Development of analytical methods for the analysis of contaminants in sediment after electro-kinetic treatment

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Introduction

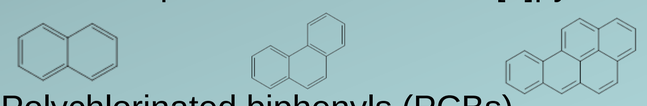
- Many organic pollutants such as **PAHs** and **PCBs** and **metal contaminants** are accumulated in the sediment
- **Consequences:**
  - Damaging aquatic ecosystems
  - Toxic for life including human-beings
- **Aims of researches:**
  - Develop extraction and analytical methods for these pollutants and their metabolites
  - Using electro-kinetic (EK) method for reducing their quantities in sediment

➢ Organic pollutants:

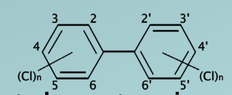
o16 polycyclic aromatic hydrocarbons (PAHs)

2 rings *3 rings* *… 5 rings*

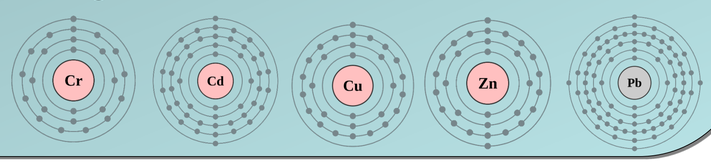
naphthalene phenanthrene benzo[a]pyrene



o 7 Polychlorinated biphenyls (PCBs)



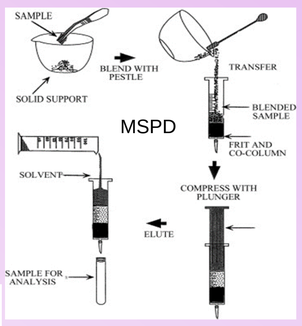
➢ Inorganics: metal contaminants



Materials & Methods

Harbor dredged sediments

Extraction methods

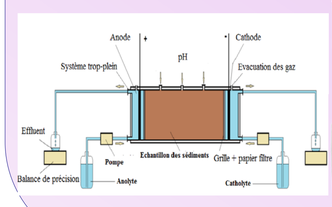


Analytic methods

ICP-AES for metals

GC-MS for PAHs and PCBs

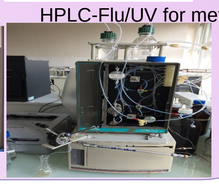
Electro-kinetic treatment



MAE Microwaves

Soxhlet

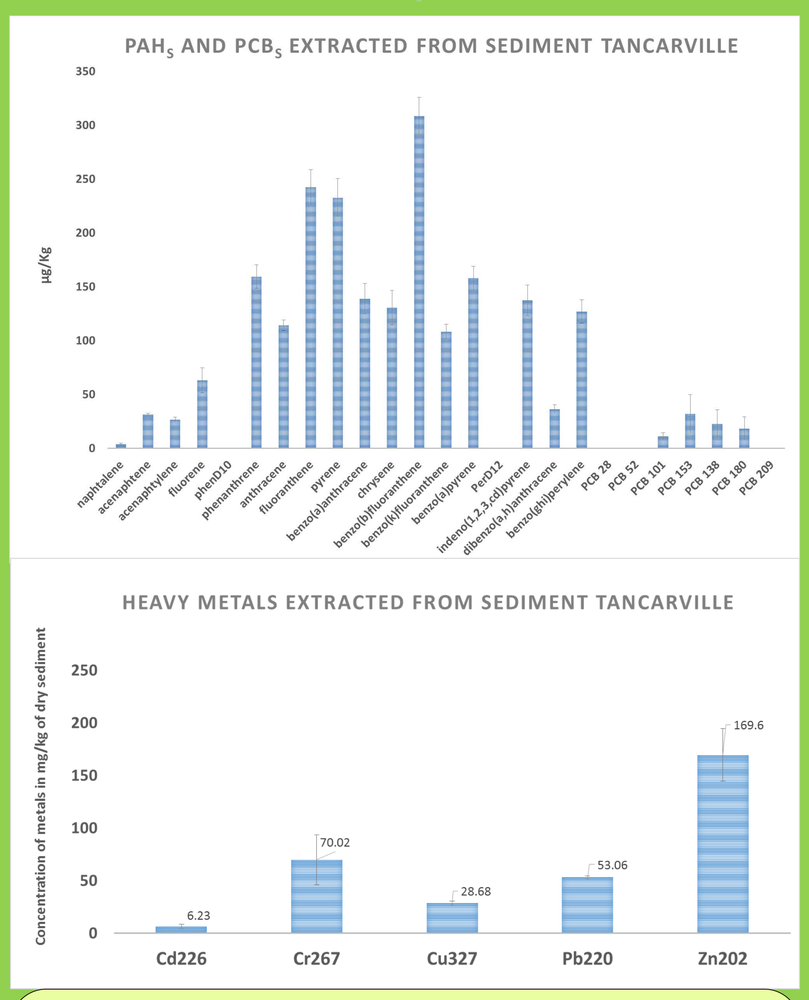
HPLC-Flu/UV for metabolites



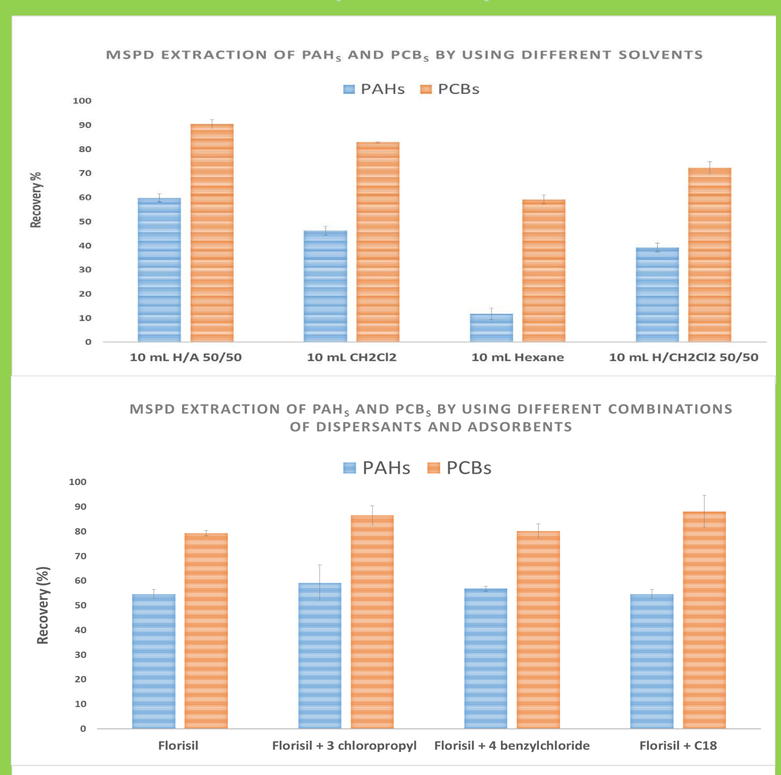
Results and discussions

GC-MS analysis of PAHs and PCBs

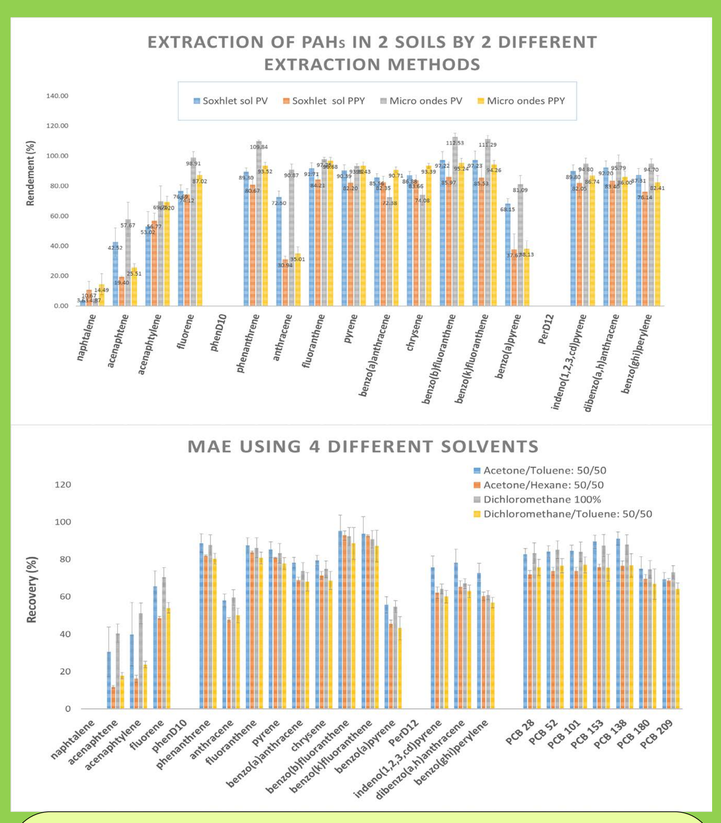
Extraction of real sediment pollutants from Tancarville



MSPD: matrix solid phase dispersive extraction



Extraction of soil organic pollutants

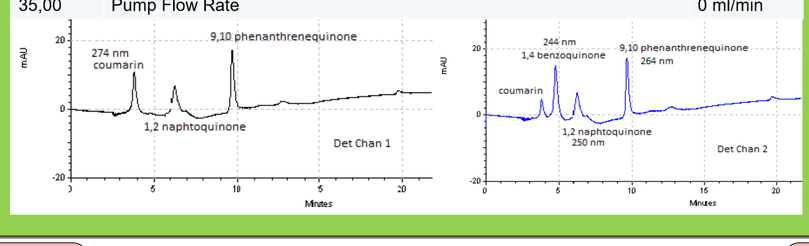


- MAE (microwave) is more efficient and faster than the traditional Soxhlet method.
- Acetone/Toluene or CH_2Cl_2 = good extracting solvents for PAHs+PCBs

- Acenaphtalene, fluorene and anthracene > N1 GEODE (< N2), other PAHs < N1.
- PCBs < N1.
- Metals < N1, but Cd > N2.

HPLC-UV analysis for PAH quinones

Temps (min)		Flow rate = 1 ml/min				
0,00	Det 168	Chan 1 Wavelength				274 nm
0,00	Det 168	Chan 2 Wavelength				244 nm
0,00	Pump			initial	B %	40 %
2,50	Pump			B %	100 %	In 5 min
5,70	Det 168	Chan 1 Wavelength				250 nm
5,70	Det 168	Chan 2 Wavelength				251 nm
8,00	Det 168	Chan 1 Wavelength				264 nm
8,00	Det 168	Chan 2 Wavelength				264 nm
30,00	Det 168	stop data				
35,00	Pump	Flow Rate				0 ml/min



- Hexane/Acetone 50/50: more efficient
- Recovery of PAHs 60% (<71,4% with MAE) and PCBs 90% (>84.5 with MAE).
- Effect of solvent volume and combinations of dispersants = not significant
- MSPD: simpler and faster than MAE

- 4 PAH quinones are separated by C18 RP column.
- Resolution is good enough.
- Problem: UV detection not enough selective (pics may coincide with other substances)

Conclusion

- ✓ GC-MS and ICP-AES are adapted to detect different pollutant families in sediment.
- ✓ Extraction efficiency: MSPD > MAE > Soxhlet.
  ⇨ MSPD still needs optimization (necessary to introduce surrogate standards to correct results related to poor reproducibility of flow-rates).
- ✓ HPLC-UV not enough selective to analyse PAHs metabolites
  ⇨ Coupling UV+fluorescence ? GC-MS after derivatization?

Perspectives

- ❑ Improving EK treatments using original additive mixtures: biosurfactants + citric acid
- ❑ Detect PAH metabolites (quinones + hydroxyl-) and reduced PCBs after EK treatment ⇨ link with sediment toxicity?
- ❑ Microcosms tests to monitor natural dissipation of contaminants in Tancarville sediment (comparison with EK treatment efficiency)