



**IMT Lille Douai**  
École Mines-Télécom  
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# A methodological proposal for the ecotoxicological characterization of continental and marine sediments for recovery

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# 1. CONTEXT AND AIM OF THE STUDY

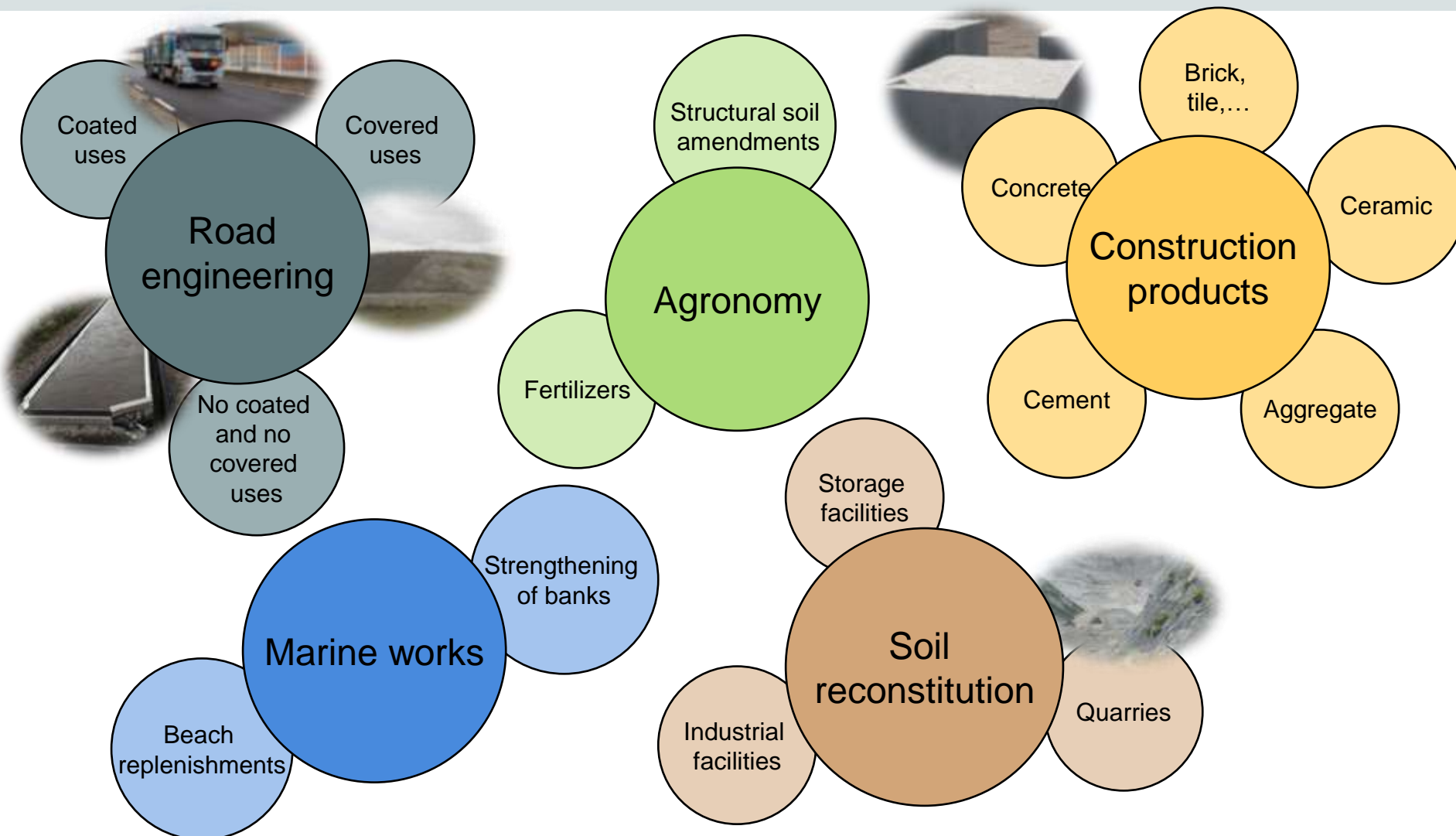


# General context

- ✓ Volume of sediments dredged and managed on-shore:
  - For French seaports →
    - 40 to 50 M m<sup>3</sup> dredged for 5 to 7 M m<sup>3</sup> managed on shore each year**
  - For French inland waterways sector →
    - on average 6 M m<sup>3</sup> for 525 000 kilometers of waterways**
  - Dams on a case-by-case basis
  
- ✓ When sediments are extracted from aquatic environment, they are considered as **waste**
  
- ✓ Sediments are classified between 3 **waste categories**:



# General context



**According to the circular of 4 July 2008: the potential reuse of dredge sediments is allowed provide that the contracting authority demonstrates environmental harmlessness for the proposed use**

# Sediments potential ecotoxicity

Sediment ecotoxicity is taken into account specifically:

- At the intrinsic hazard characterization level: **HP14 protocol (France)**
- At the environmental and health risk assessment level: e.g. **SEDIGEST project**

HP14 protocol carried out on **47 marine sediments** and **27 continental sediments**:

Variation of thresholds in TU	Microtox threshold: 10TU	<i>Brachionus</i> threshold: 100TU	Cultivated oat germination thresholds: 10TU	Cultivated oat Growth threshold: 10TU
	% of sediments classified as hazardous based on the selected threshold			
Current thresholds	0	1,4	5,4	5,4
2 x as strict	0	2,8	24,3	25,7
3 x as strict	0	8,1	43,2	41,9
5x as strict	5,4	9,5	50,0	51,4

(BRGM ,Mouvet 2013)



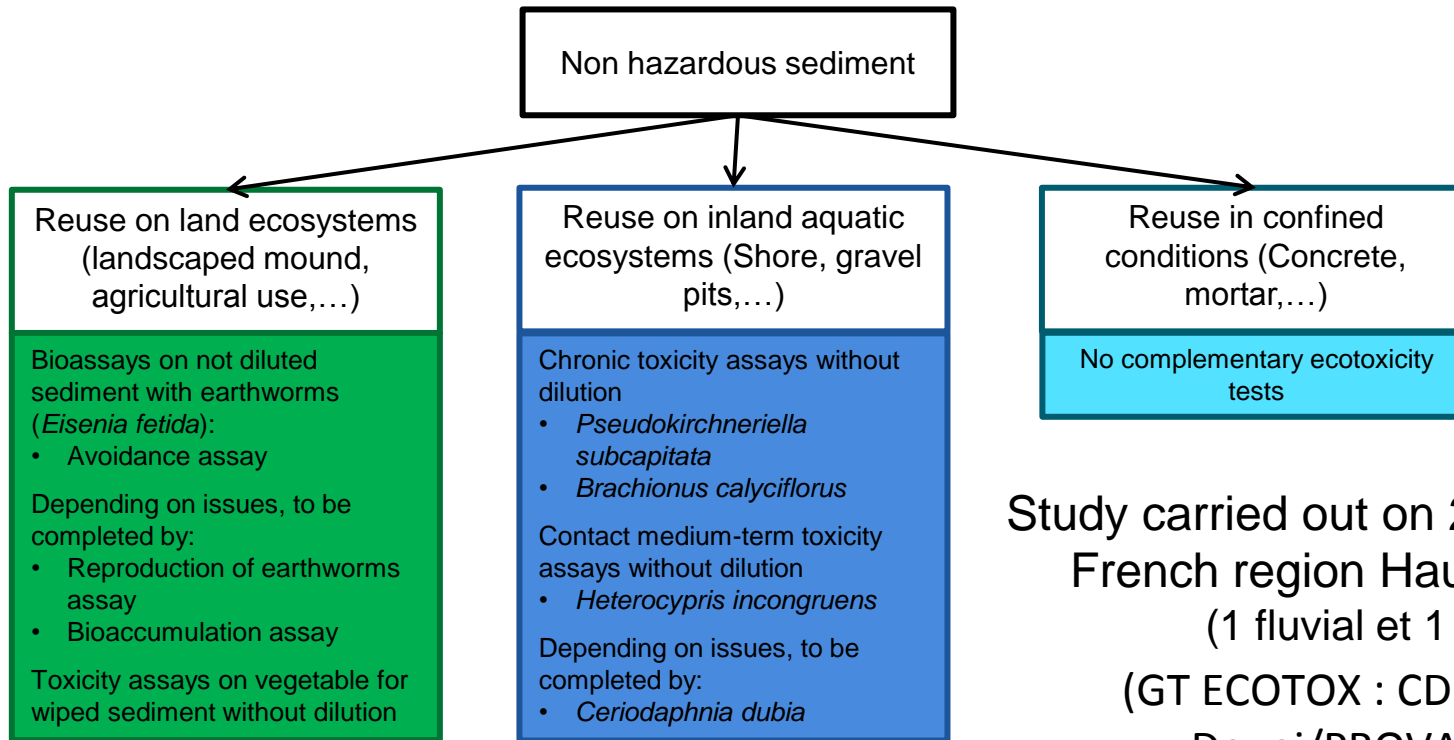
Sediment ranking is very sensitive to the threshold values for the various ecotoxicological tests of the battery tested



Ranking in « non-hazardous waste » **But** there are residual toxic effects

# Study initiated in 2014 as part of the SEDIMATERIAUX approach for the ecotoxicological characterization of non-hazardous sediments

Proposal for screening of sediments intrinsic ecotoxicity based on various reuse scenarios



Study carried out on 2 sediments from French region Hauts-de-France (1 fluvial et 1 marine)  
(GT ECOTOX : CD2E /IMT Lille Douai/PROVADEMSE)

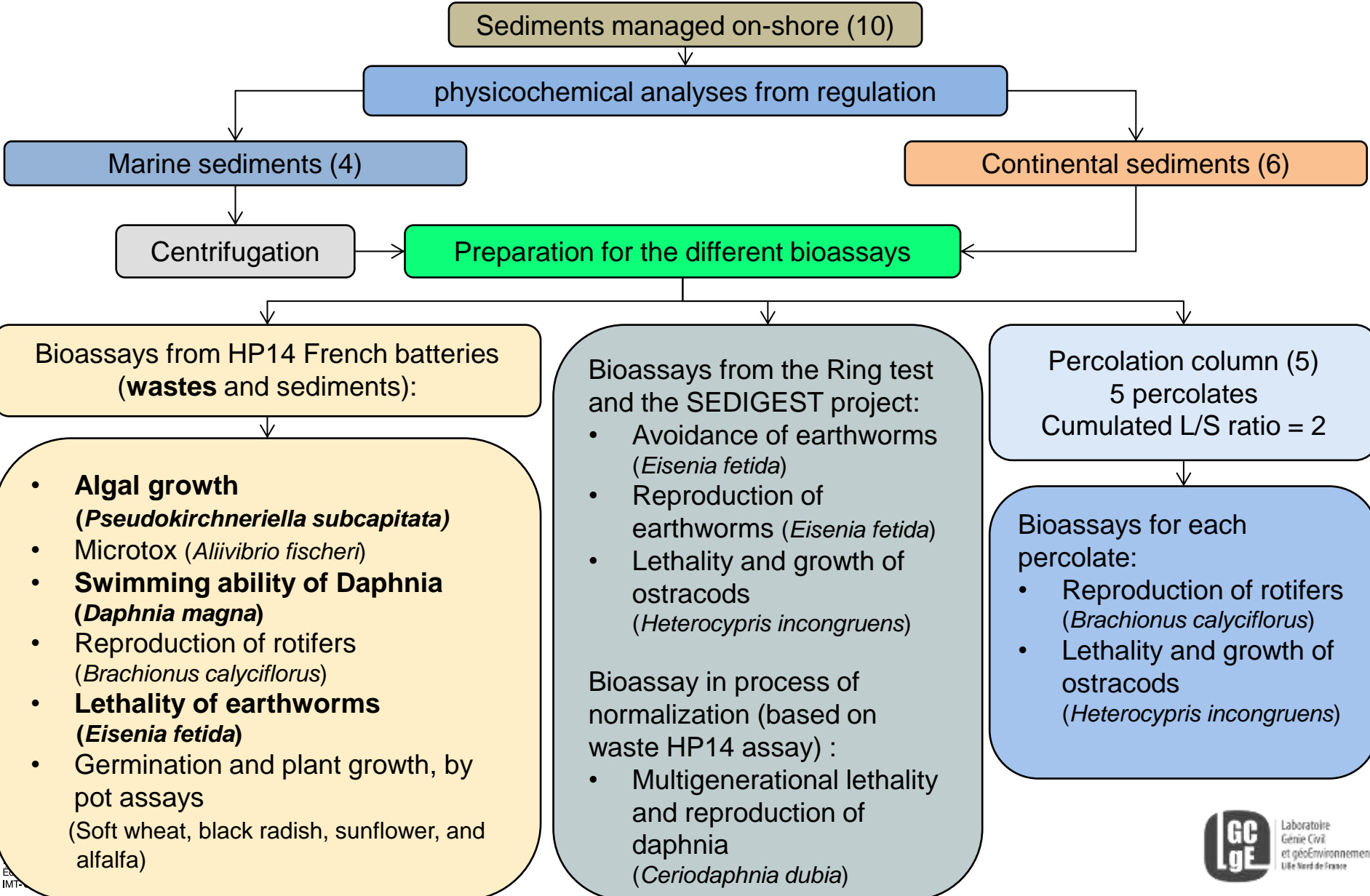
## Aim:

Development of a battery of bioassays for screening the **intrinsic ecotoxicity of non-hazardous sediments** likely to be reuse on land as part of **voluntary approaches** such as **SEDIMATERIAUX**

## 2. EXPERIMENTAL APPROACH



# Experimental approach





# 3. PHYSICOCHEMICAL CHARACTERIZATION



# Physicochemical characterization

## 4 fluvial sediments

- SF1 EDF 2015
- SF2 MEL 2014
- SF3 VNF 2015
- SF4 Deûle 2016

## 1 lake sediment

- SL Roubaix 2016

## 1 dam sediment

- SB EDF 2015

## 4 marine sediments

- SM1 GPMD 2014
- SM2 GPMD 2015
- SM3 GPMD « butte » 2014
- SM4 GPMD « traité » 2015

sediments	SF1	SF2	SF3	SF4	SB	SL	SM1	SM2	SM3	SM4	S1 Threshold	Hazardous threshold « worst-case »
As	4,2	7,1	7,0	99,4	17,0	8,4	24,7	25,0	15,8	4,2	30	330
Cd	0,2	3,3	0,8	306,4	0	0,5	2,0	2,0	1,1	< 0,07	2	530
Cr	35,5	102,8	56,2	63,9	44	69,3	94,8	82,0	54,4	93,8	150	250*
Cu	19,4	189,7	67,5	117,7	29,1	28,6	57,0	68,0	43,6	35,5	100	4000
Hg	< 0,05	3,5	< 0,05	23,1	< 0,05	< 0,05	< 0,05	< 0,05	0,1	0,2	1	500
Ni	23,1	48,4	25,5	27,1	42,1	35,8	20,8	21,0	15,3	46,1	50	130
Pb	13,9	610,4	82,6	3646,2	18,8	64,5	185,7	261,0	247,1	52,0	100	1000
Zn	93,9	1450,9	395,7	4194,2	109,1	160	793,9	613,0	378,6	202,6	300	7230
Total PCB	< 0,01	0,4	0,02	0,03	NA	< 0,01	< 0,07	0,05	0,1	< 0,01	0,68	50
Total HAP	1,2	25,4	9,0	NA	0,2	5,0	4,7	6,7	3,8	2,7	22,8	500
% < 63µm	70,6	48,2	78,5	67,7	92,8	96,0	78,8	49,4	38,9	20,2		
Q <sub>sm</sub>	0,21	2,16	0,53	23,22	0,32	0,42	1,02	0,94	0,63	0,44		

\*Cr VI

$$Q_{sm} = \frac{\sum \frac{C_i}{S_i}}{n}$$

1<sup>st</sup> approach: Hazardous for HP4, HP5, HP6, HP7, HP8, HP10, HP11, HP13 criteria (INERIS « worst-case ») : **SF4** (Pb 3646,2 mg/kg)

**All the sediments studied aren't hazardous to the HP14 property**

## S1 threshold

- SF2
- SF3
- SF4
- SM1
- SM2
- SM3

Need to apply  
the HP14  
battery

## Qsm

- SF2
- SF3
- SF4
- SM1
- SM2
- SM3

Upper to Qsm

## Inert threshold

- SF2
- SF4
- SL
- SM1
- SM2
- SM3

Non inert  
sediments

## Hazardousness

- SF4\*

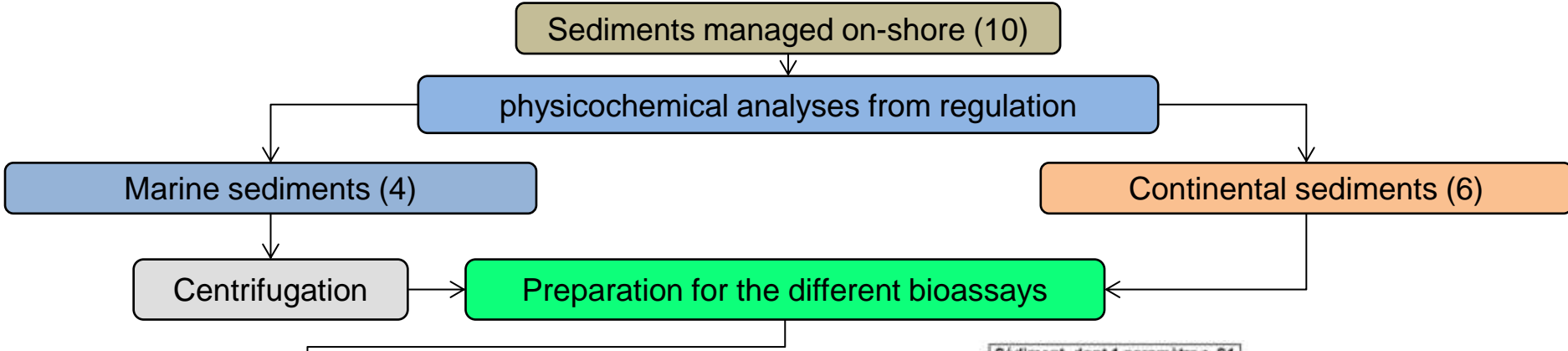
Hazardous  
sediment

\*for the properties HP4 à HP8, HP10,  
HP11, HP13 (INERIS « worst-case »)

# 4. APPLICATION OF HP 14 PROTOCOLS FOR RESIDUAL TOXICITY DETECTION

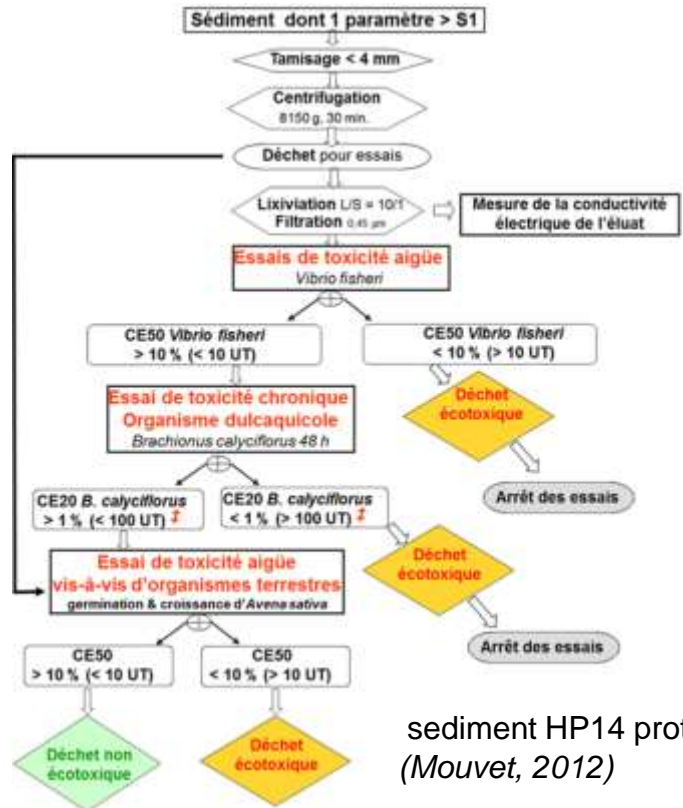


# Methodological approach



Bioassays from HP14 French batteries (wastes and sediments):

- **Algal growth** (*Pseudokirchneriella subcapitata*)
- Microtox (*Aliivibrio fischeri*)
- **Swimming ability of Daphnia** (*Daphnia magna*)
- Reproduction of rotifers (*Brachionus calyciflorus*)
- **Lethality of earthworms** (*Eisenia fetida*)
- Germination and plant growth, by pot assays (Soft wheat, black radish, sunflower, and alfalfa)



sediment HP14 protocol (Mouvet, 2012)

# Results : Bioassays from HP14 battery

- Eluate assays**
- Inhibition of algal population growth *Pseudokirchneriella subcapitata* (NF EN ISO 8692)
  - Bacterial luminescence inhibition testing *Aliivibrio fischeri* (Microtox) (NF EN ISO 11348-3)
  - Inhibition of the mobility of *Daphnia magna* (NF EN ISO 6341)
  - Inhibition of reproduction of rotifers *Brachionus calyciflorus* (NF ISO 20666)

→ Very low toxic effects observed  
 → Not discriminating bioassays

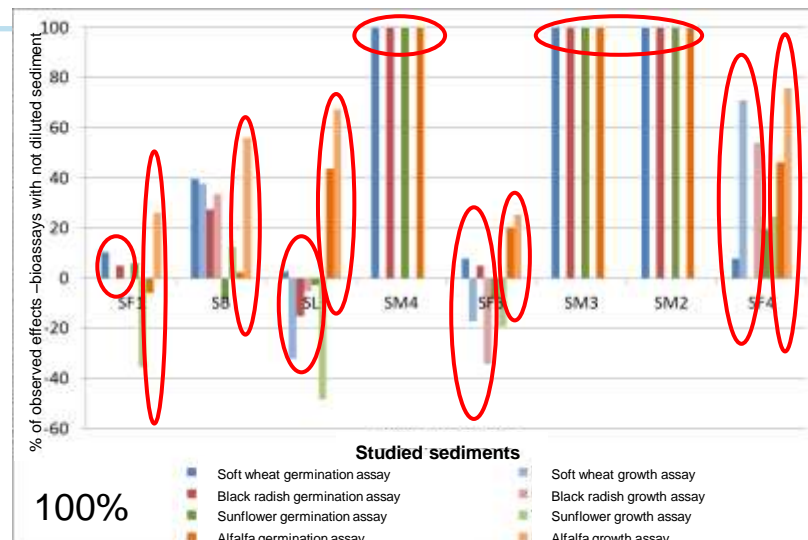
No toxic effects observed  
 → Not discriminating bioassay

Measured and consistent toxic effects  
 → **Relevant test for bioassay battery**

- Solid assays**
- Lethality of earthworms *Eisenia fetida* (NF ISO 11268-1)
  - Germination and plant monocot and dicot growth (Soft wheat, black radish, sunflower, and alfalfa) (NF EN ISO 11269-2)

→ No toxic effects observed for 10%, 100%: max. marine sediments, significant for SF4  
 → Not discriminating bioassay

SM : no germination, no growth



Effects measured: SF, SB, SL (no apparent correlation with measured contamination)

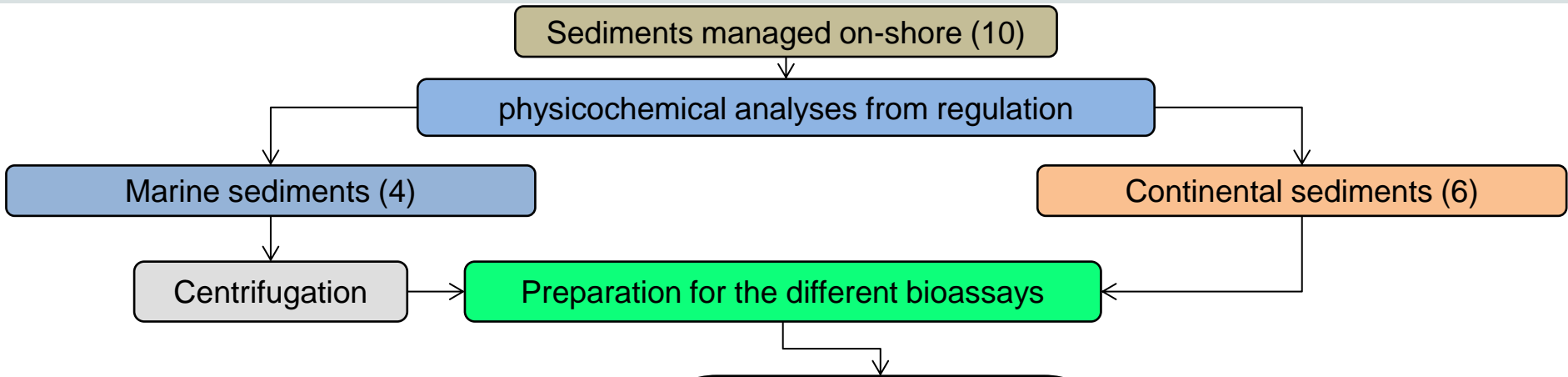
Similar effects for wheat and radish but more consistent for wheat for SF4

Alfalfa more sensitive  
 → **The selected species are soft wheat and alfalfa**

# 5. COMPLEMENTARY TESTS



# Methodological approach



Generation 1 on 10 days  
neonates from the 7<sup>th</sup> day

Generation 2 on 10 days  
neonates from the 7<sup>th</sup> day

Generation 3 on 10 days  
neonates from the 7<sup>th</sup> day

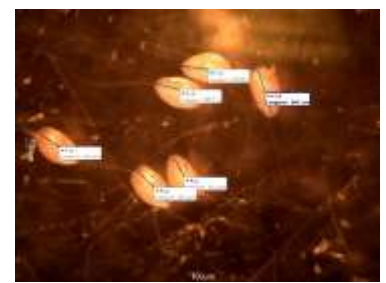
Tests carried out on batch  
eluates because of the  
medium renewal time

Bioassays from the Ring test and the SEDIGEST project:

- Avoidance of earthworms (*Eisenia fetida*)
- Reproduction of earthworms (*Eisenia fetida*)
- Lethality and growth of ostracods (*Heterocypris incongruens*)

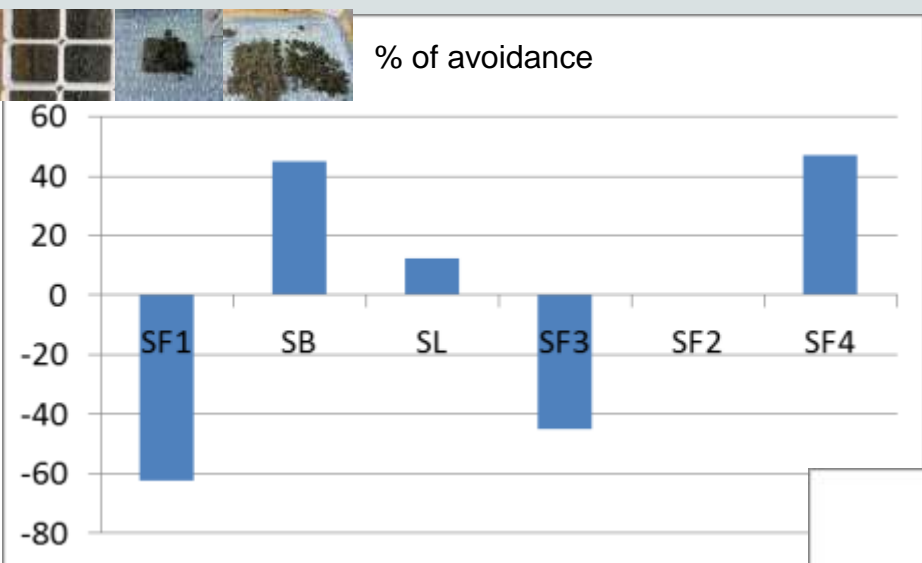
Bioassay in process of normalization (based on waste HP14 assay):

- Multigenerational lethality and reproduction of daphnia (*Ceriodaphnia dubia*)





# Results : Complementary tests carried out on *Eisenia fetida*



- SF4 repelling: known contamination
- SB and SL repelling: Hypotheses of not analyzed contaminants, texture (SB),...
- SF1 and SF3 attraction : nutrients relative to control soil (ISO)

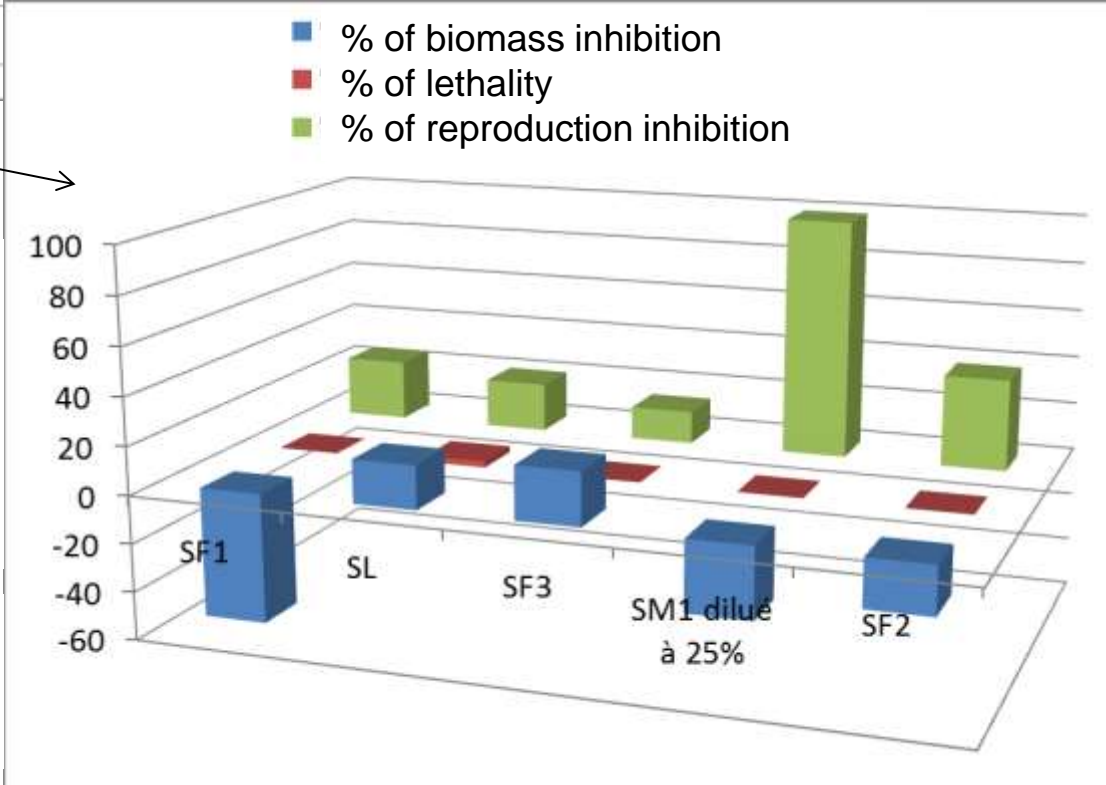
SM: max effects → Biased by the presence of NaCl despite the initial centrifugation

→ relevant test **but** requires adaptations in its implementation

## Reproduction of earthworms *Eisenia fetida* (5 replica by condition)

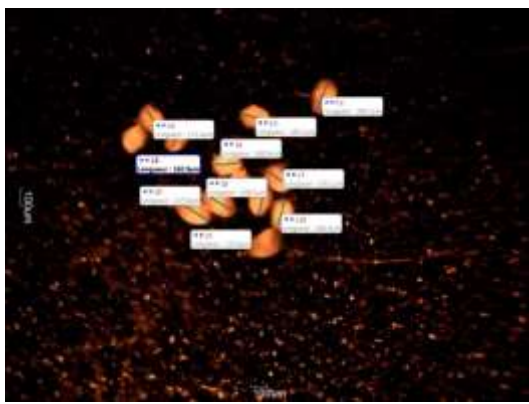
- NaCl effects: diluted SM1
- Biomass intake not correlated with contamination
- No observed lethality (Coherent with the HP14 results)
- Reproduction effects correlated in the contamination (ACP confirmation)

→ Relevant test except SM **But** requires adaptations in its implementation (preparation of matrices and control)



# Results : Complementary tests carried out on ostracods

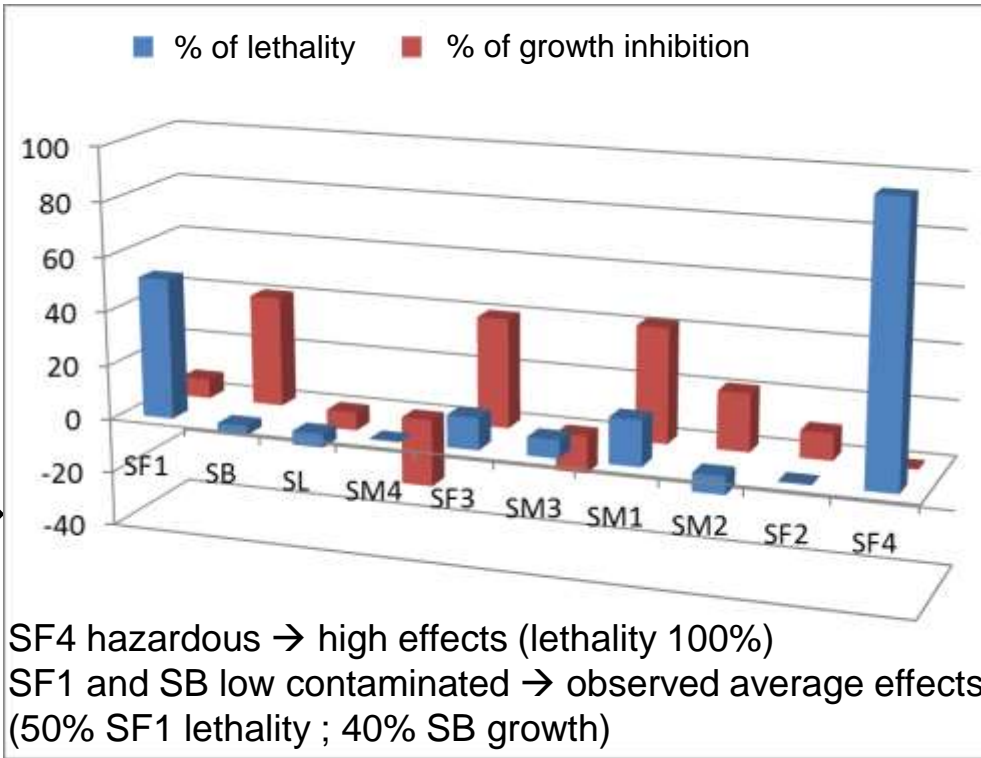
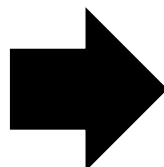
## Lethality and growth of ostracods *Heterocypris incongruens* (NF ISO 14371)



Measuring the initial size of ostracods (10)



Measurement of surviving ostracods size after 6 days of incubation



SF4 hazardous → high effects (lethality 100%)  
 SF1 and SB low contaminated → observed average effects (50% SF1 lethality ; 40% SB growth)

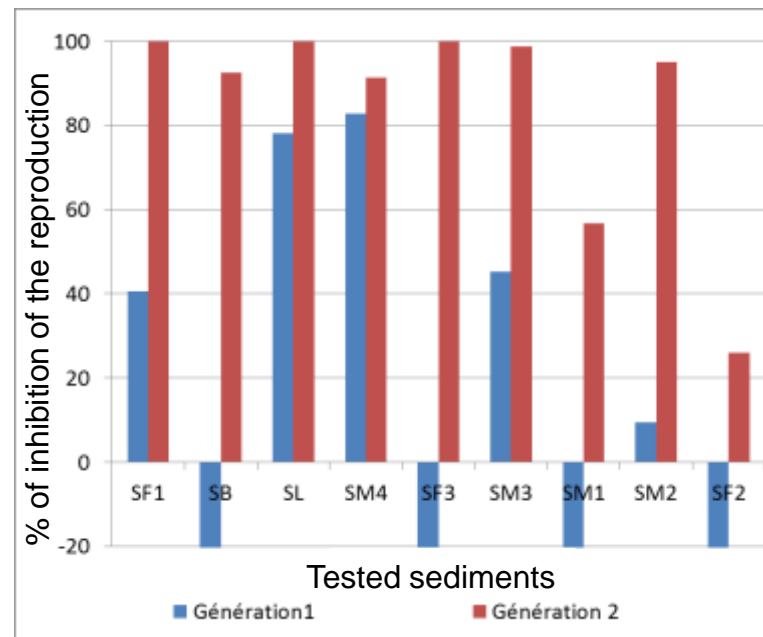
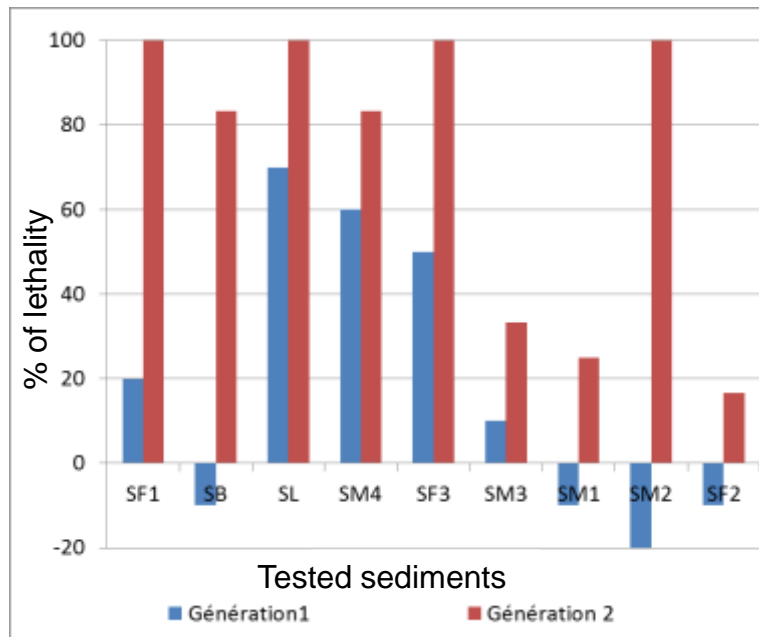
**Average to low observed effects in disorder compared to chemical contamination ranking**

**The test is sensitive to contamination of sedimentary matrices : relevant test for SF, SB, SL, and SM**

# Results : Complementary tests carried out on *Ceriodaphnia dubia*

## Multigenerational lethality and reproduction of daphnia test

(based on NF ISO 20665)



No 3<sup>rd</sup> generation due to poor generation 2 reproduction on the 7<sup>th</sup> day

Increased effects observed for the 2<sup>nd</sup> generation for all sediments tested

Including when there are no effects for the first generation ←

→ Unanalyzed contaminants (organic substances?), cocktail effect, etc...

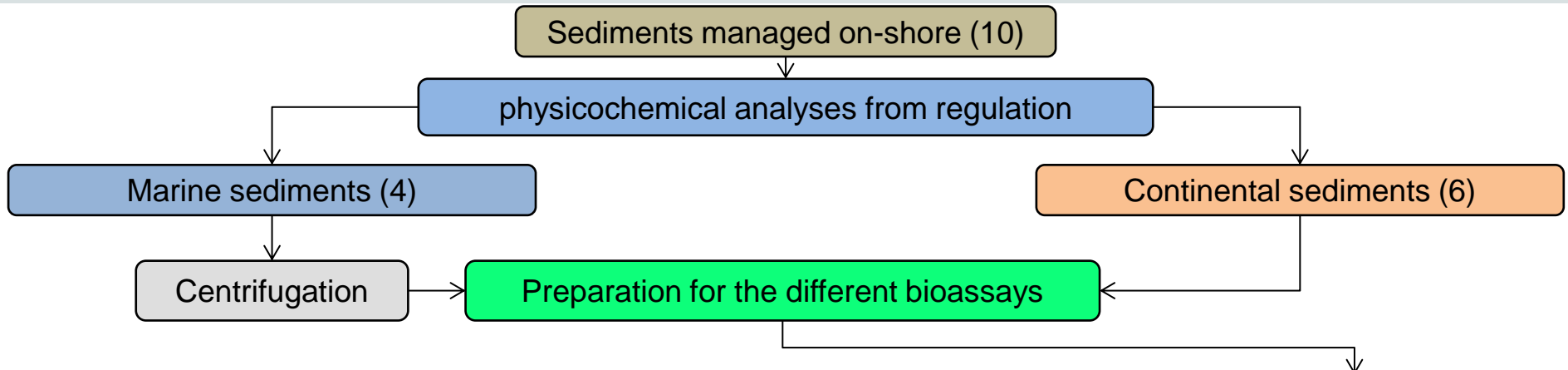
### Relevant test for the bioassay battery

Recommendation of the test on undiluted and diluted eluates to X%  
(to be defined)

# 6. TESTS WITH THE PERCOLATION COLUMN LEACH PROCESS



# Methodological approach



Eluate tests with column extraction

↳ Different release over longer term  
 ➔ Potential of mobilization of contaminants

Percolation column (5)  
 5 percolates  
 Cumulated L/S ratio = 2

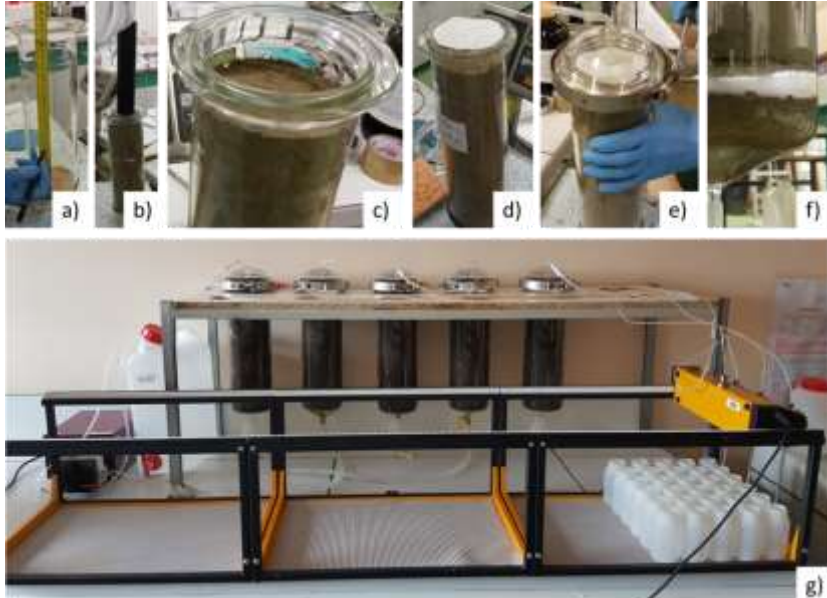
Assays for each percolate:

- Reproduction of rotifers (*Brachionus calyciflorus*)
- Lethality and growth of ostracods (*Heterocypris incongruens*)

Percolate 1	•L/S ratio: 0,1 •Volume: 1 L
P2	•L/S ratio: 0,2 •Volume: 1 L
P3	•L/S ratio: 0,5 •Volume: 3 L
P4	•L/S ratio: 1 •Volume: 5 L
P5	•L/S ratio: 2 •Volume: 10 L

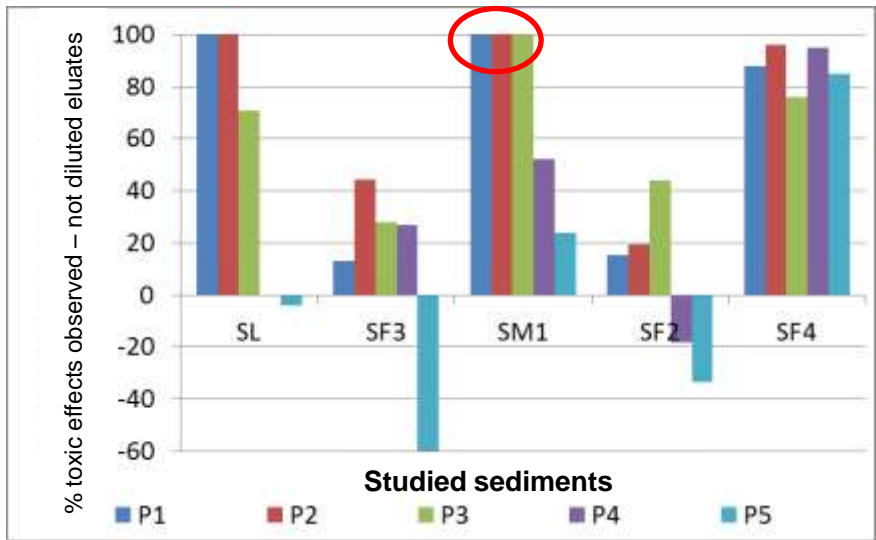
Volumes for ecotoxicological assays ➔

Column: height 30 cm ; diameter 20 cm



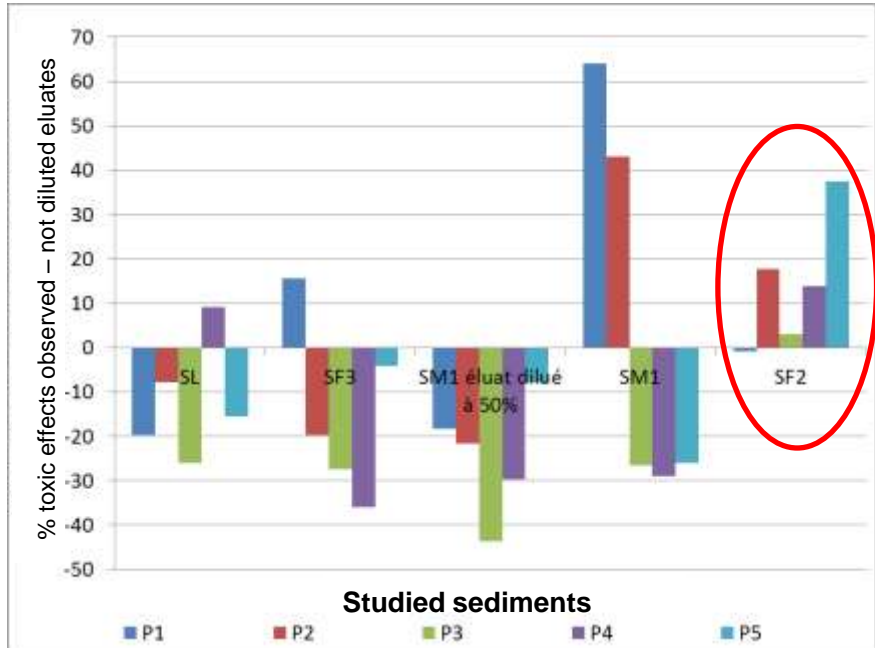
# Results of bioassays with column percolation

## Reproduction of rotifers on *Brachionus calyciflorus* test (8 Replica)



Lethality with SM1 undiluted P1, P2 and P3  
 Observed effects tend to decrease with increase in L/S ratio  
 BUT in general, salts are more mobilized with this extraction method  
 On the first percolates, more important toxic effects than batch testing are observed

➔ Possibility to study toxicity more finely



## Lethality and growth of ostracods test *Heterocypris incongruens* (6 Replica)

Max. lethality to SF4 (like tested on sediment)

Effects observed on SF2 increase with L/S ratio  
 ➔ Higher than on sediment

Observed toxic effects SL and SF3 lower than on sediment

As rotifer test results, salt mobilization to SM1

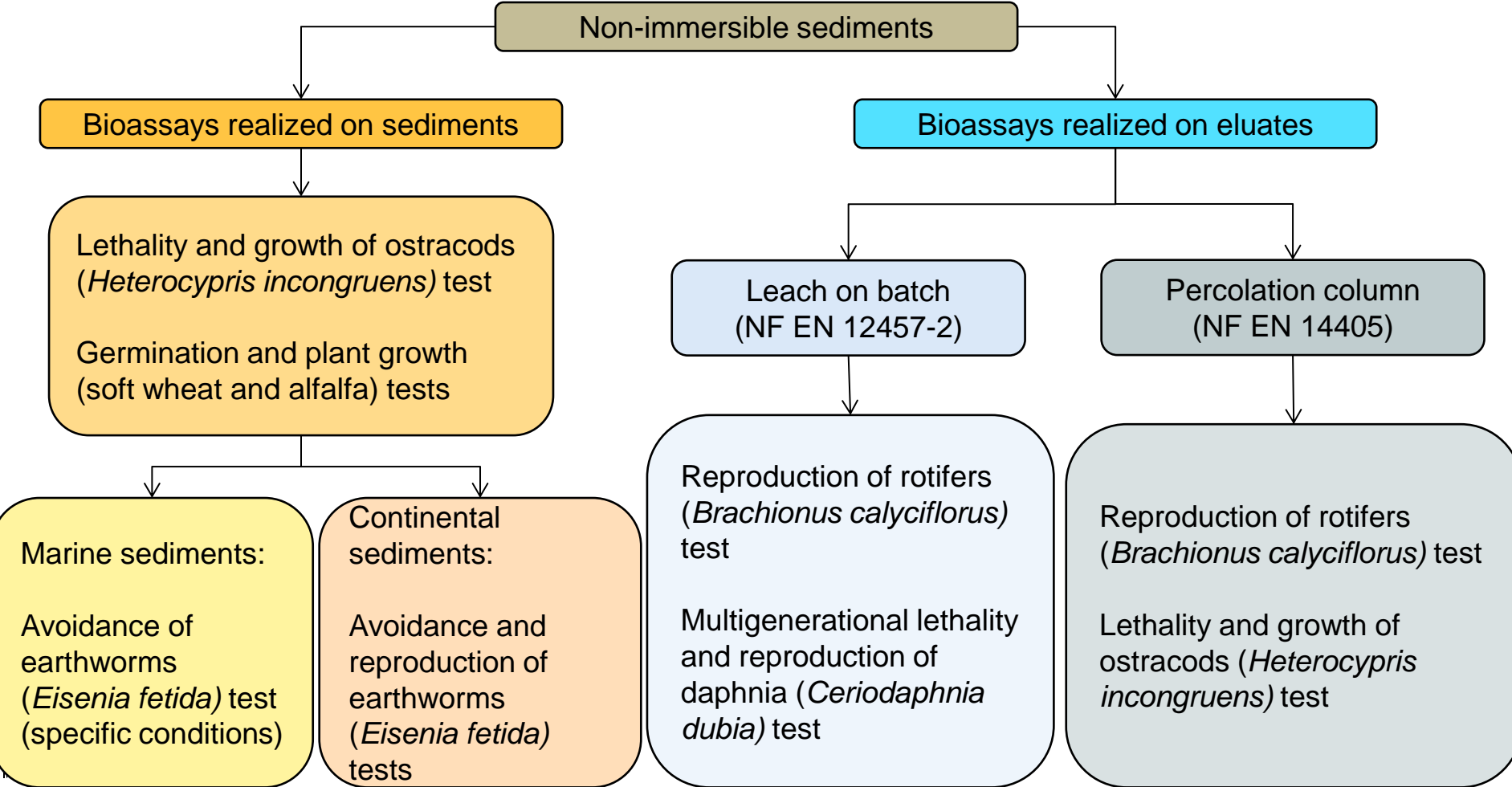
➔ Complementary assay to *Brachionus calyciflorus* test

# 7. CONCLUSIONS



# Conclusions

- Detection of ecotoxic effects on non-hazardous sediments
- No correlation between analysed contaminants content and observed ecotoxic effects
- Confounding factors, effects of synergy/antagonism, variation of bioavailability



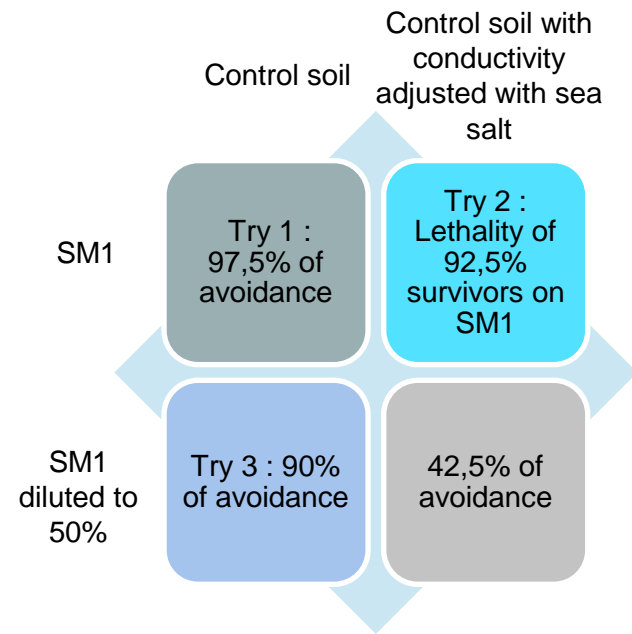


# 8. PERSPECTIVES



# Perspectives

- Test the protocol on other sediments for application as part of the regional SEDIMATERIAUX approach
- Adapt some bioassays (especially earthworms avoidance test)



## Avoidance effects of salts: Possibility of adaptation of bioassay (exemple to SM1)

- Try 1: residual salts potentially responsible for the avoidance
- Try 2: Salt potentially responsible for lethality and significant avoidance contribution
- Try 3: Non-proportional reduced avoidance
- Try 4: Salt without impact on the avoidance, relatively high avoidance despite dilution

- Study about setting thresholds to be adopted

THANK YOU FOR YOUR  
ATTENTION

