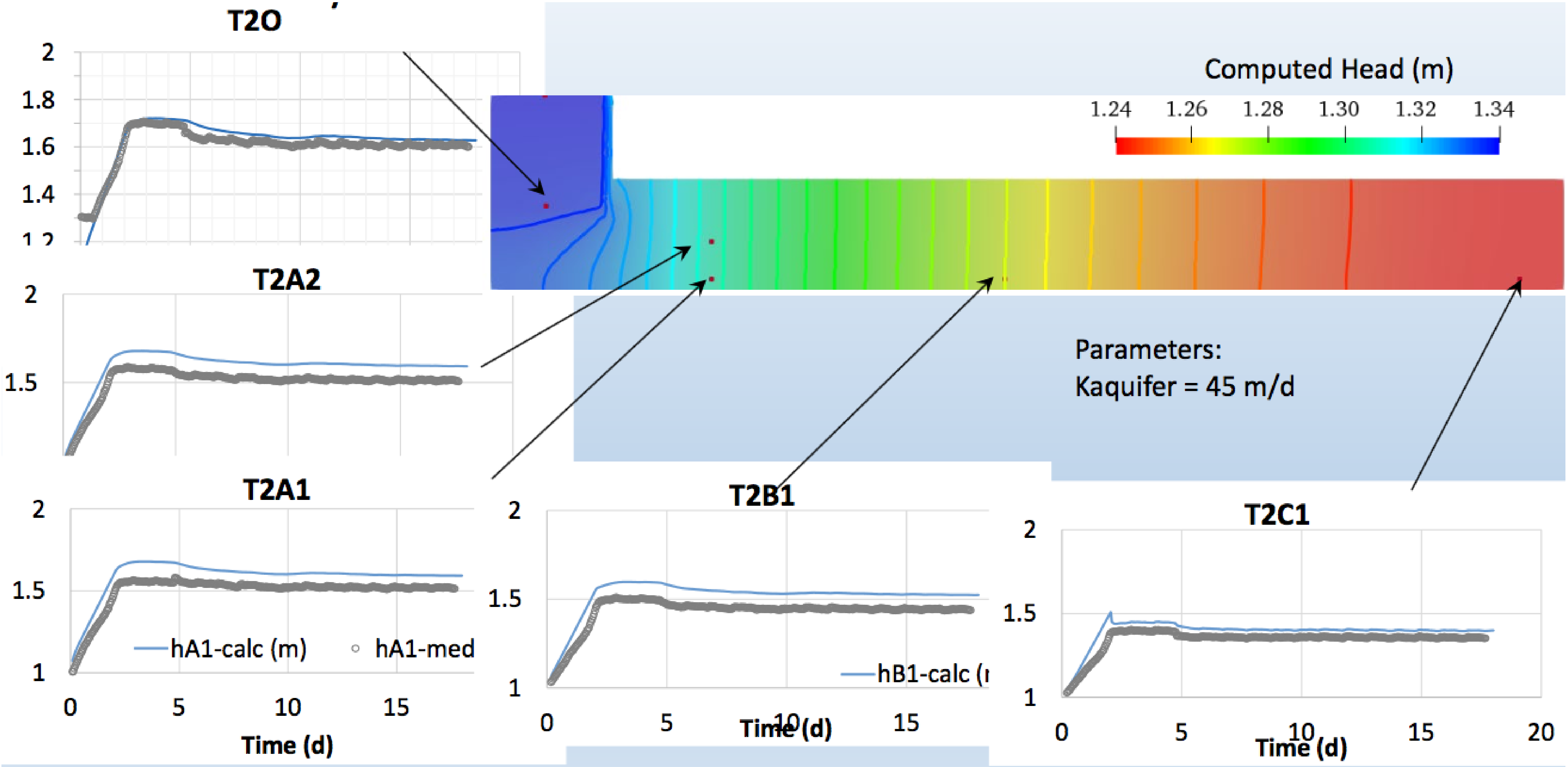
15x15 excavated structure

And it includes 6 independent realities

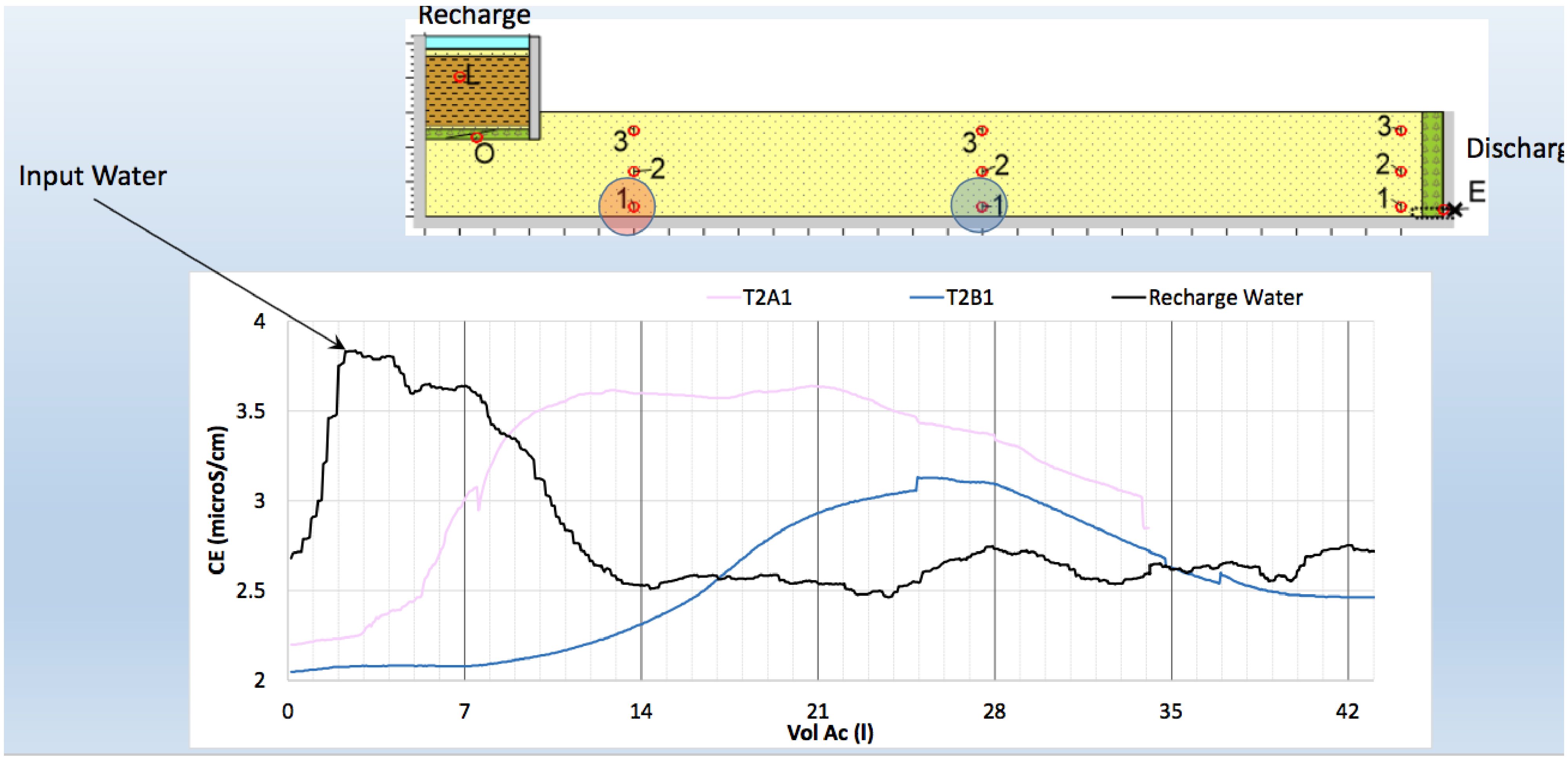
Hydraulic Characterization

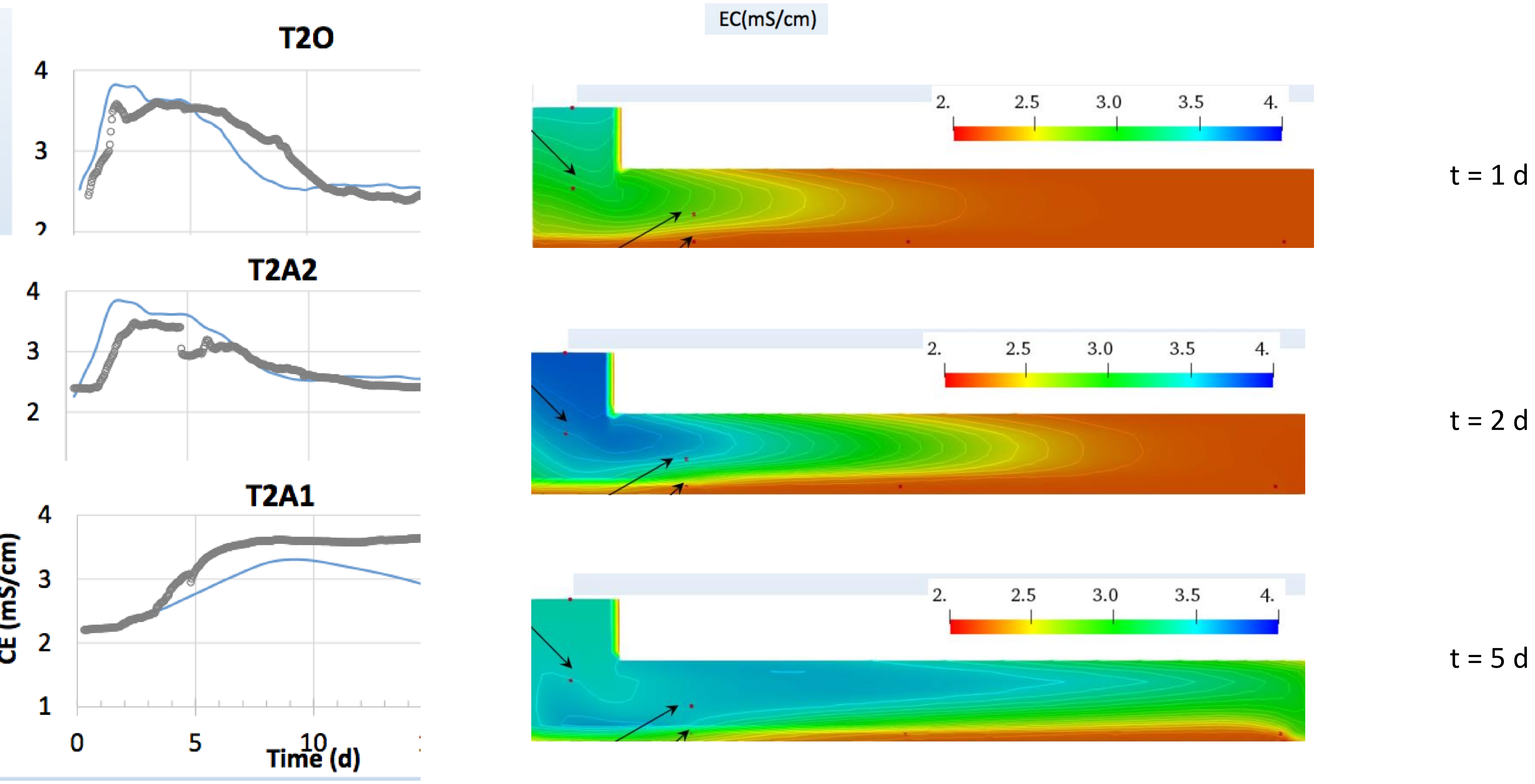
- Final objective: studying the water quality evolution from the inlet point (Managed Aquifer Recharge) to the outlet.
- Step 1: characterize the system from the hydrogeological (and hydrogeochemical) point of view.
- Data: We estimated the flow and conservative transport parameters using measurements (taken during recharge operation) of hydraulic heads and EC.

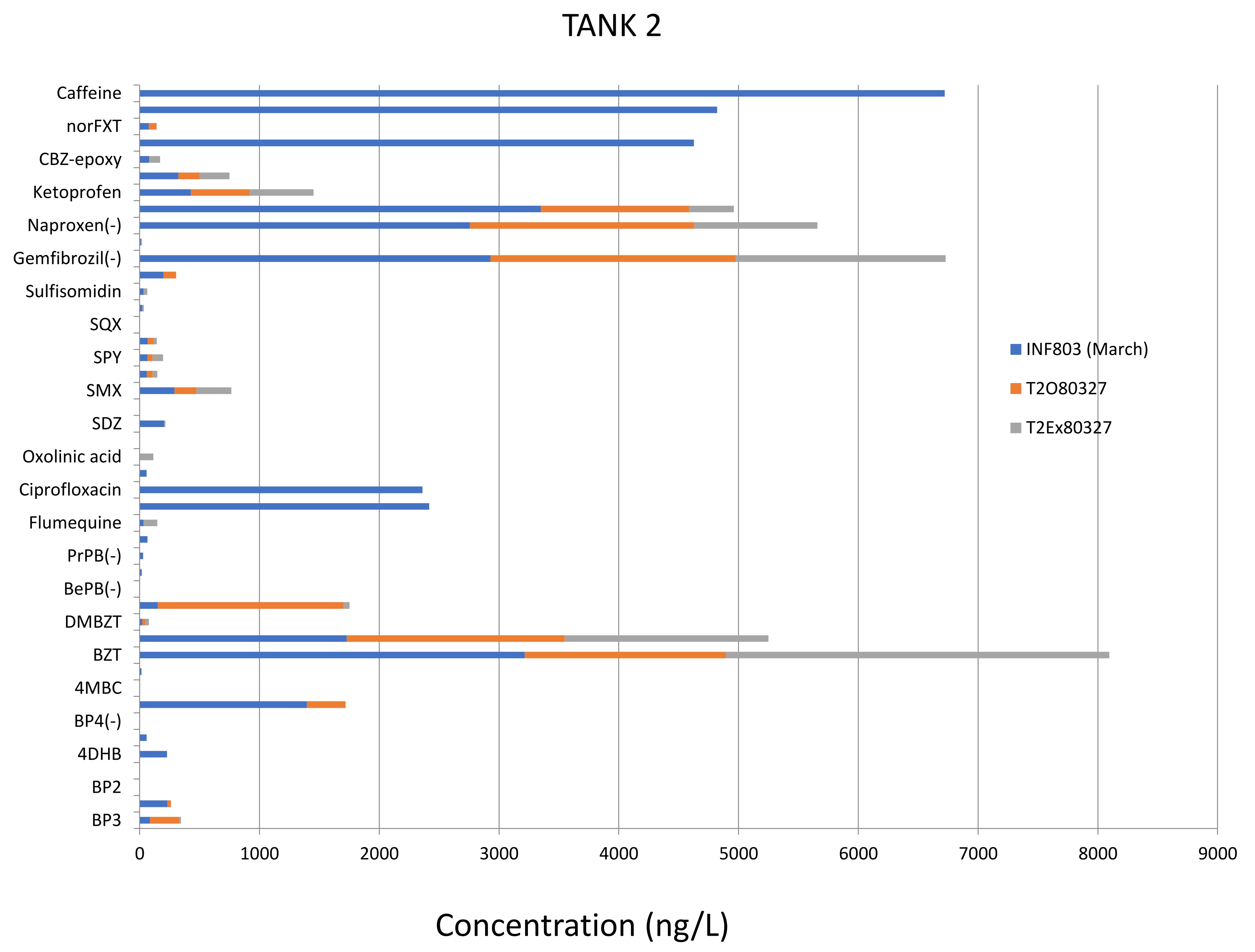
Hydraulic Characterization:
Flow boundary and initial conditions



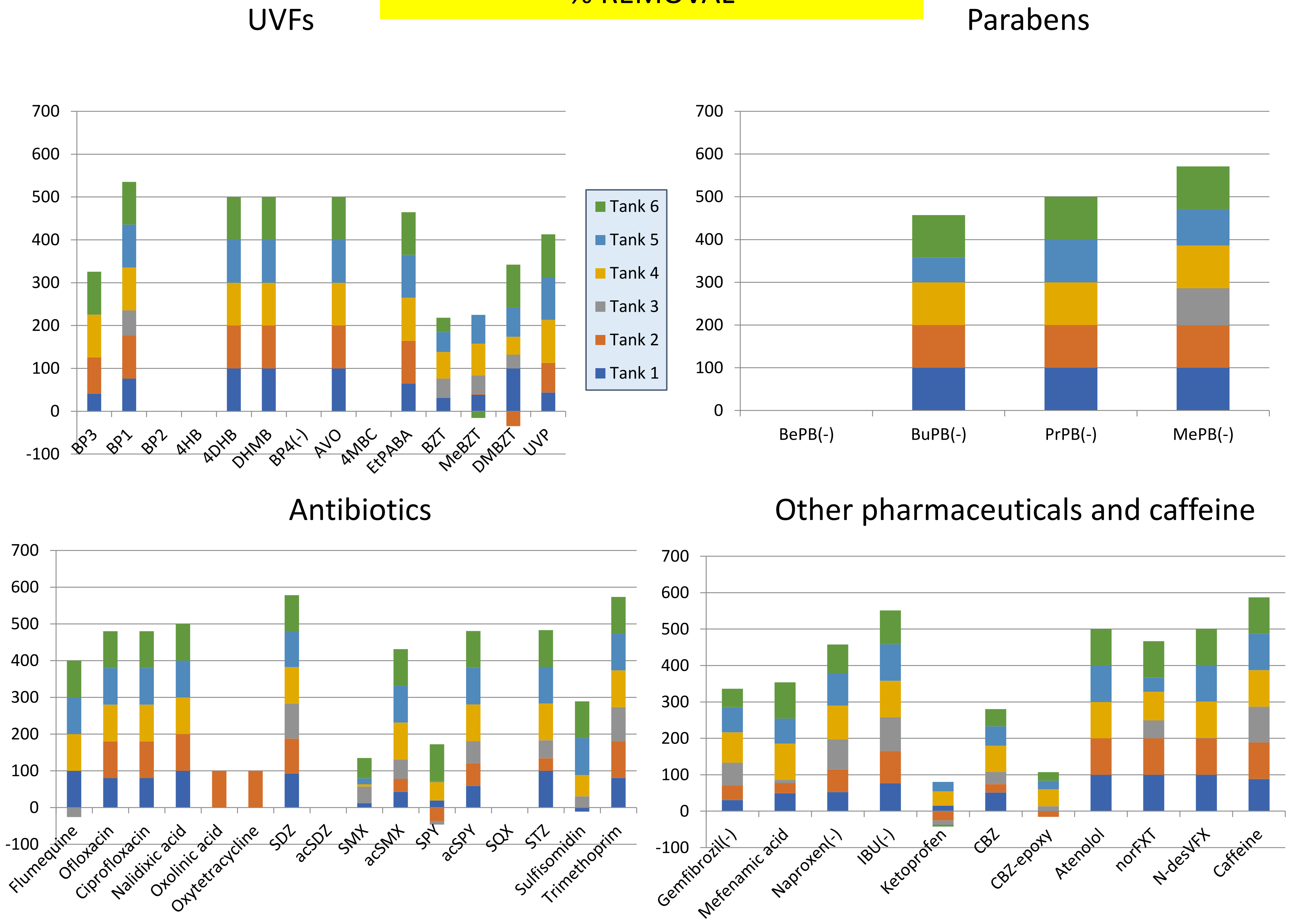
Transport Characterization: EC measurements




T2O
T2A2
T2A1
CE (mS/cm)
Time (d)
0
5
10
EC(mS/cm)
2.
2.5
3.0
3.5
4.
t = 1 d
t = 2 d
t = 5 d


TANK 2
Caffeine
norFXT
CBZ-epoxy
Ketoprofen
Naproxen(-)
Gemfibrozil(-)
Sulfisomidin
SQX
SPY
SMX
SDZ
Oxolinic acid
Ciprofloxacin
Flumequine
PrPB(-)
BePB(-)
DMBZT
BZT
4MBC
BP4(-)
4DHB
BP2
BP3
0
1000
2000
3000
4000
5000
6000
7000
8000
9000
Concentration (ng/L)
INF803 (March)
T2O80327
T2Ex80327

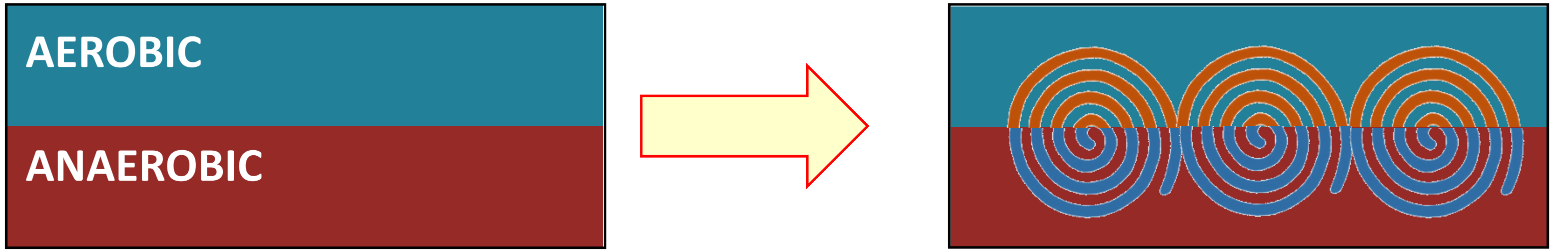
% REMOVAL



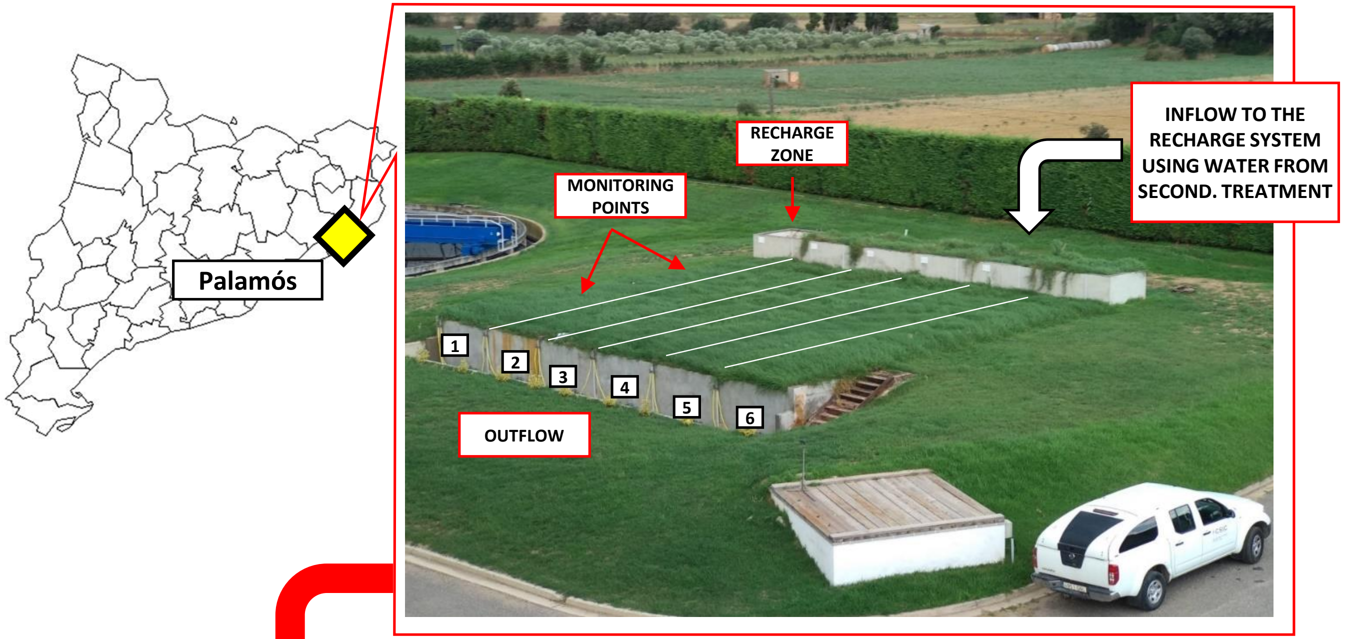
UVFs
Parabens
Antibiotics
Other pharmaceuticals and caffeine
Tank 6
Tank 5
Tank 4
Tank 3
Tank 2
Tank 1
700
600
500
400
300
200
100
0
-100
BP3
BP1
BP2
4HB
4DHB
DHMB
BP4(-)
AVO
4MBC
EtPABA
BZT
MeBZT
DMBZT
UVP
BePB(-)
BuPB(-)
PrPB(-)
MePB(-)
Flumequine
Ofloxacin
Ciprofloxacin
Nalidixic acid
Oxolinic acid
Oxytetracycline
SDZ
acSDZ
SMX
acSMX
SPY
acSPY
SOX
STZ
Sulfisomidin
Trimethoprim
Gemfibrozil(-)
Mefenamic acid
Naproxen(-)
IBU(-)
Ketoprofen
CBZ
CBZ-epoxy
Atenolol
norFXT
N-desVFX
Caffeine

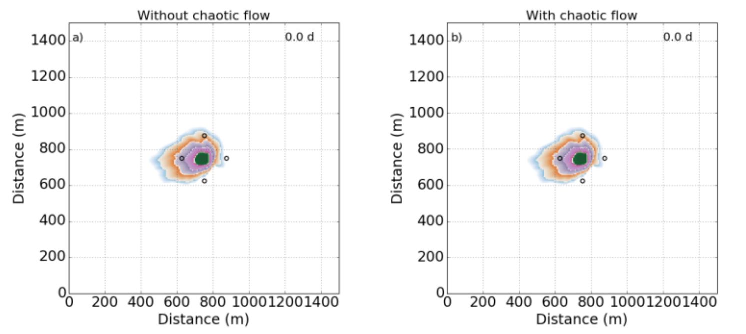
Having Fun # 1: Chaotic Flow

The general idea would be to mix the surface (and aerobic) water with the deeper (and anaerobic) one, with the aim of enhance the ubiquity of redox states.

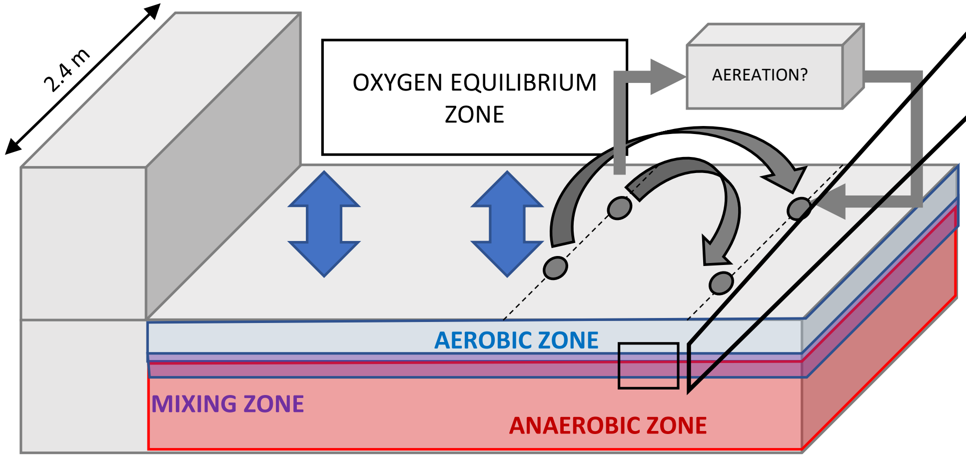


Having Fun # 1: Chaotic Flow





Rodríguez-Escales et al. 2017

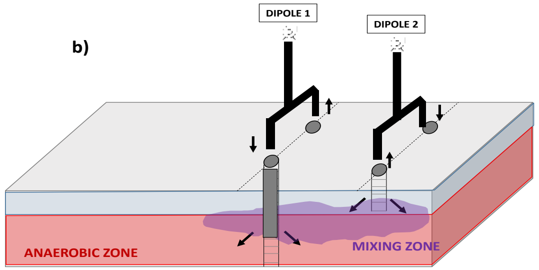


The problem:
strong stratification of redox conditions in the tanks.

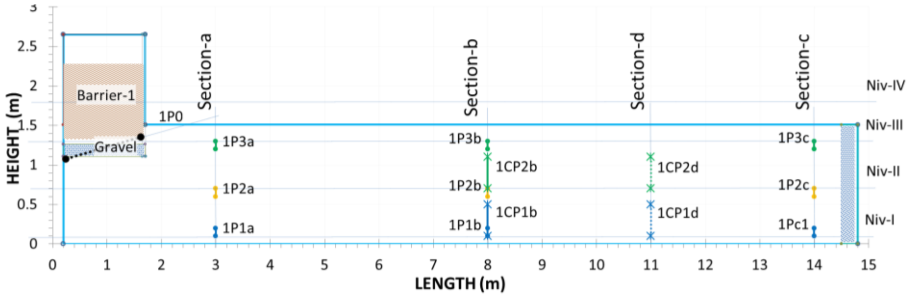
Application of "chaotic flow" to increase the ubiquity of redox potentials

Right now, TANK 1 and 6 are equipped with wells for chaotic flow.

b)

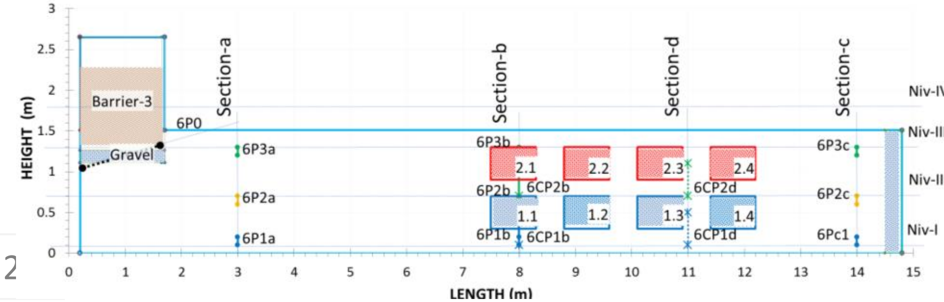


TANK 1



Homogeneous
Barrier: Compost 49%, Sand 49%, Clay 2%
Plants: Yes

TANK 6



Heterogeneous
Barrier: Compost 39%, Sand 59%, Clay 2%
Plants: Yes

We want to install 30 oxygen sensors that can monitor continuously oxygen concentration.

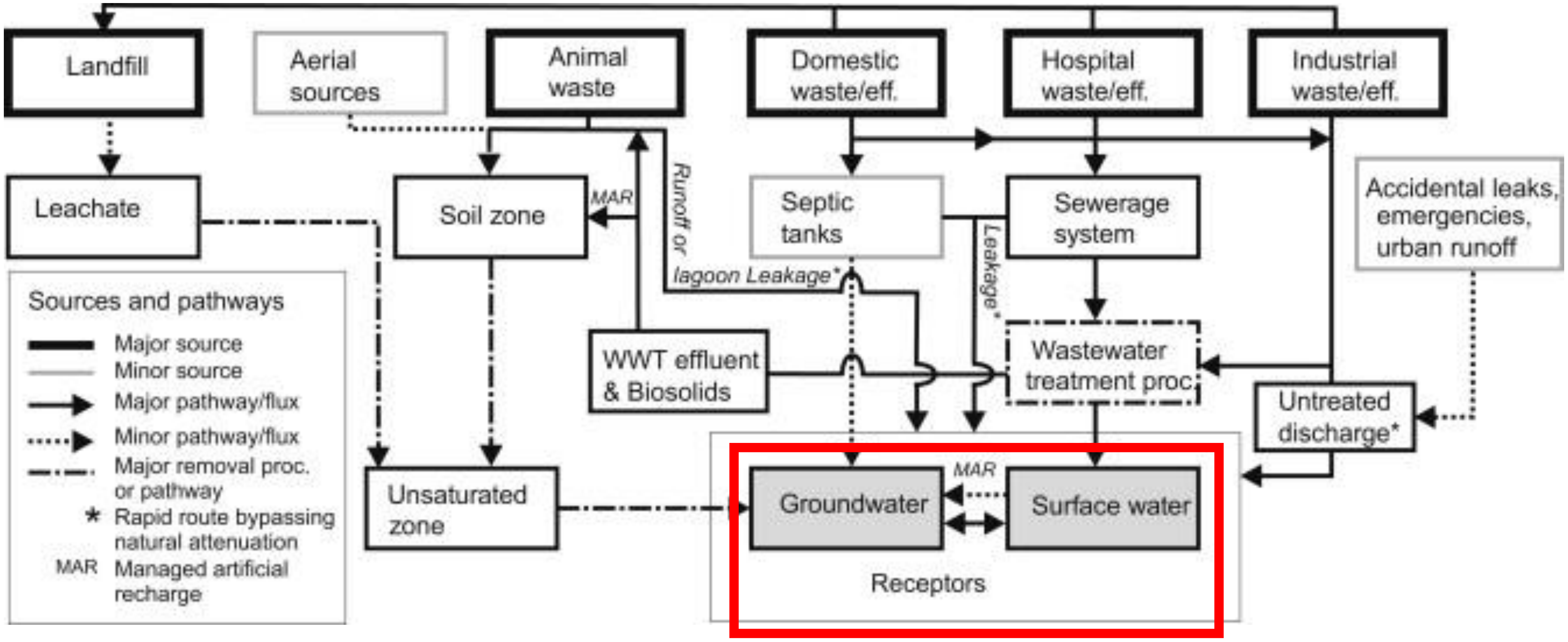
Non intrusive monitoring in mixing zone… and integrated measure at the outflow?

- Generate maps of oxygen in continuous way.
- Redox sensors
- Measure total carbon at the end of the tank
- Use isotopes to trace the reaction rate

Having Fun # 2: Effect of redox conditions and influence of biomass adsorption in UltraViolet-filter's fate

UV filters are used as **personal and care products** like sunscreens and cosmetics (perfumes, creams or shampoos), and in **a number of industrial** applications

Some of them are <u>endrocrine disruptors</u> and have estrogenic activity



Lapworth et al. (2012). EP.

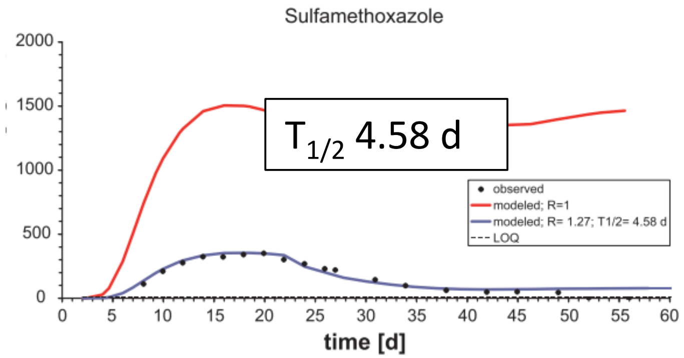
Sorption and **degradation** (mainly by co-metabolism) are two of the most important processes affecting the fate of UV-filters.

Degradation of UV filters

Most of current models of **degradation of EOCs** only focus on apparent processes

$$\boxed{\frac{\partial C}{\partial t} = -kC \quad \Rightarrow \quad T_{1/2} = \frac{\ln K}{2}}$$

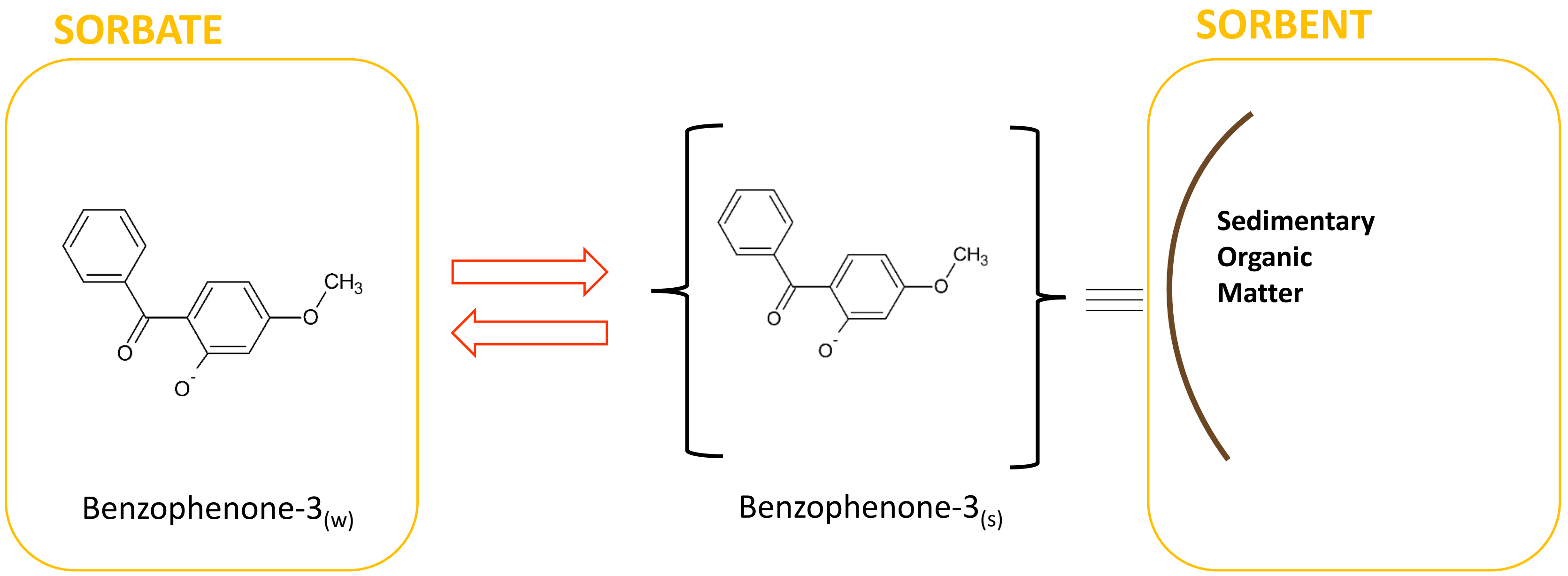
Shaffer et al. 2014. Water Research



There is a lack of models incorporating the real processes that are occurring
Metabolite formation is not contemplated or misunderstood. Metabolites can be more dangerous than parent compounds

Sorption of organic compounds

Sorption also has been treated as a "phenomenological" process.

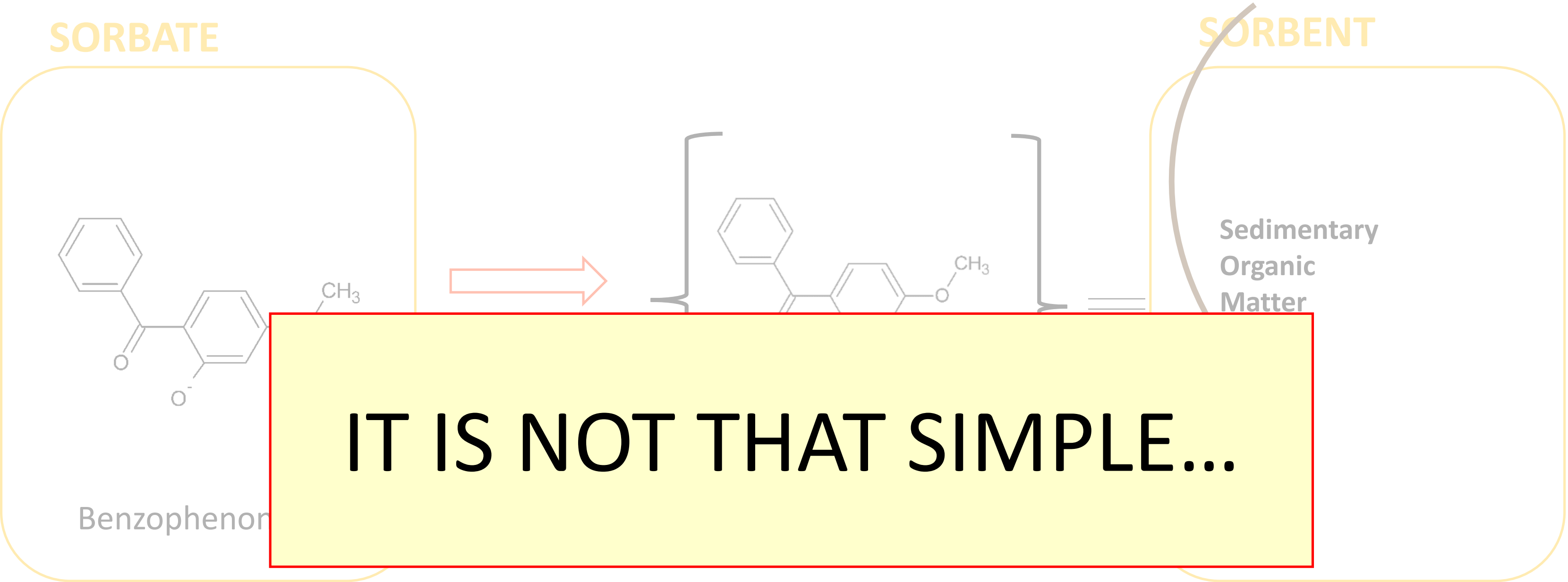


$$K_{d,i} = \frac{C_{i,s}}{C_{i,w}} = \frac{C_{i,om} f_{oc}}{C_{i,w}} = K_{oc,i} f_{oc}$$

- It is generalistic
- Behaviour of all sorbats (EOCs) is the same…
- Behavious of all sorbents (SOM) is the same…

Sorption of organic compounds

Sorption also has been treated as a “phenomenological” process.



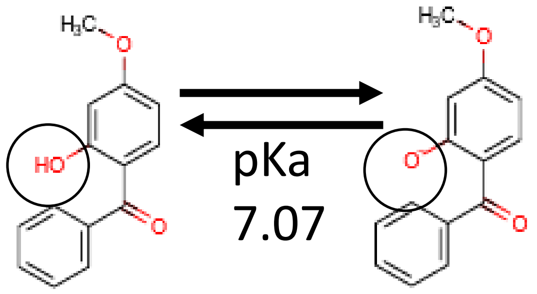
IT IS NOT THAT SIMPLE…

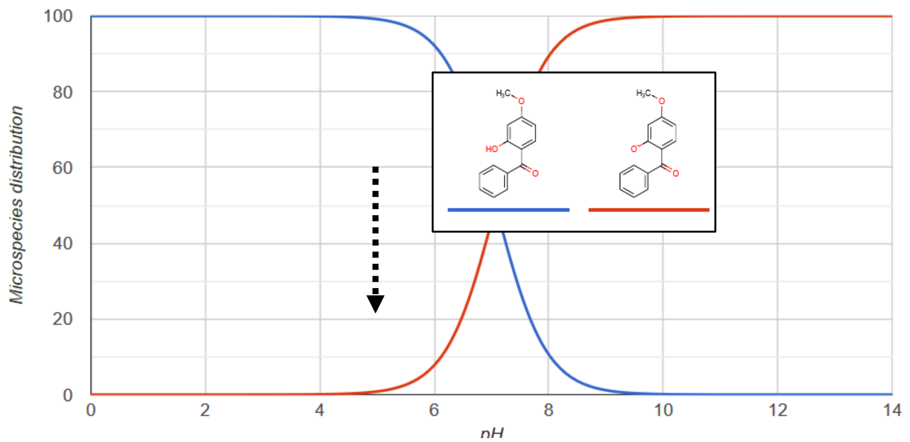
$$K_d = \frac{C_w}{C_s} = K_{oc} f_{oc}$$

- It is generalistic
- Behaviour of all sorbats (EOCs) is the same…
- Behavious of all sorbents (SOM) is the same…

Simplicity removed

- Not all EOCs are neutral.
- Ionics EOCs can form ionic forms at certain pH values





- Organic matter is the most important surface, but not the only one… mineral surfaces can act as sorbents
- SOM is not the only organic matter Surface… what about biomass/biofilms??? It is solid, it is organic… can act as a sorbent?

Exploring K_d

Ionic interactions→ only important for ionic EOCs… (and mainly for cationic EOCs)

$$K_d = \frac{C_{om} f_{om} + C_{min} A + \boxed{C_{ie} \sigma_{ie} + C_{rxn} \sigma_{rxn} A}}{C_{w,neut} + C_{w,ion}}$$

If organic matter is present in the aquifer, mineral interaction can be neglected

$$\boxed{K_d = \frac{C_s}{C_w} = \frac{C_{om} f_{oc}}{C_w} = K_{oc} f_{oc}}$$

So the standard formula applies only when there is organic matter in the system and the compound is neutral

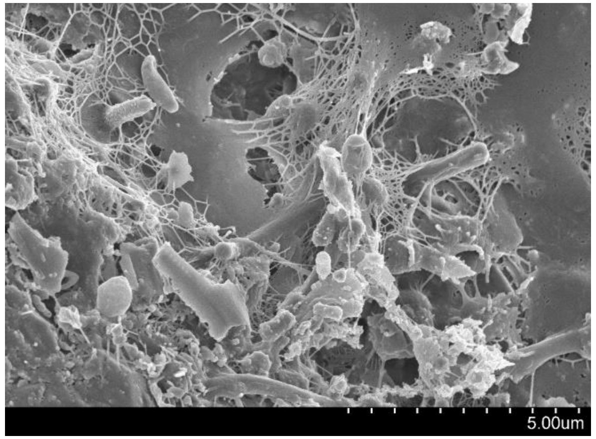
So, what is solid organic matter in an aquifer?

Sedimentary organic matter

- It is the traditional SOM (0.01-0.1)
- Normally, it is recalcitrant.

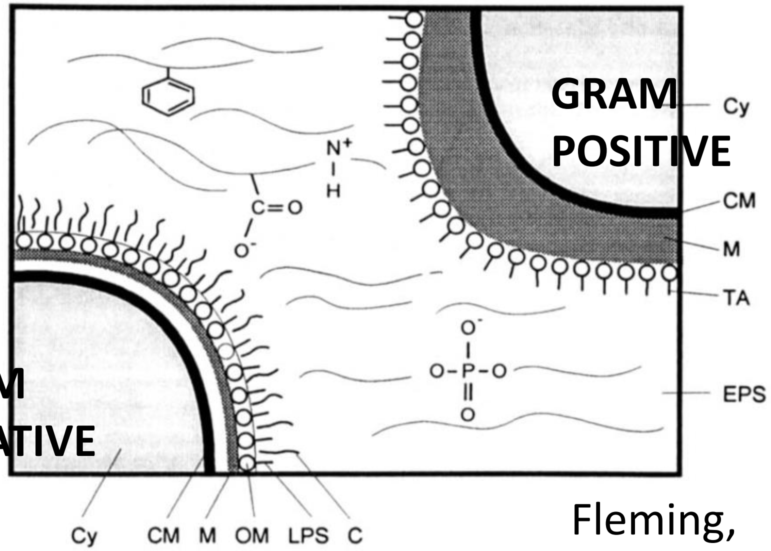
Biomass

- Not important if aquifers are not biologically active
- Biomass can be an important sorbent → biofilm formation (e.g. during MAR bioremediation…)



Biomass in detail

Biomass



Fleming, 1995

Figure 1. Different compartments in a biofilm, including a gram-negative (left) and a gram-positive organism (right). CY = cytoplasm; CM = cytoplasmic membrane; M = murein; OM = outer membrane; LPS = lipopolysaccharide; C = capsule; LTA = lipoteichoic acid.

- Regardless whether aquifers are biologically active or not, biomass can be an important sorbent

Can act biomass as a sorbent?

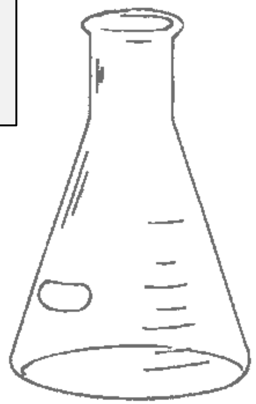
- **EPS** (Extacellular Polysacarids Substances) (**cationic and anionic groups**, apolar groups form proteins and groups with a high hydrogen bonding potential)
- **Outer membrane** (lipopolysaccharides of gram-negative cells and lipoteichoic acids in gram + cells)
- **Cell Wall** (consistin of N-acetyglucosamine and N-aceltymiramic acid, offering cationic & anionic sites)
- **Cytoplasmic membrane** (lipophilic region)
- **Cytoplasm** (hydrophilic region)

Developing a model. First: experiments

Liu et al (2013) performed a set of experiments evaluating the **degradation of a cocktail of UV-filters** under different redox conditions. They measured the temporal evolution of water and solid concentrations.

Organic matter (acetate)
Electron acceptor
UV-filter cocktail
Groundwater
Sediment

Total time: 80 d

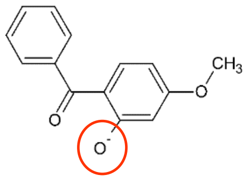
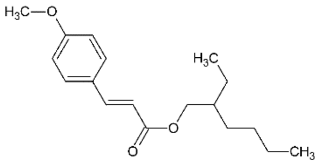
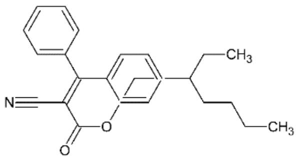
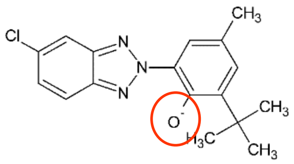
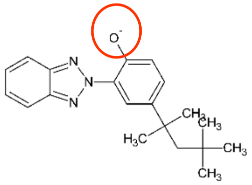


AEROBIC CONDITIONS

ANAEROBIC CONDITIONS

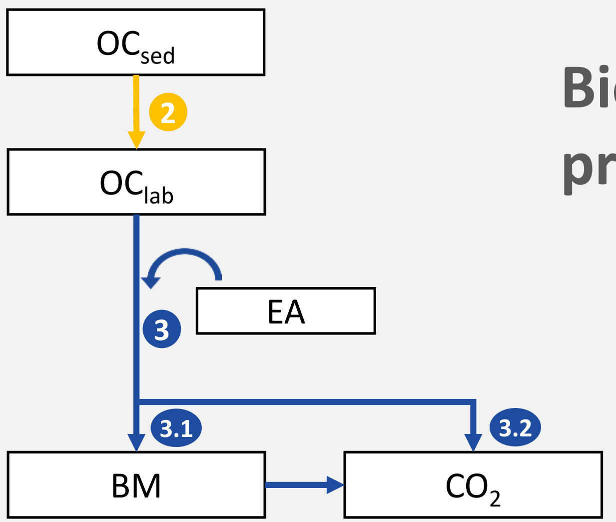
Nitrate conditions
Sulfate reduction conditions

The “stuff”

	BP3	OMC	OC	UV-326	UV-329
					
$logK_{oc}$	3.10	4.09	5.61	6.07	6.55
$logD_{ow}$	3.13	5.38	6.78	5.33	5.9
pK_a	7.07	-	-	10.08	9.30
	Ionic anionic	**neutral**	**neutral**	**Ionic anionic**	**Ionic anionic**

Benzophenone-3	BP3
Octyl 4-methoxycinnamatte	OMC
Octocrylene	OC
2-(3-t-butyl-2-hydroxy-5-methylphenyl)5-chloro benzotriazole	UVA-326
2-(2’-hydroxy-5’-octylphenyl)-benzotriazole	UVA-329

The conceptual model



Bio-geo-chemical processes

1 UV-filter sorption

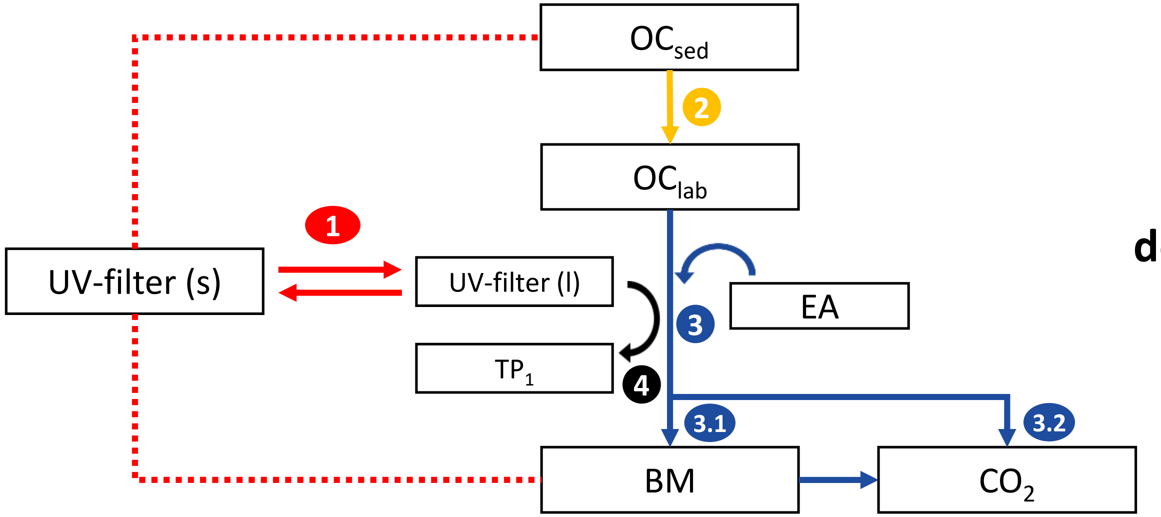
2 Hydrolisis of OC_{sed}

3 Oxidation of labile OC and growth of biomass

4 Co-metabolic degradation of UV-filters

OC_{sed} Sedimentary organic carbon
OC_{lab} Labile organic carbón
TP Transformation product
EA Electron Acceptor
BM Biomass

The conceptual model



UV-filter's fate depends on biogeochemical processes

⇩

Coupling sorption and degradation of UV filters with geochemical models

1 UV-filter sorption

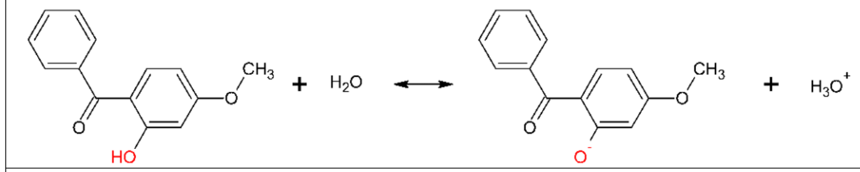
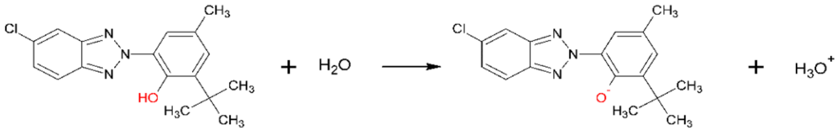
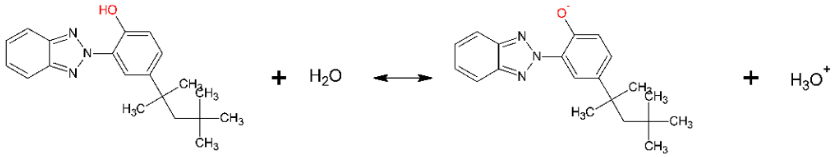
2 Hydrolisis of OC_{sed}

3 Oxidation of labile OC and growth of biomass

4 Co-metabolic degradation of UV-filters

OC_{sed} Sedimentary organic carbon
OC_{lab} Labile organic carbón
TP Transformation product
EA Electron Acceptor
BM Biomass

Numerical model. Step 1: extension of the database

Reaction	logK
Speciation reactions of ionic UV filters (1)	
Speciation of BP3 (as an organic acid) and formation of BP3$^-$ 	6.93
Speciation of UVA-326 (as an organic acid) and formation of UVA-326$^-$ 	10.08
Speciation of UVA-329 (as an organic acid) and formation of UVA-329$^-$ 	9.3

1. Definition of new master species
2. Definition of reactions
3. Definition of logk

Numerical model. Step 2: cumulative sorption model (OM and biomass)

$$K_{d_{UV,TOT}} = \sum_{j=1}^{j} \sum_{i=1}^{i} K_{d_{i,j}}$$

where "i" is referred to the different sorbents (1=SOM and 2=biomass), and "j" to the different form of the UVs (1=neutral and 2=ionic form).

Sedimentary Organic Matter:

If the presence of ionic compound is relevant

$$K_{d_{UV,SOM}} = \frac{[K_{oc,UV_0} + K_{oc,UV_{[-]}} K_a/[H^+]] f_{oc}}{1 + \frac{K_a}{[H^+]}} \xrightarrow{K_a/[H^+] << 1000} K_{d_{UV,SOM}} \simeq \frac{K_{oc,UV_0} f_{oc}}{1 + \frac{K_a}{[H^+]}}$$

Biomass:

$$K_{d_{UV,X}} = \frac{[K_{X,UV_{[-]}} K_a/[H^+]] f_{sites}}{1 + \frac{K_a}{[H^+]}}$$

Only ionic compounds interact with biomass

Numerical model. Step 3: Co-metabolic degradation, coupling geochemistry and UV filter degradation

Degradation of labile organic matter under different electron accetpors:

$$r_{ED} = -k_{max} \frac{[ED]}{[ED]+K_{S,ED}} \frac{[EA]}{[EA]+K_{S,EA}} [X]$$

$$r_{EA} = Qr_{ED} - Sb[X]$$

$$r_X = -Y_h r_{ED} - b[X]$$

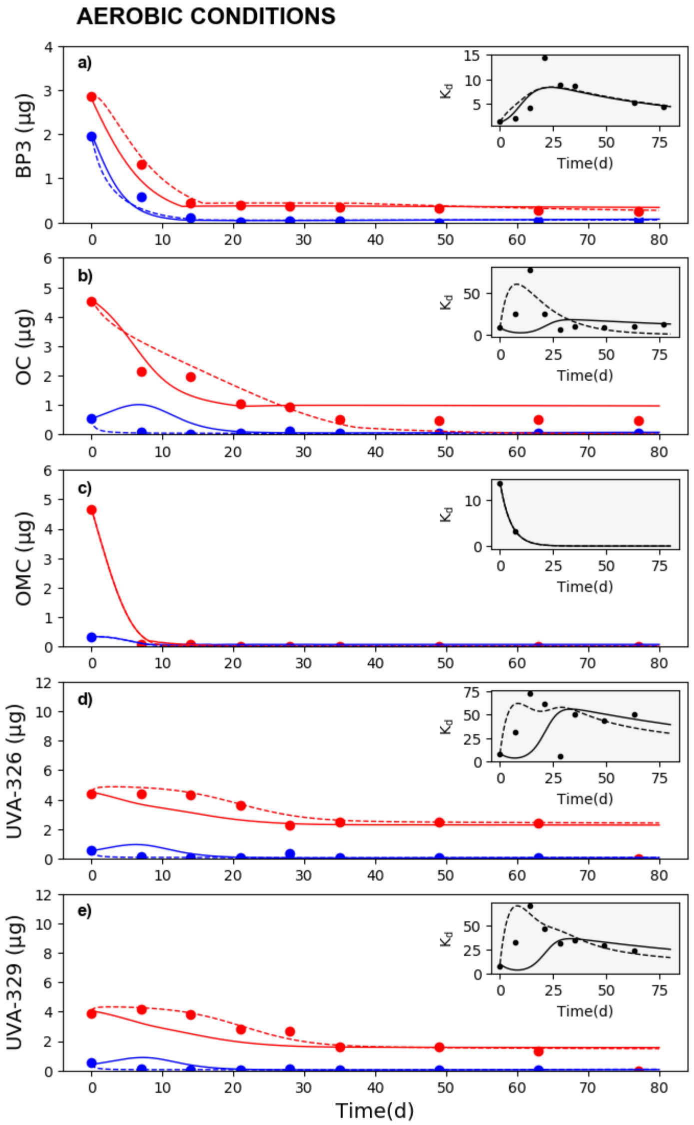
Release of labile organic matter from Sedimentary Organic Matter

$$r_{DOC} = -k_{max}[SOM]$$

Release of labile organic matter from Sedimentary Organic Matter

$$r_j = -C_j k_{j,i} F_i \qquad j = BP3, OMC, OC, UV326, UV329$$

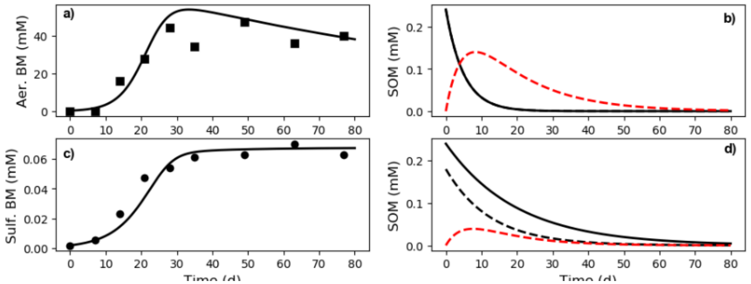
Numerical modeling. Results



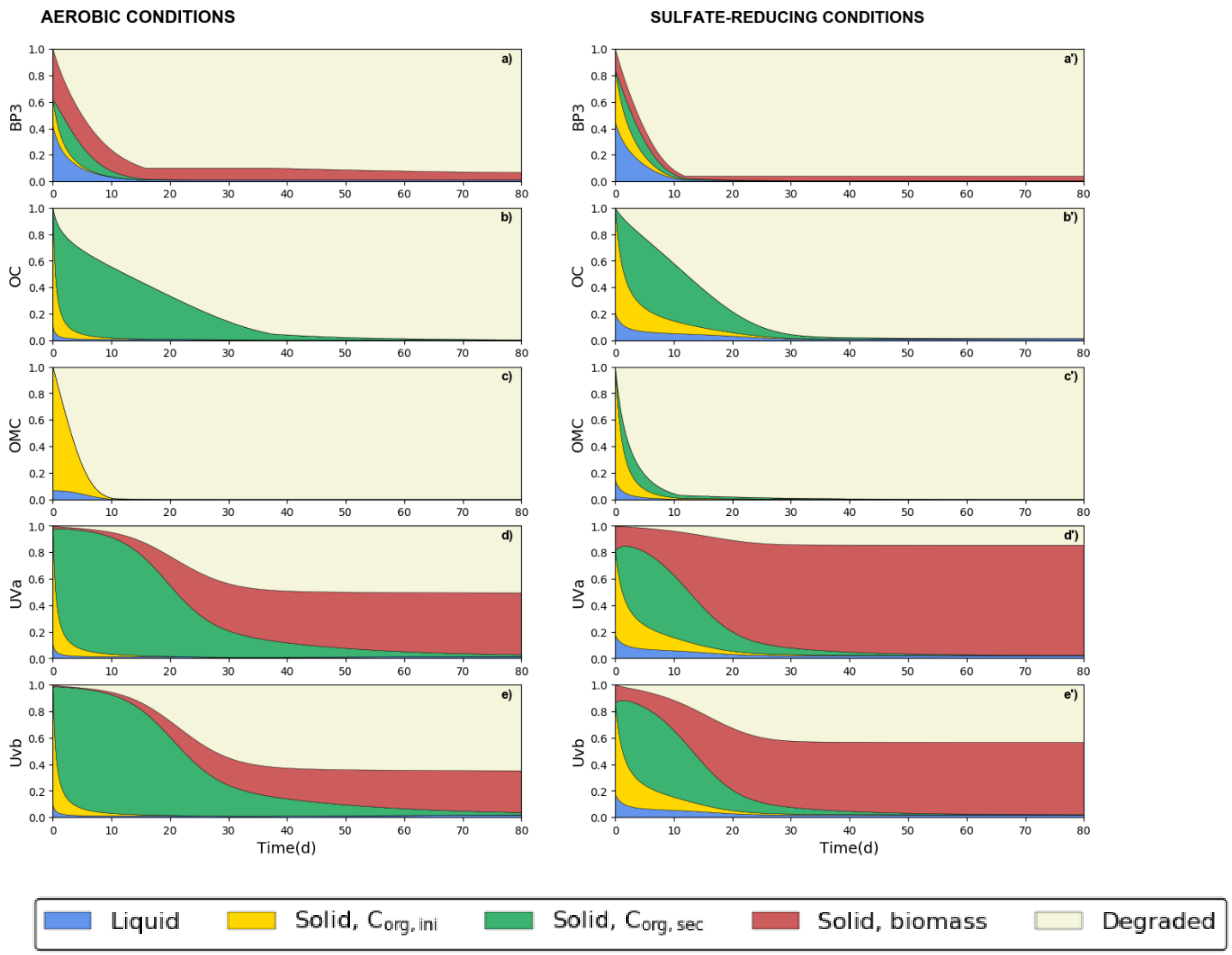
- UV-filters mass is fitted but there is a problema in Kd…
- It seems we miss some process…
- We hypothesized that an intermediate surface appear from initial SOM

Biomass surface

SOM surface



Numerical modeling. Results



GHS

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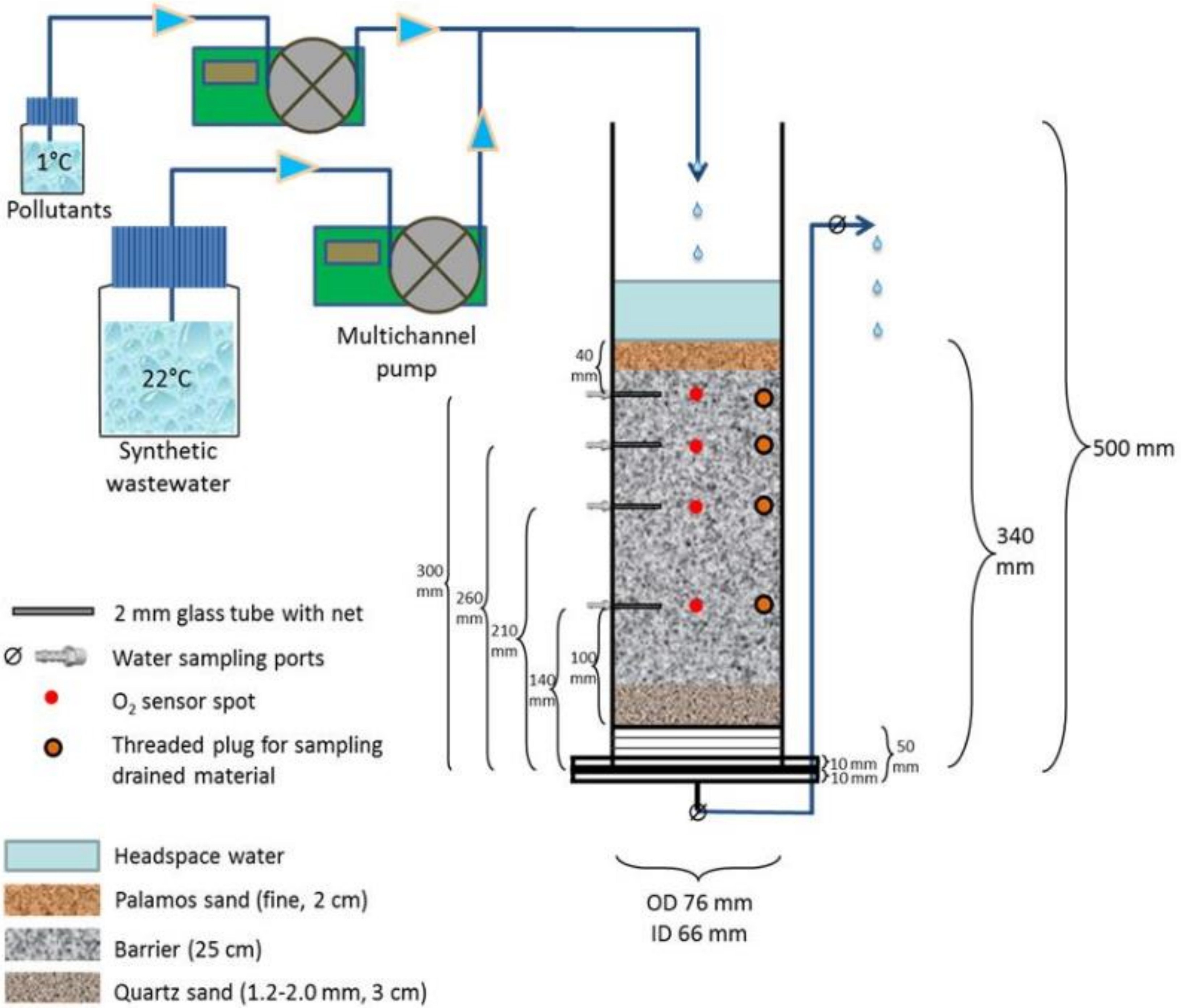
What we are doing right now…

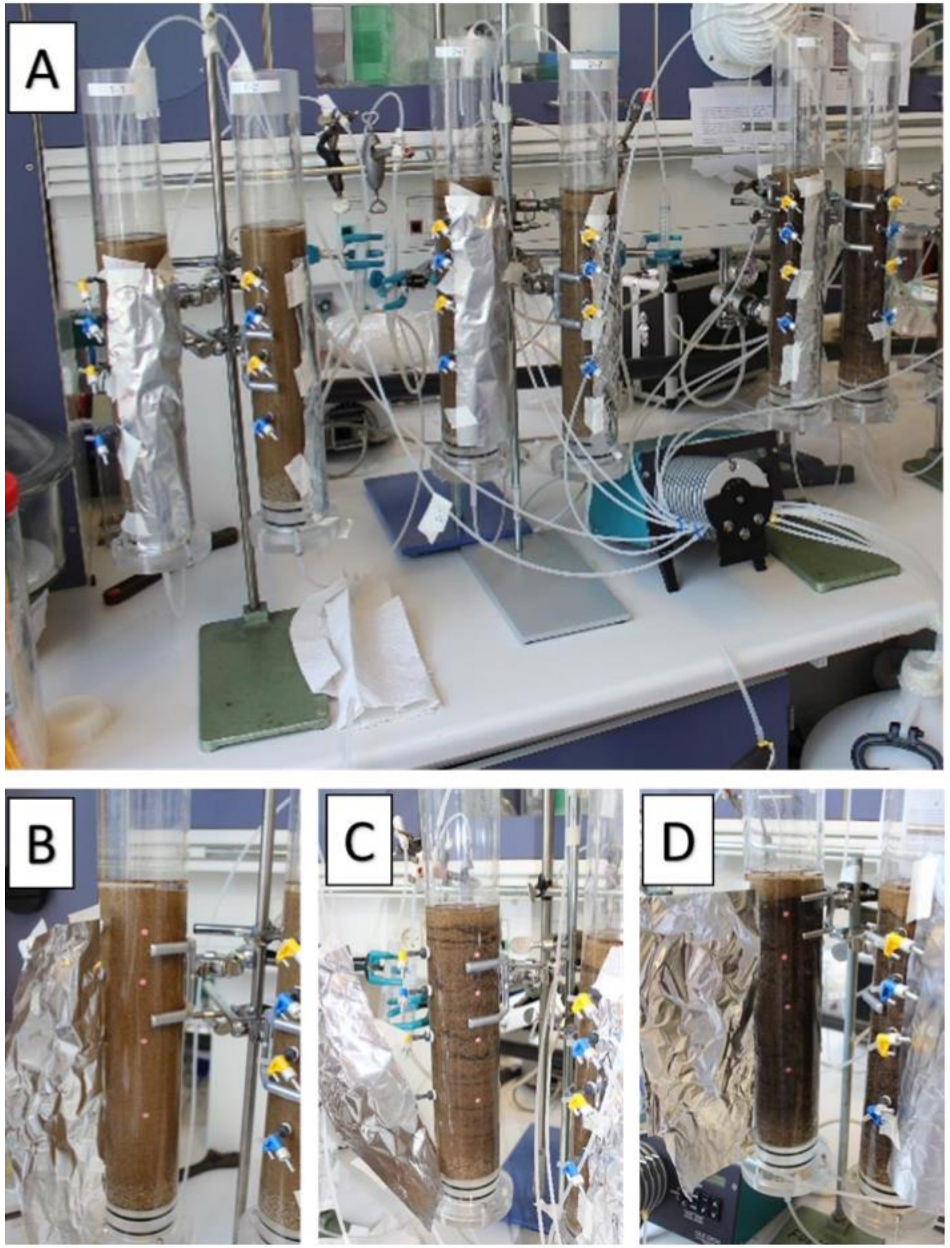
- Injecting UV filters in Palamos site.
- We feed the water from the secondary treatment, dope it with UV filters and check its fate as a function of location and time
- We record O2 distribution (optic fiber)
- We collect samples within the facility and in the outlet

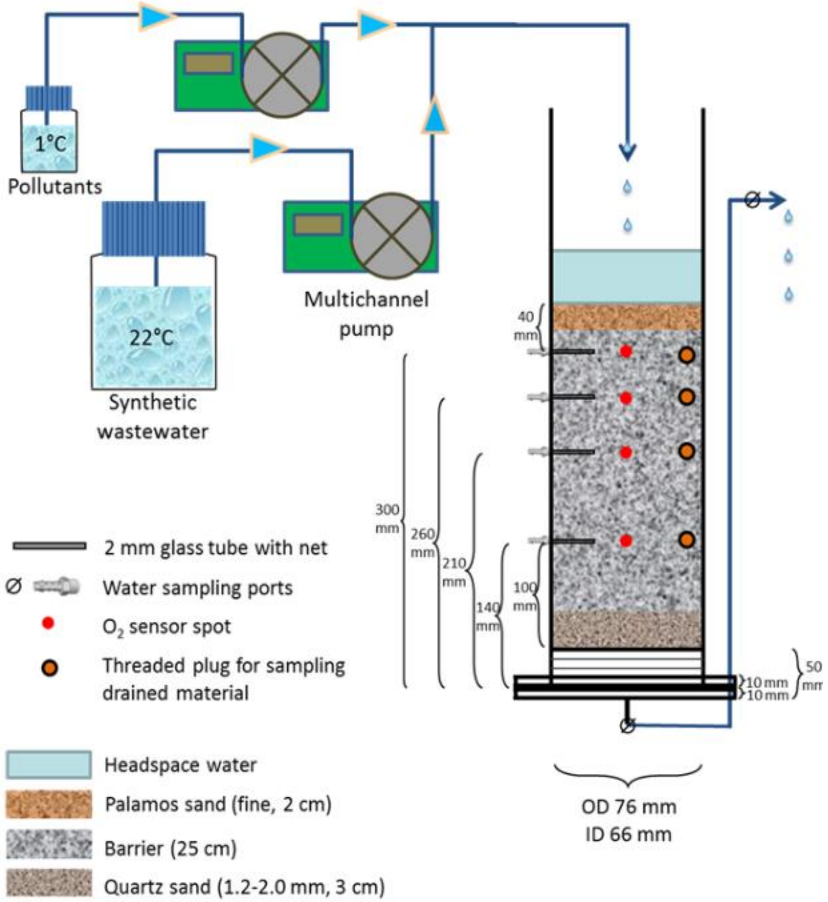
Second big line of work:

Laboratory columns

Set-up







Flow rate: 0.5 mL / min = 30 mL / h

Empty-bed **residence time: 24 h**

Tracer breakthrough after ~10 h

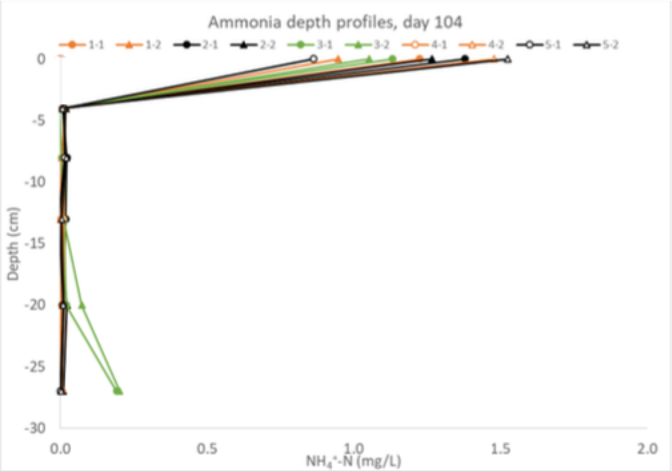
The scientific questions

- **Does a reactive barrier (with compost) improve efficiency of a MAR system?**
- **Does inoculation matter?**

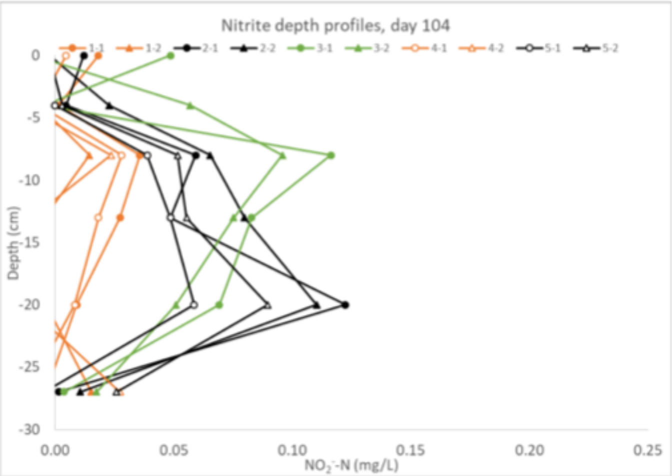
column	material	inoculation
1-1	sand	yes
1-2	sand	yes
2-1	10% compost	yes
2-2	10% compost	yes
3-1	50% compost	yes
3-2	50% compost	yes
4-1	sand	no
4-2	sand	no
5-1	10% compost	no
5-2	10% compost	no

Some Results

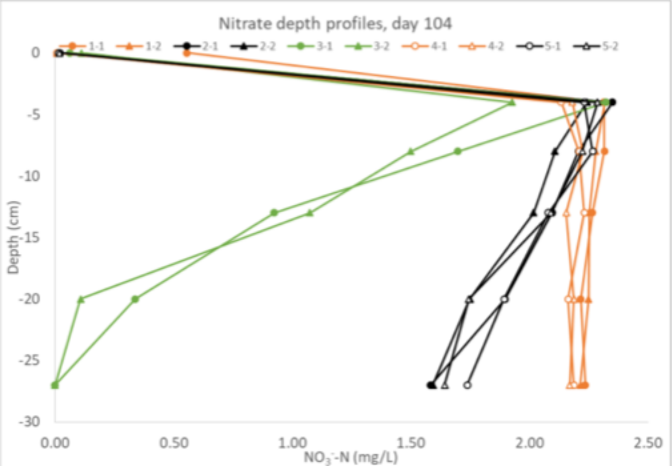
NH_4

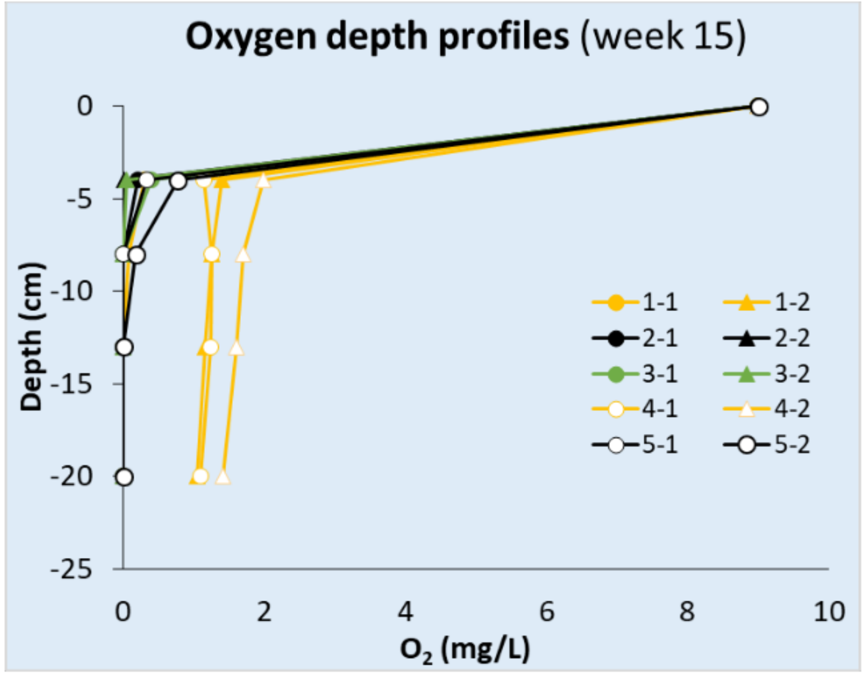


NO_2



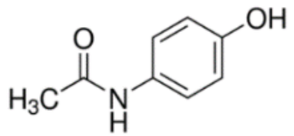
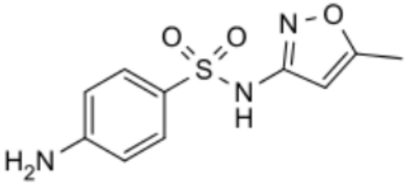
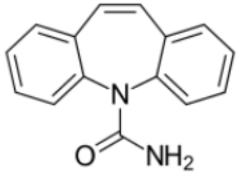
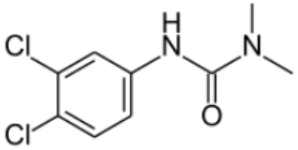
NO_3

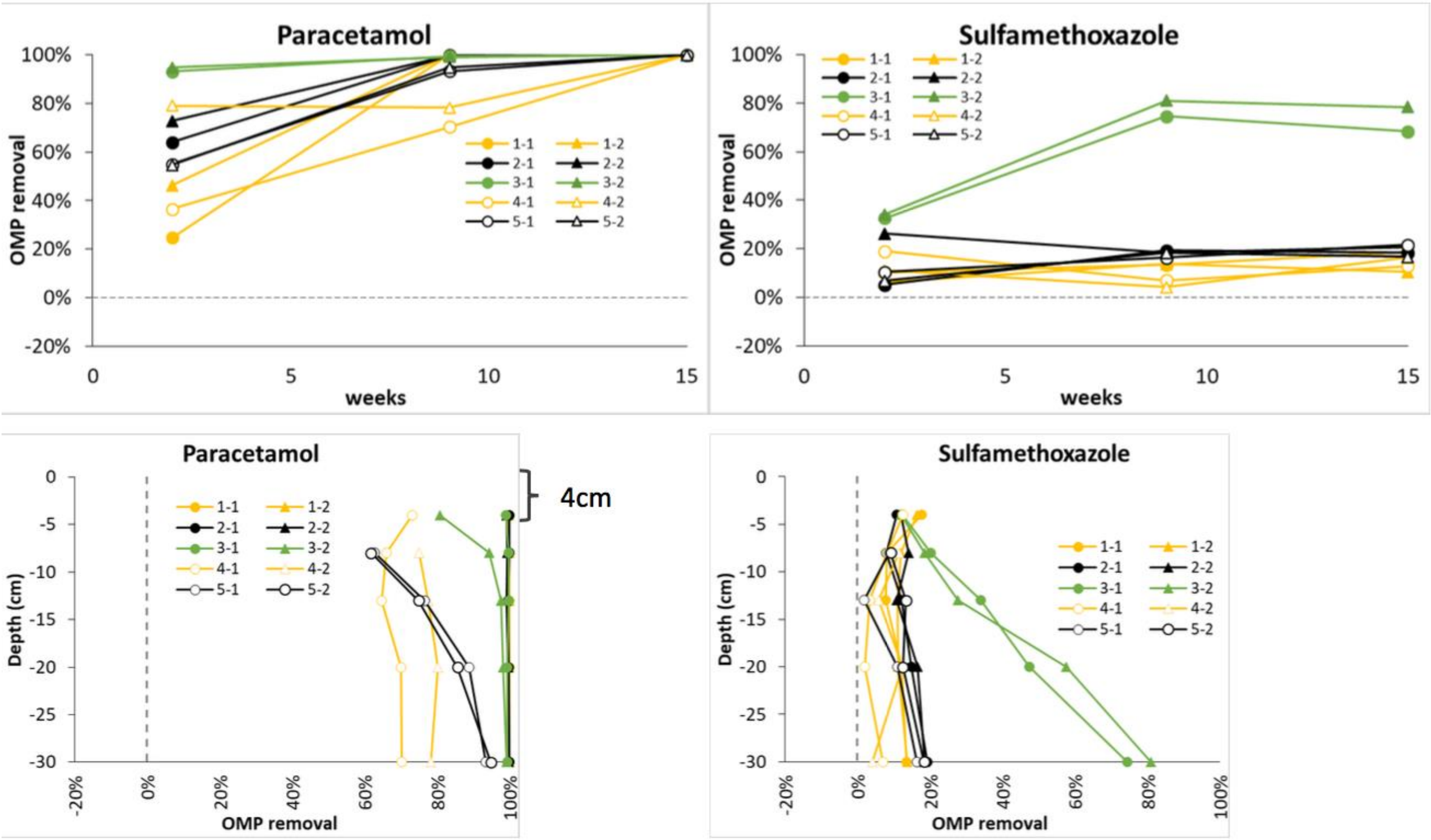


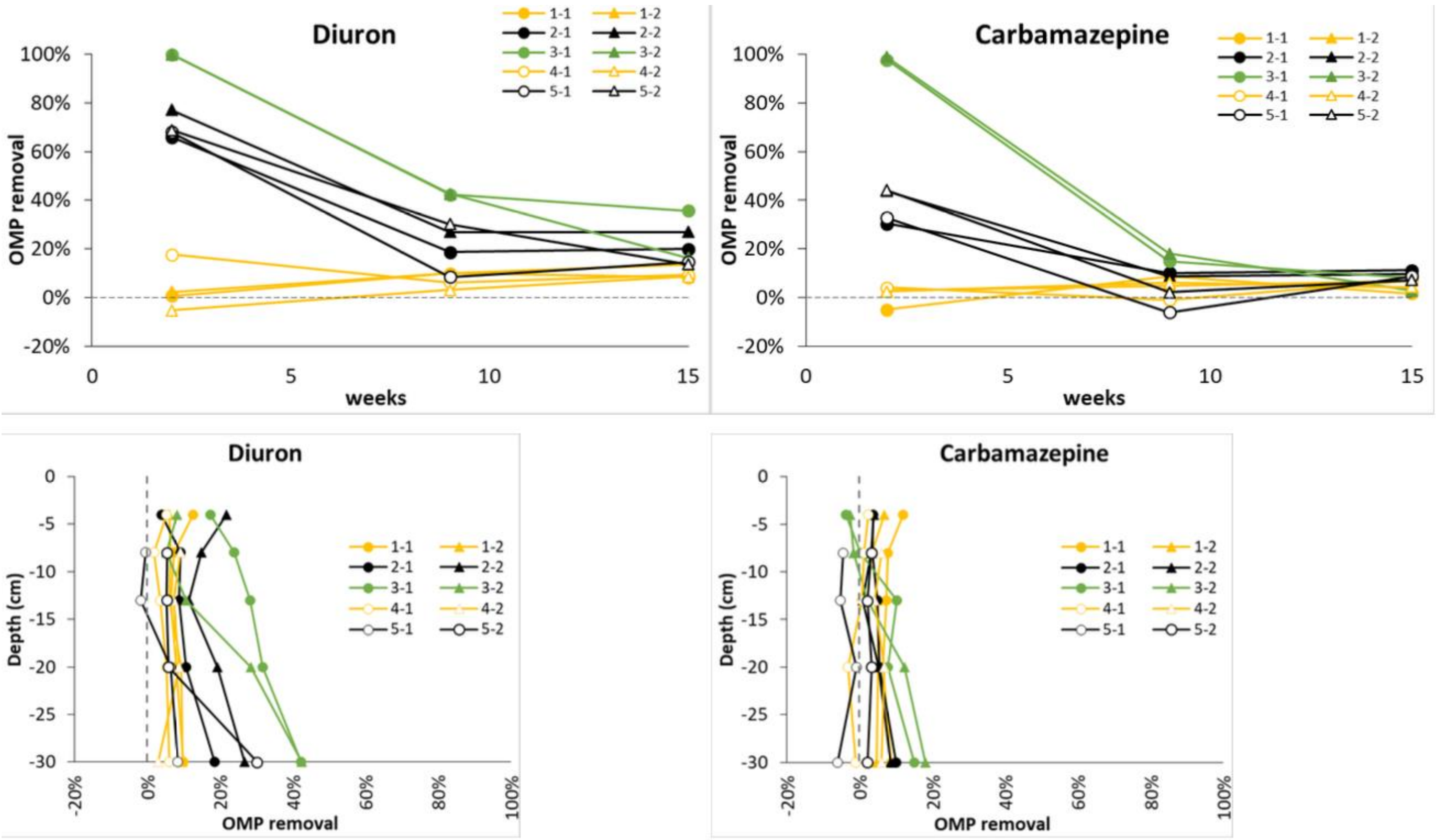


column	material	inoculation	
1-1	sand	yes	
1-2	sand	yes	
2-1	10% compost	yes	
2-2	10% compost	yes	
3-1	50% compost	yes	
3-2	50% compost	yes	
4-1	sand	no	
4-2	sand	no	
5-1	10% compost	no	
5-2	10% compost	no	

Emerging organic compounds involved

Name	Abbreviation	CAS number	Structure	Category
Paracetamol (Acetaminophen)	PAR (APAP)	103-90-2		analgesic
Sulfamethoxazole	SMX	723-46-6		antibiotic, sulfonamide
Carbamazepine	CBZ	298-46-4		psychiatric drug
Diuron	DCMU	330-54-1		Pesticide and biocide

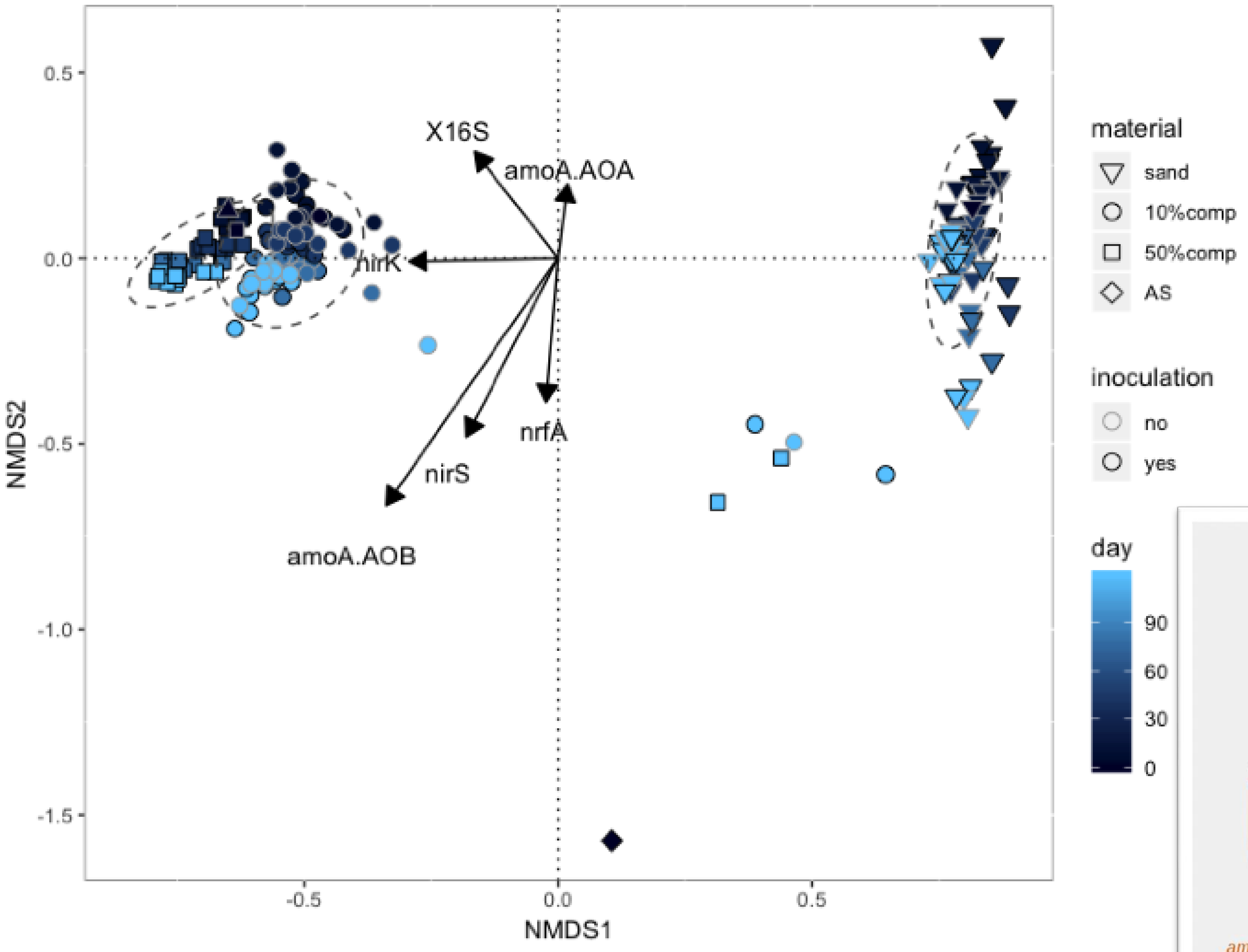

Paracetamol
OMP removal
weeks
Sulfamethoxazole
1-1
1-2
2-1
2-2
3-1
3-2
4-1
4-2
5-1
5-2
Depth (cm)
4cm

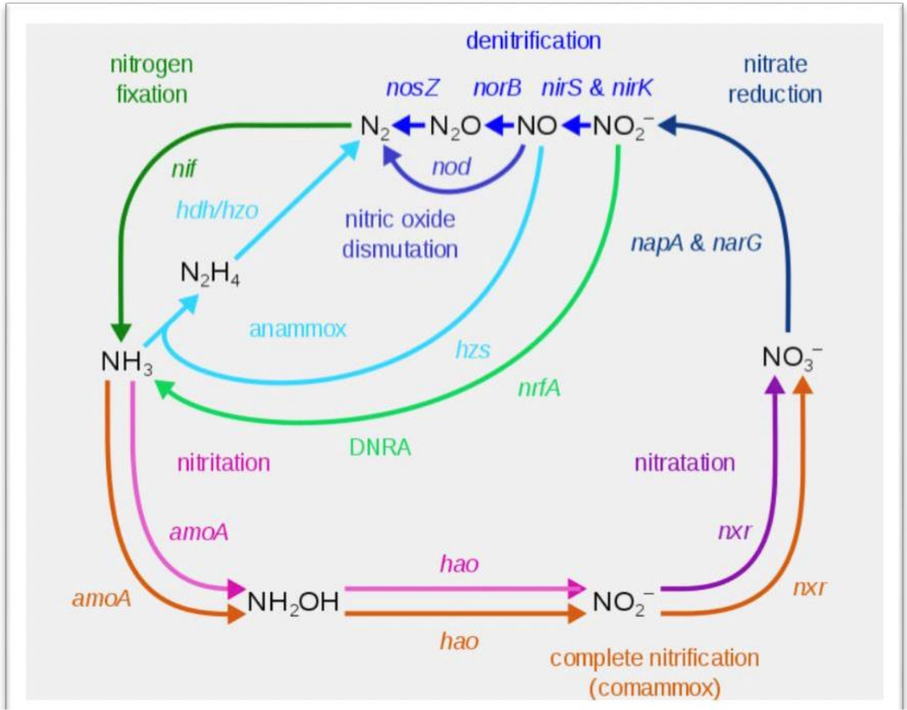


Diuron
Carbamazepine
OMP removal
weeks
Depth (cm)
1-1
1-2
2-1
2-2
3-1
3-2
4-1
4-2
5-1
5-2
100%
80%
60%
40%
20%
0%
-20%
0
5
10
15
-5
-10
-15
-20
-25
-30

Results

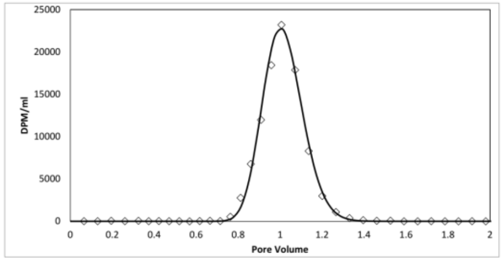
- DNA analyses:
    - microbial community structure (16S sequencing)
    - abundance of N-transformation genes (qPCR)


X16S
amoA.AOA
nirK
nrfA
nirS
amoA.AOB
NMDS1
NMDS2
material
sand
10%comp
50%comp
AS
inoculation
no
yes
day
90
60
30
0

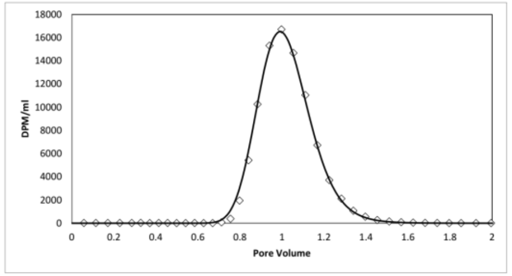

denitrification
nitrogen fixation
nosZ
norB
nirS & nirK
nitrate reduction
N_2
N_2O
NO
NO_2^-
nif
nod
hdh/hzo
nitric oxide dismutation
napA & narG
N_2H_4
anammox
hzs
nrfA
NH_3
NO_3^-
DNRA
nitritation
nitratation
amoA
nxr
hao
NH_2OH
NO_2^-
complete nitrification (comammox)

Conservative tracer tests

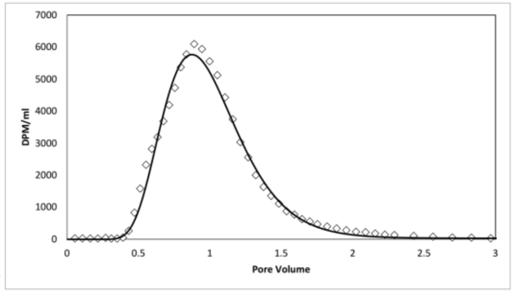
Evaluating the effect of heterogeneity (compost + biofilm) in hydraulic properties.



COLUMN 2.1 – 90% SAND AND 10% COMPOST



COLUMN 3.1 – 50% SAND AND 50% COMPOST



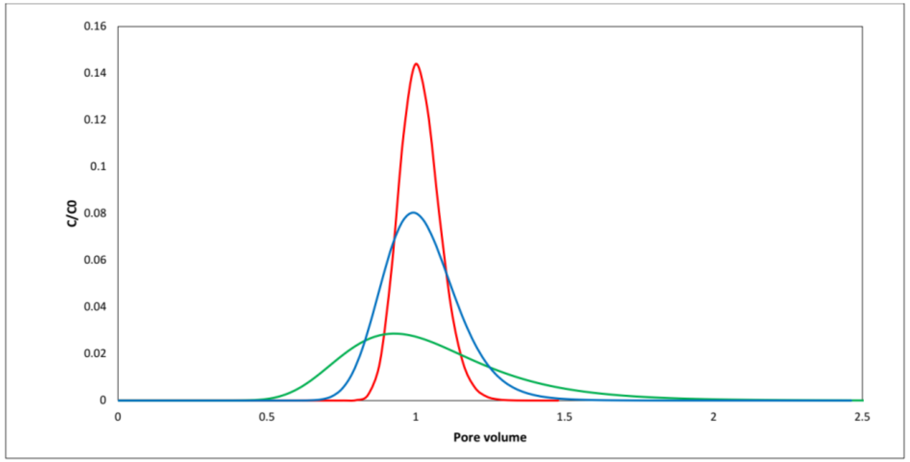
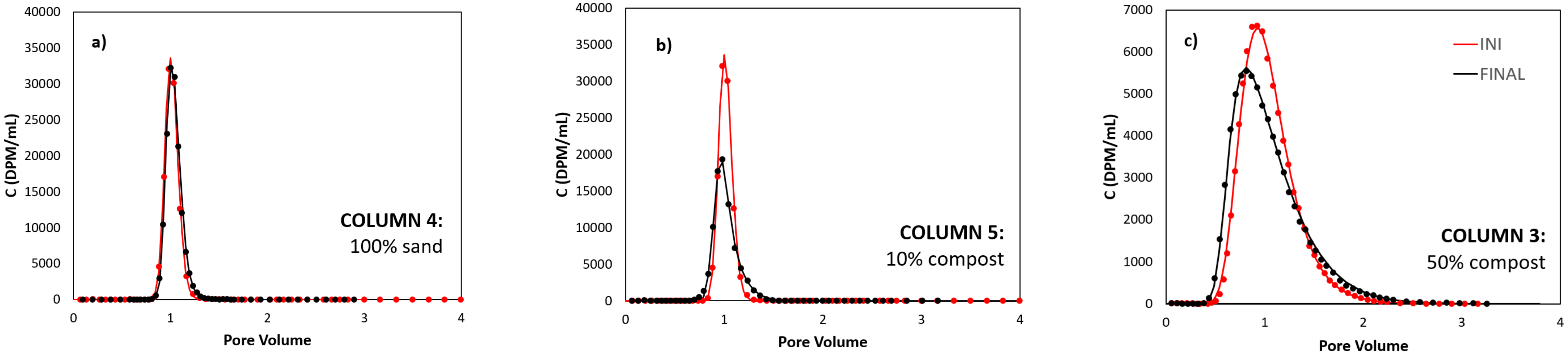


Figure 1. Tracer distribution along pore volume for columns 4.1, 3.2 and 2.1. Red line represents column 4.1, blue line represents column 2.1 and green line represents column 3.2. For C/C0 ratio values look into the following Figures for each featured column.

The little ones…

- **Conservative tracer test model (line) and data (points), at the beginning (red) and at the end of the experiment (black)**
- **Therefore, this is the impact of the formation of biofilm**



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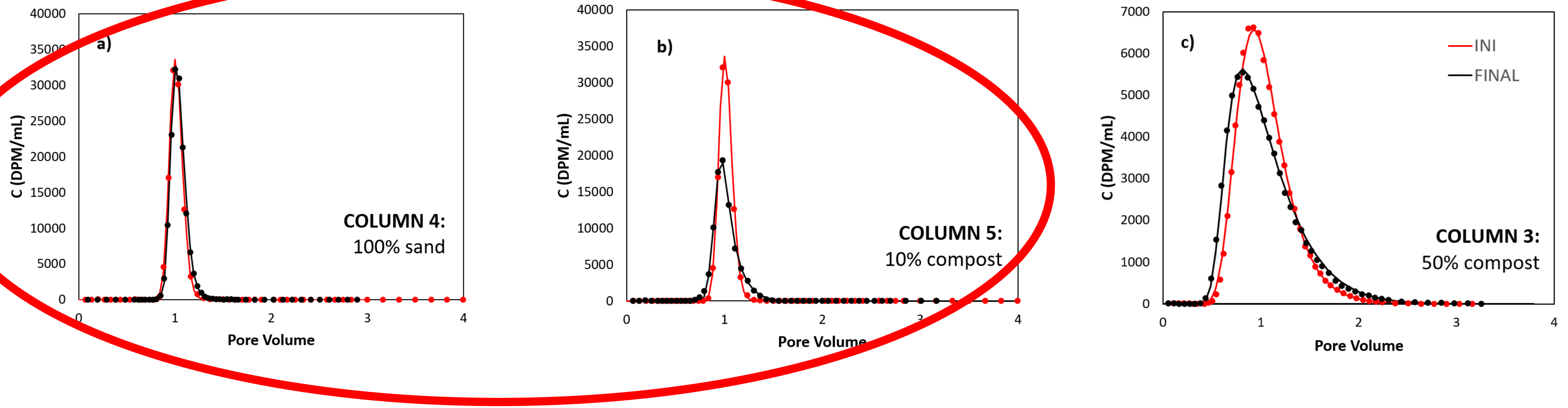


Conservative tracer tests

- **Conservative tracer test equations (advection/dispersion, dual domain parameter and mobile/immobile porosity ratio)**

$$\phi_m \frac{\partial C_{m,i}}{\partial t} = -q \frac{\partial C_{m,i}}{\partial x} + \phi_m \frac{D\partial^2 C_{m,i}}{\partial x^2} - \Gamma_i$$

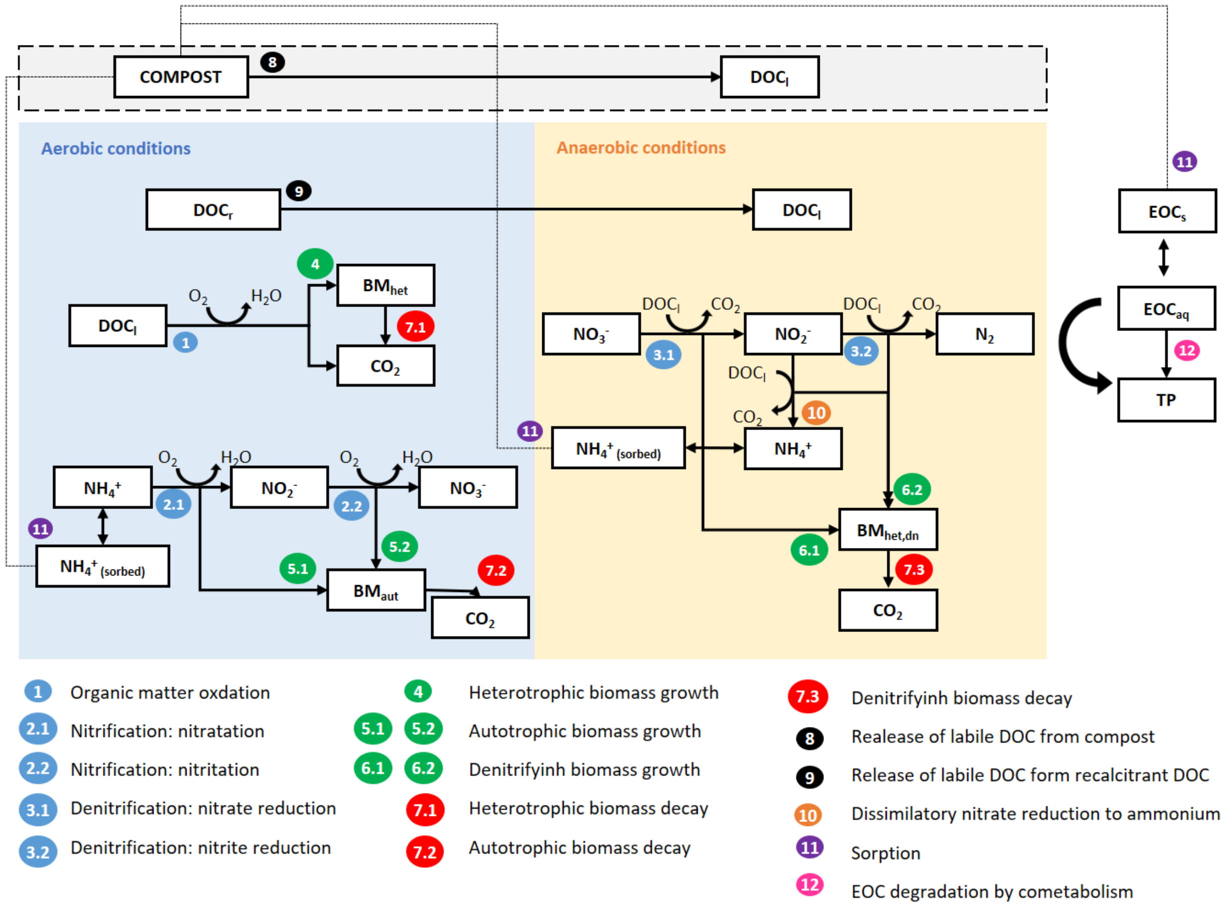
$$\Gamma_i = \alpha\phi_{im}\left(C_{m,i} - C_{im,i}\right)$$

$$\frac{\phi_{im}}{\phi_m} = \frac{\phi_t}{\phi_m} - 1$$

	COLUMN 1 (100% sand)	COLUMN 2 (90% sand, 10% compost)	COLUMN 3 (50% sand, 50% compost)
Porosity	0.424-0.435	0.506-0.518	0.471
Dispersivity (cm)	~0.04	~ 0.20	~0.7
Partioning coeficient	0.93 ± 0.03	0.985±0.019	0.94
Mass transfer coeficient	0.000768 ± 0.000621	0.00005316 ± 0.00000517	0.00008893 ± 0.00015089

Geochemistry mapping

- **We include: Organic matter oxidation, nitrification, denitrification, Dissimilatory nitrate reduction and compost release.**

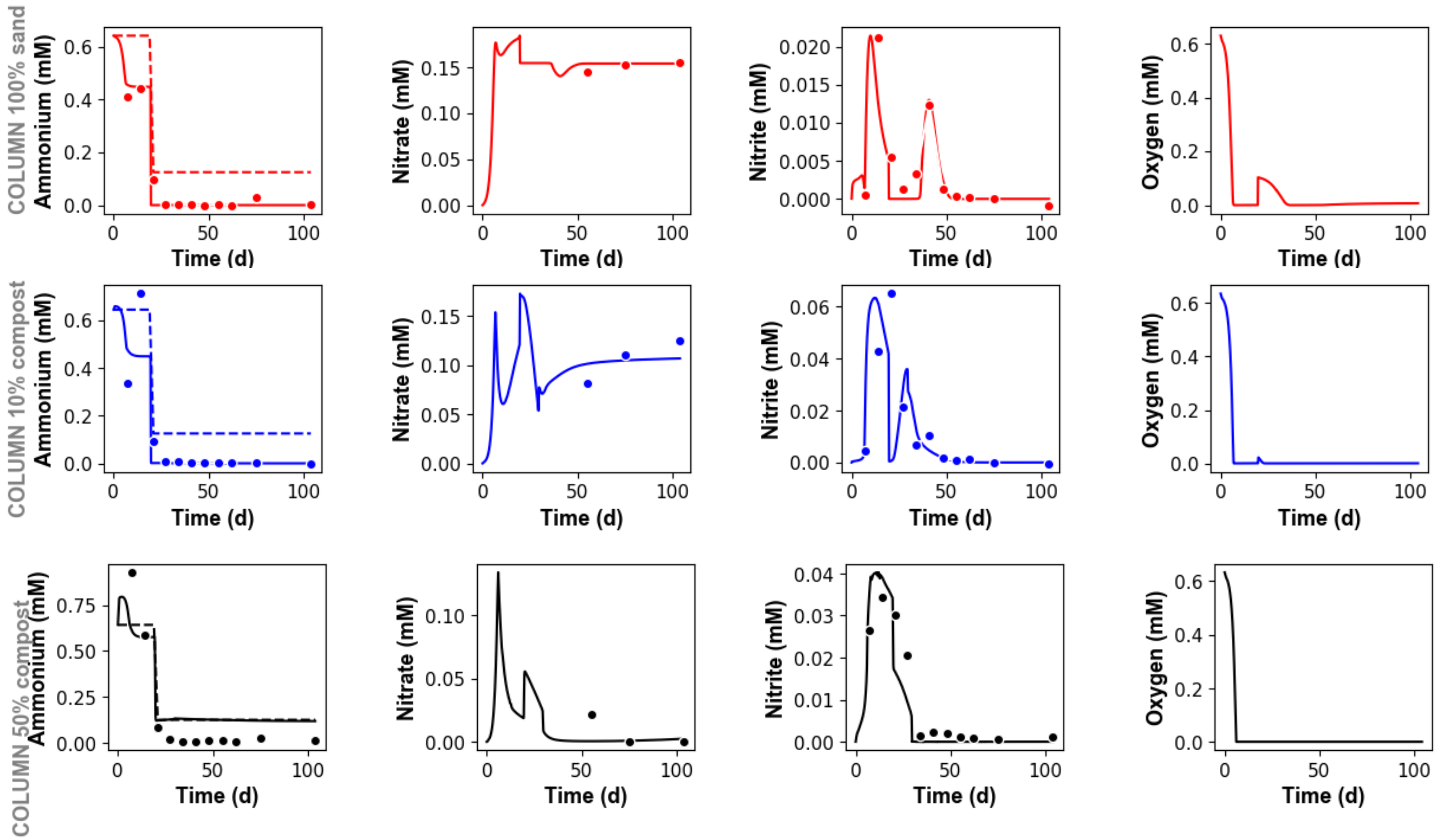


Geochemistry mapping

- **Kinetic equations and stoichiometry for the conceptual model reactions**

Process and reaction	**Process rate**											
	C_{org}	**CO2**	**NH_4^+**	**NO_3^-**	**NO_2^-**	**N_2**	**H^+**	**O_2**	**HCO3-**	**Compost**	**Bio**	
Nitrification (nitritation)	-	$-\frac{0.11056}{5}$	- 0.1722	-	$+\frac{1}{6}$	-	+0.333	$-\frac{0.8894}{4}$	$-\frac{0.11056}{20}$	-	-	$K'_{max}\cdot\frac{[NH4]}{[NH4]+K_{s\,NH4}}\cdot\frac{[O2]}{[O2]+K_{s\,O2}}\cdot Bio_{aut}\cdot\frac{K_{i\,NO2}}{[NO2]+K_{i\,NO2}}$
Nitrification (nitratation)	-	-0.018	-	+0.5	-0.018	-	-0.003	-0.2271	-	-	-	$K'_{max}\cdot\frac{[NO2]}{[NO2]+K_{s\,NO2}}\cdot\frac{[O2]}{[O2]+K_{s\,O2}}\cdot Bio_{aut}\cdot\frac{K_{i\,NO3}}{[NO3]+K_{i\,NO3}}$
Aerobic oxidation of org. matter	-0.25	+0.13	$+\frac{0.6}{20}$	-	-	-	-	-0.1	$+\frac{0.6}{20}$	-	-	$K'_{max}\cdot Bio_{het}\cdot\frac{[Doc]}{[Doc]+K_{s\,Doc}}$
Compost release	+0.95	-	+0.05	-	-	-	-	-	-	-1	-	$r'_{max}\cdot[Compost]$
Denitrification (nitrate reduction)	-0.25	+0.161	-	-0.2679	+0.25	-	-0.018	-	-	-	-	$K'_{max}\cdot\frac{[C_{org}]}{[C_{org}]+K_{s\,Corg}}\cdot\frac{K_{i\,O2}}{[O2]+K_{i\,O2}}\cdot Bio_{het}\cdot\frac{[NO_3^-]}{[NO_3^-]+K_{s\,NO3-}}$
Denitrification (nitrite reduction)	-0.25	+0.154	-	-	-0.186	+0.167	-0.186	-	-	-	-	$K'_{max}\cdot\frac{[C_{org}]}{[C_{org}]+K_{s\,Corg}}\cdot\frac{[NO_2^-]}{[NO_2^-]+K_{s\,NO2-}}\cdot\frac{K_{i\,O2}}{[O2]+K_{i\,O2}}\cdot Bio_{het}\cdot\frac{k_I}{[NO_3^-]+k_I}$
Biomass (auto)	-	-	-	-	-	-	-	-	-0.2	-	+1	$(Y_{nitrite}\cdot NO2-\ rate)+(Y_{nitrate}\cdot NO3-\ rate)-decay\cdot Bio_{aut}$
Biomass (hetero)	-	-	-	-	-	-	-	-	-0.2	-	+1	$(Y_{O2}\cdot O2\ rate)+(Y_{denitit}\cdot Denitit\ rate)+(Y_{denitat}\cdot Denitat\ rate)+(Y_{DNRA}\cdot DNRA\ rate)-decay\cdot Bio_{het}$
DNRA	-2	+2	+1	-1	-	-	-	-	-	-	-	$K'_{max}\cdot\frac{[C_{org}]}{[C_{org}]+K_{s\,Corg}}\cdot\frac{K_{i\,O2}}{[O2]+K_{i\,O2}}\cdot Bio_{het}\cdot\frac{[NO_3^-]}{[NO_3^-]+K_{s\,NO3-}}$

Geochemistry mapping. Model results



Reactive transport. EOCs

- **Reactive transport model equations for the sorption model of the EOCs.**

$$\boxed{1} \quad \alpha_{EOCs} = \frac{1}{1 + K_a / [H^+]}$$

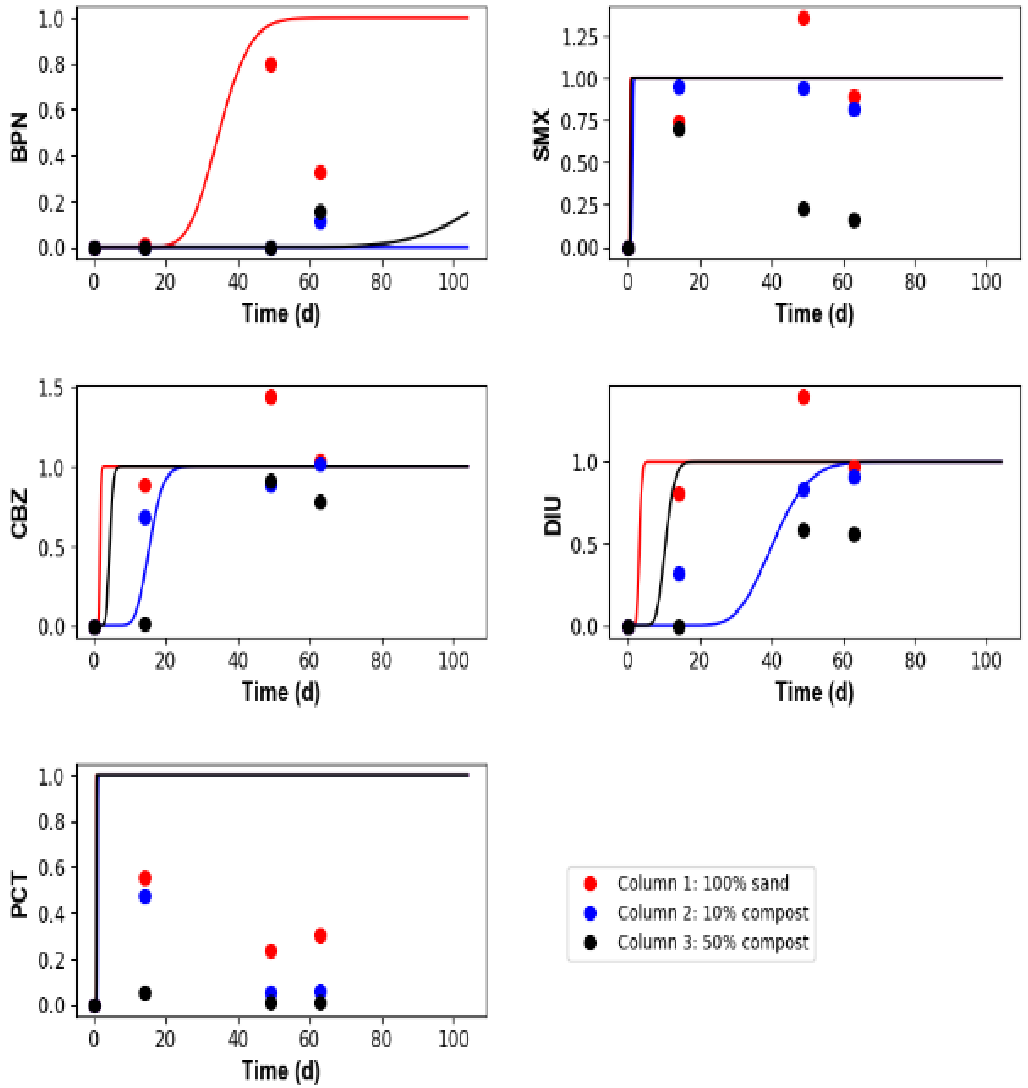
$$\boxed{2} \quad K_d = K_{oc} \times f_{om}$$

$$\boxed{3} \quad K_{d_{EOC,TOT}} = \sum_{i=1}^{i} K_{d_i}$$

$$\boxed{4} \quad \log K_{d,PHREEQC} = \log K_{oc} - \log m_{org} = \log K_{oc} - \log(10^{100})$$

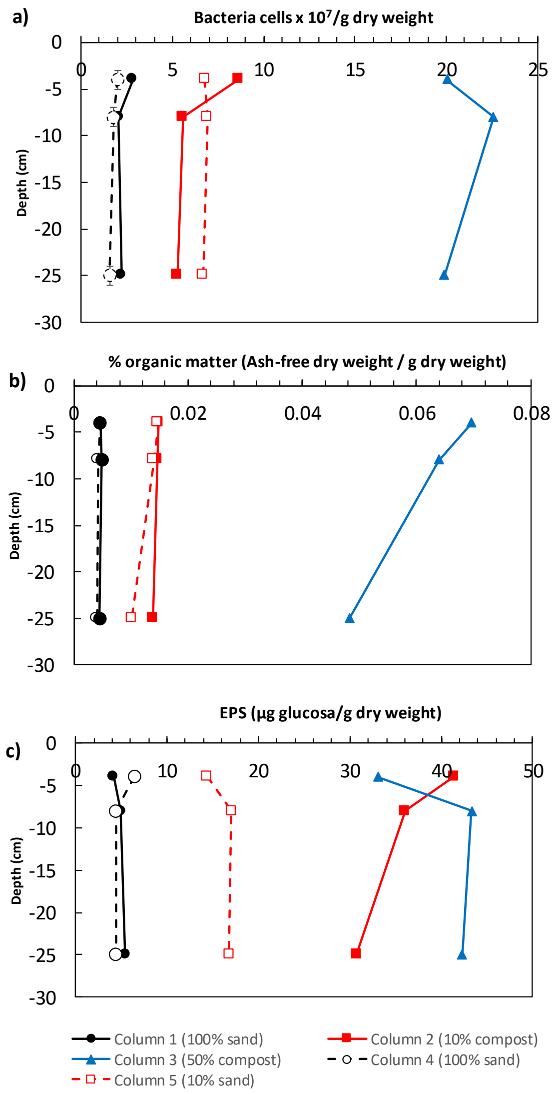
EOCs equations for the fraction of neutral compound (1), specific sorption partitioning (2), total sorption partitioning (3) and total sorption to organic matter (4)

Reactive transport. EOCs



But we love challenges…

- **Data for percentage of organic matter, EPS content and bacteria quantification**



Conclusions

Plenty of them; it was a 3-y project with several partners and large amount of work

Messages to convey

Flow and transport in porous media are difficult to understand because there are a myriad of processes taking place simultaneously that are correlated

Studying water quality evolution needs to incorporate in an integrated way hydro-bio-geo-chemical data, and it should be interpreted together.

Degradation of a compound is affected by the presence of other compounds, the redox state (driven by surface processes of recharge, presence of OM, biomass existence or evolution), the pH, mineralogy, and obviously flow pattern.

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Fun for years to come, and more research (and money) is needed