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Herbicides and nonylphenol in sewage sludge: occurrence and ecotoxicological impacts

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Phytopharmacie
Versailles
Xénobiotiques
Toulouse

CECOTOX
Lausanne

Santé Publique et
Environnement
Chatenay-Malabry

Biochimie
Paris

Gestion Biologique
des Sols
Angers

Rationale

Scientific

- **Selection of sludge and contaminants**
  - **type of process and sludge treatment in WWTP**
  - **persistent organic pollutants ?**
  - **analytical methods**
  - **variation of concentration in sludge with time and WWTP**

- **Transfer of contaminants**
  - **amounts and forms**
  - **waters, plants, webfoods**

- **Impact of contaminated sludge**
  - **soil properties and microbial activity**
  - **écotoxicology**
  - **relationships environment / health, EDCs**

Socio-economic

- **Management and sustainability of sludge spreading onto agricultural soils**

Objectives of our research program

- ***Determine the occurrence of selected pesticides in sludge samples***

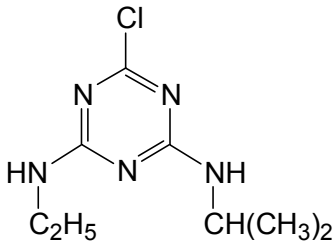
  - development of analytical methods
  - annual monitoring in various sludge samples

- ***Identify possible ecotoxicological effects of the pesticides when applied onto the soils in contaminated sludge***

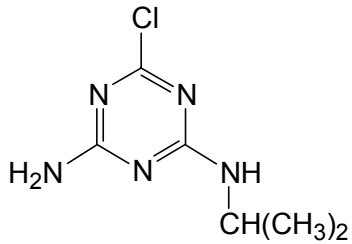
  - fate of the pesticides in the soil
  - ecotoxicity on selected organisms

The chemicals

➢ Atrazine



DEA



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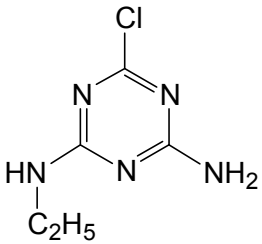
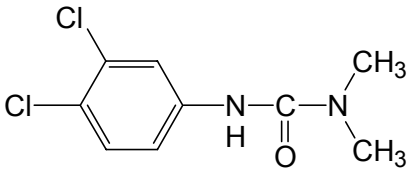
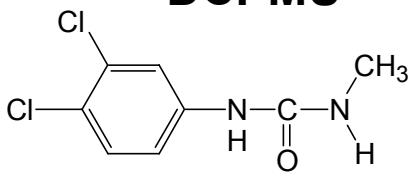
DIA



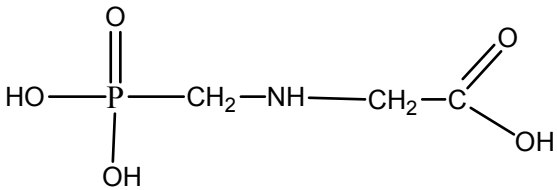
➢ Diuron



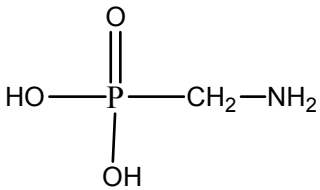
DCPMU



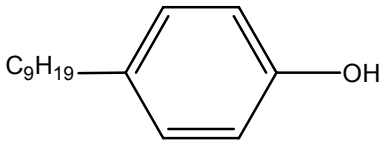
➢ Glyphosate



AMPA



➢ Nonylphenol



1 – Sludge samples contain pesticides

≈ 80 samples from 4 WWTPs during 1 year near Versailles (France)

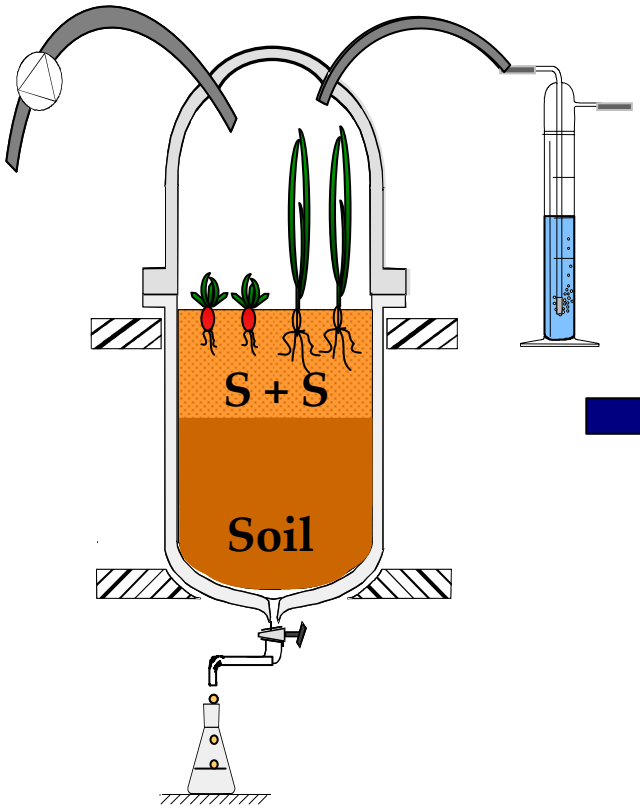
Compound	Dried	Composted	Limed 1	Limed 2
Atrazine + met	/	/	/	/
Diuron ●	11.2	26.5	20.0	46.6
DCPMU	/	/	/	/
Glyphosate ●	1.1	1.0	1.4	0.4
AMPA ●	20.3	12.4	11.5	2.8
Nonylphenol ●	61.7	132.8	90.9	62.5

● ppb (µg/kg dry sludge) / ● ppm (mg/kg)

French regulation: PCB 0.8 and BaP 2 ppm / EC regulation: nonyphenol 50 ppm

► *It is necessary to study their fate in the soil*

Light 16 h, 21°C / darkness 8 h, 18°C



Transformation

- **Mixture of labelled Gly/Diu/NP at 6/17/7 ppm**
- **Soil**
- **Soil + spiked sludge at 30 and 6 T/ha**

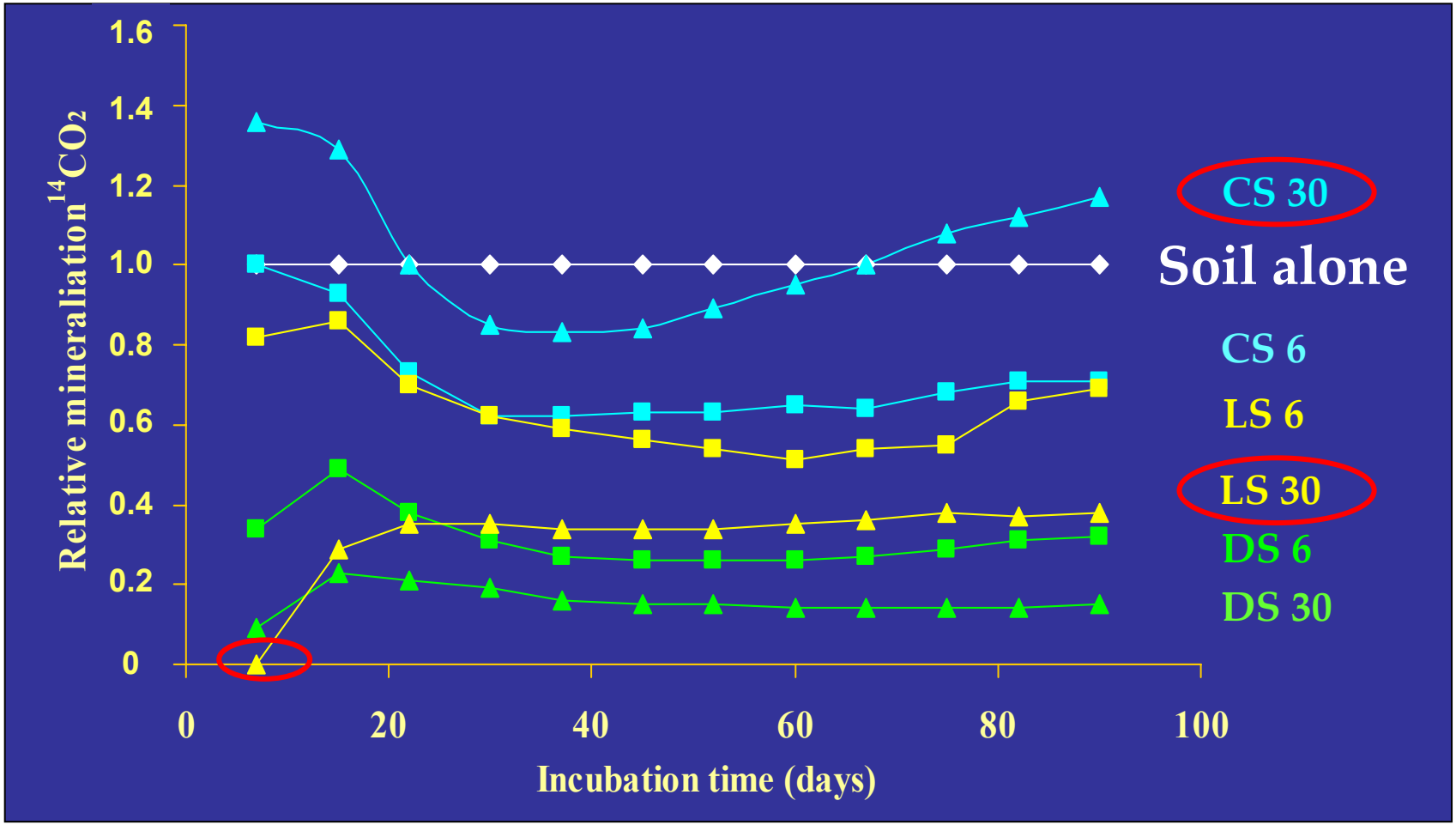
► mineralised / extractable / stabilised metabolic maps

Mobility

- Transfer soil / higher plant

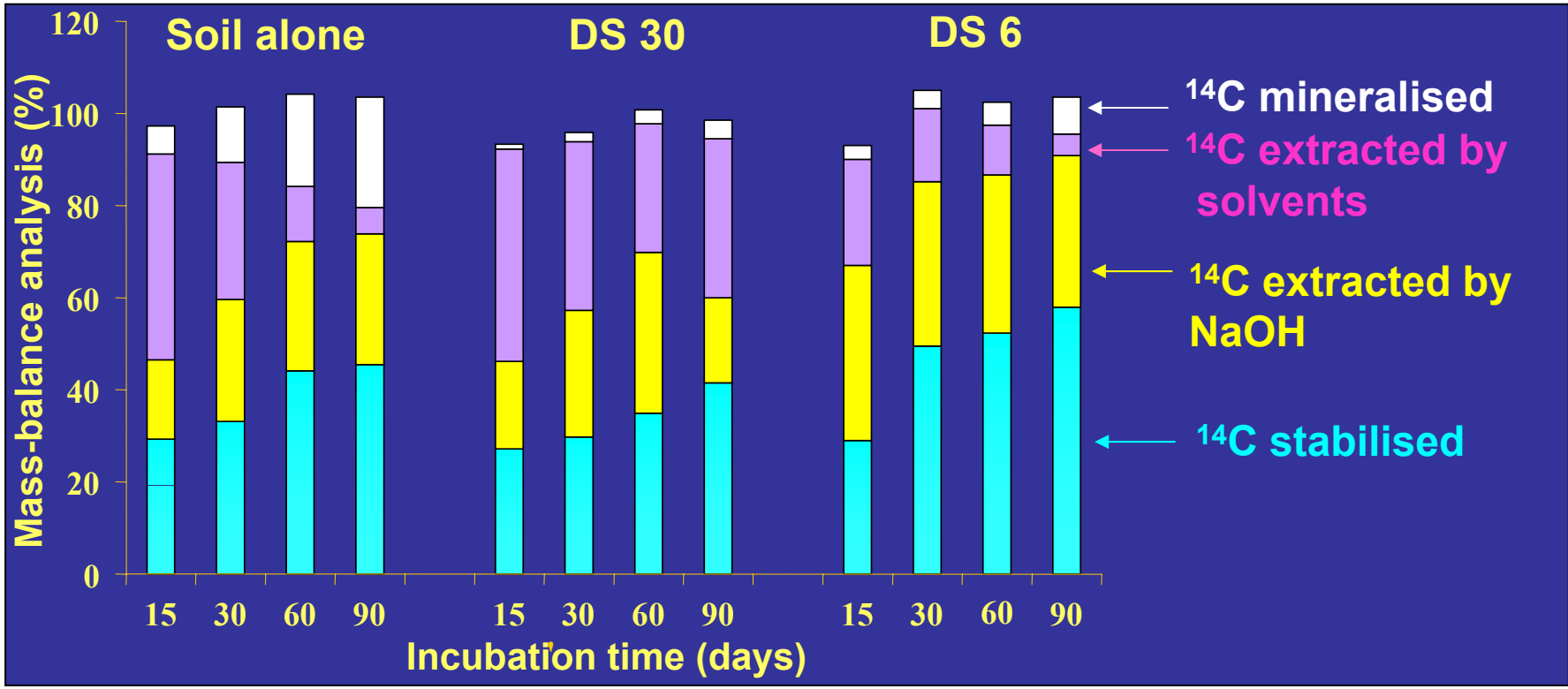
Leachates → **Water contamination**

2 – The mineralisation of the pesticides is decreased



- Composted sludge has contrasted effects at high dose
- Limed sludge induces a 7-day lag phase at high dose

3 – The persistence of the pesticides is increased



With time
Mineralisation increased
Extractability decreased
Bound residue increased

With sludge at 30 T/ha
- **Mineralisation decreased**
- **Extractability maintained**

With sludge at 6 T/ha
- **Intermediate situatio**

4 – Leachates contain traces of pesticides

Less than 1 % of initial radioactivity with a rainfall of 20 mm

Conditions	D + 45 µg/L	D + 60 µg/L	D + 90 µg/L
Soil alone	2.1	1.6	1.2
+ Dried sludge (30 T/ha)	2.5	0.9	1.2
+ Dried sludge (6 T/ha)	0.5	0.3	0.2
Soil alone	14.5	16.2	11.0
+ Composted sludge (30 T/ha)	14.9	12.5	15.2
+ Composted sludge (6 T/ha)	4.8	5.2	4.7
Soil alone	14.5	16.2	11.0
+ Limed sludge (30 T/ha)	29.6	25.2	25.4
+ Limed sludge (6 T/ha)	3.4	3.8	3.7

- **Nonylphenol is the main compound detected using HPLC**

5 – Sludge modify the transfer of pesticides from the soil to higher plants

Less than 0.05 % of the radioactivity during the period of growth

Conditions	Radish (15 days) µg/g	Wheat (45 days) µg/g
Soil alone	/	0.9
+ Dried sludge (30 T/ha)	/	0.07
+ Dried sludge (6 T/ha)	/	/
Soil alone	6.0	0.7
+ Composted sludge (30 T/ha)	1.1	1.2
+ Composted sludge (6 T/ha)	1.8	1.6
Soil alone	6.0	0.7
+ Limed sludge (30 T/ha)	4.3	0.1
+ Limed sludge (6 T/ha)	/	1.1

t is necessary to study their ecotoxicological impac

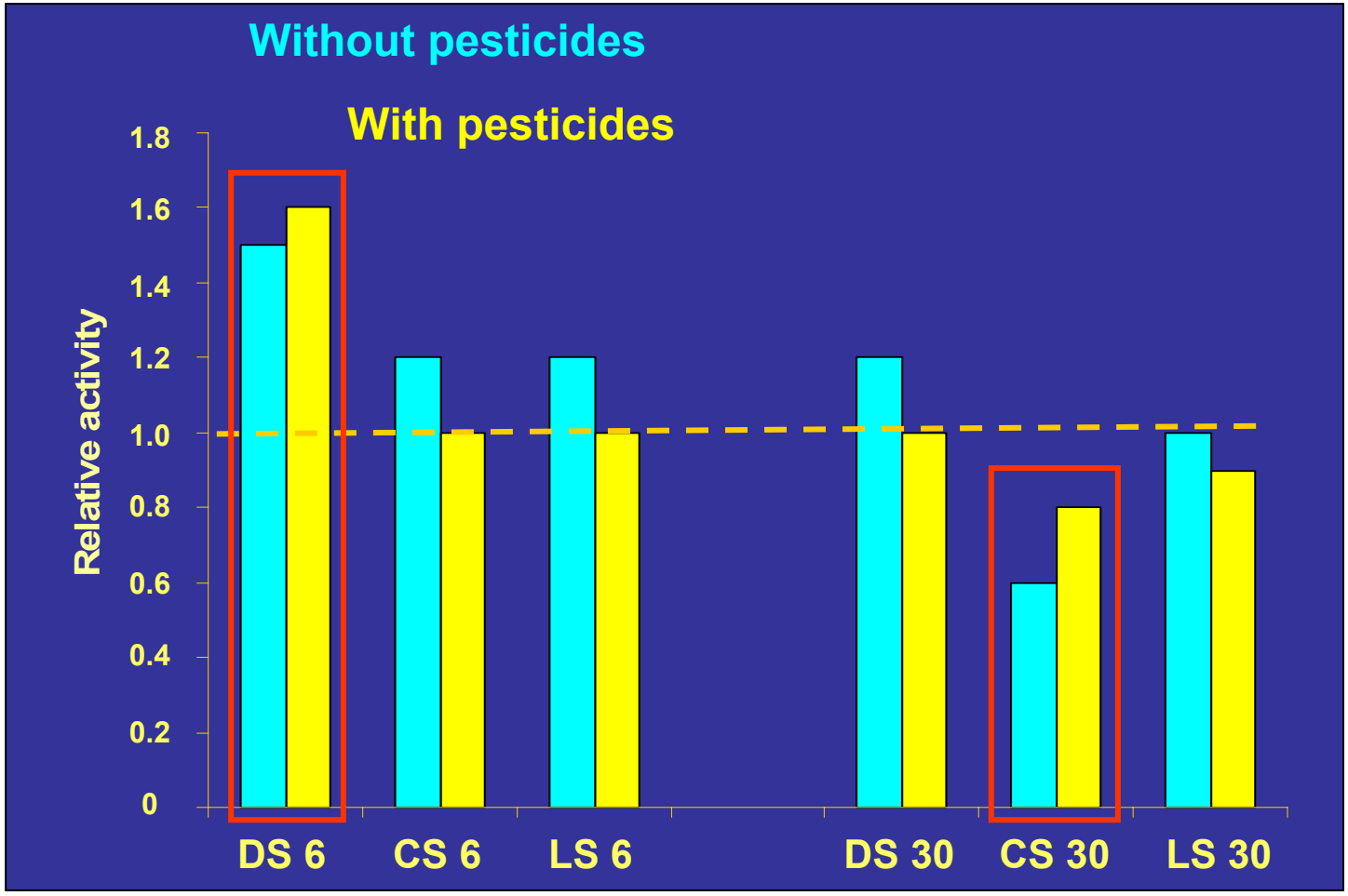
Assays including 3 ecological levels

- **Degraders**
  - microbial functional diversity
    - « litter bag »
  - structural diversity
    - PLFA
- **Producers**
  - germination (%, delay) of plants
  - biomass
- **Consumers**
  - mortality, reproduction of springtails

Estrogenicity assessment
human cell lines MELN

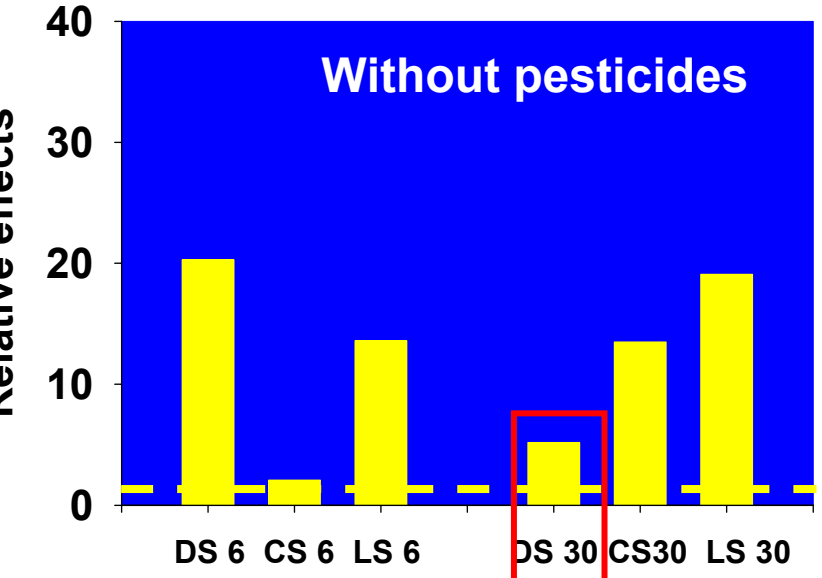
Risk for humans

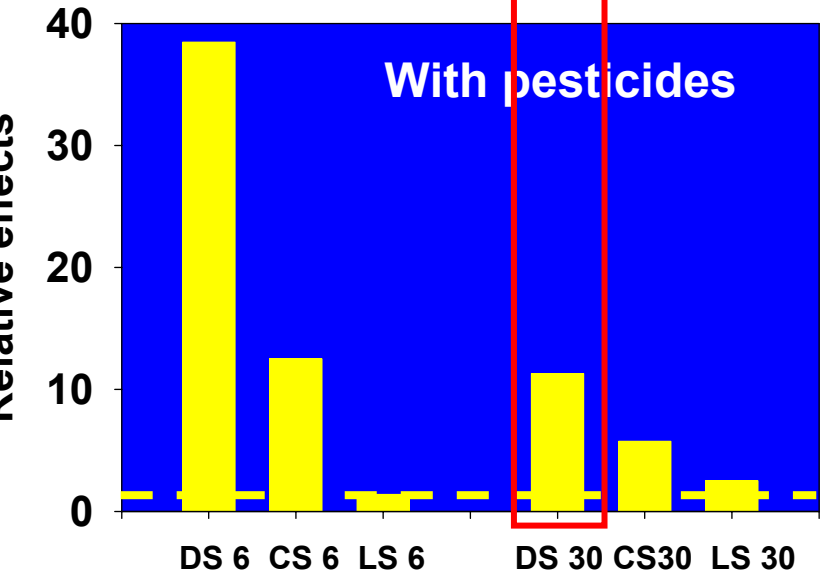
6 – Pesticides have no clear impact on the litter-bag



- Dried sludge increases the soil biological activity at low dose
- Composted sludge decreases the soil activity at high dose

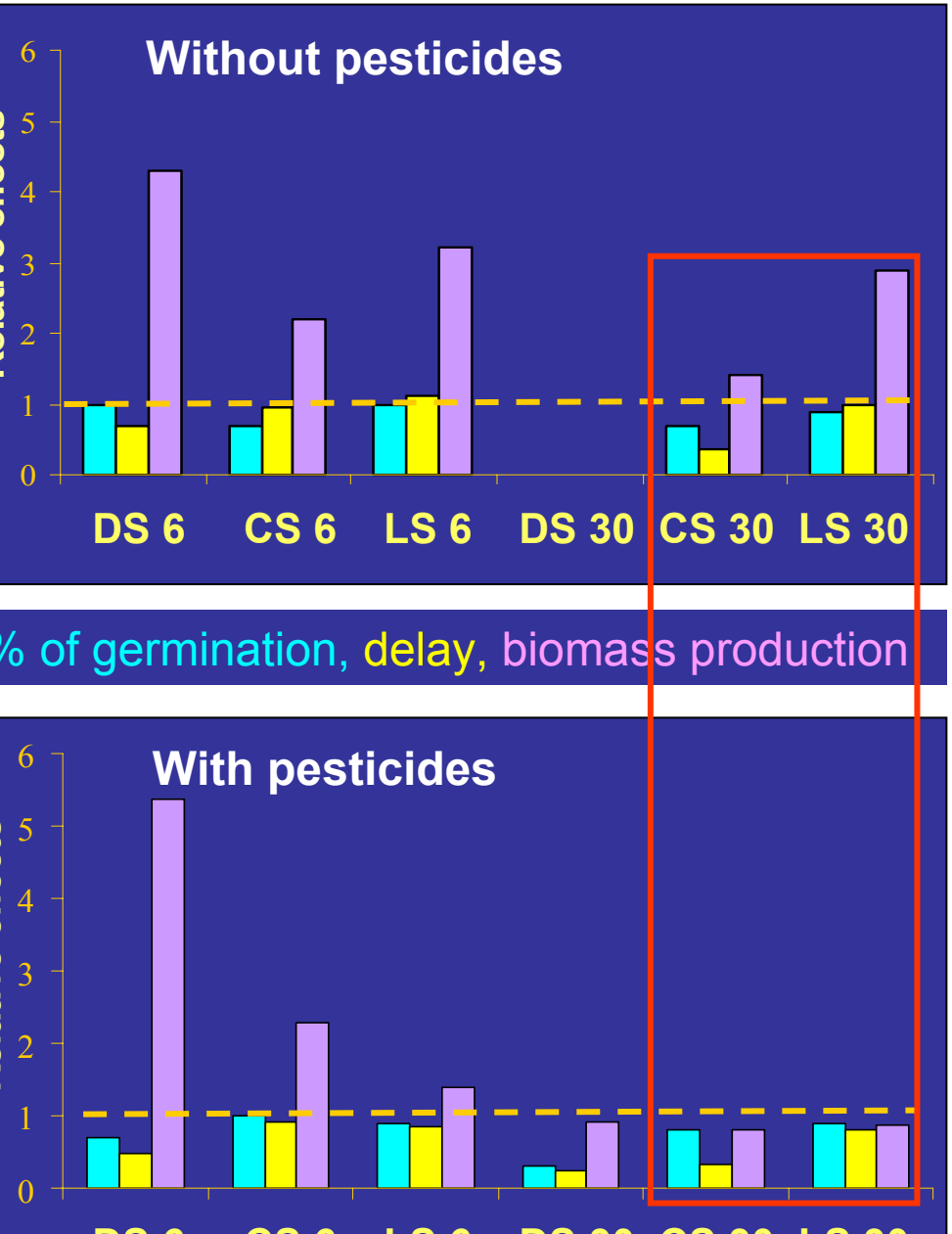
7 – The fungi/bacteria ratio is increased





- Sludge intake generally increases the fungi/bacteria ratio
- The highest effects are noticed with the dried sludge at low dose
- There is no clear effect of pesticides
- Dried sludge at high dose induces the development of fungi at the soil surfac

8 – Sludge are phytotoxic on wheat at high doses

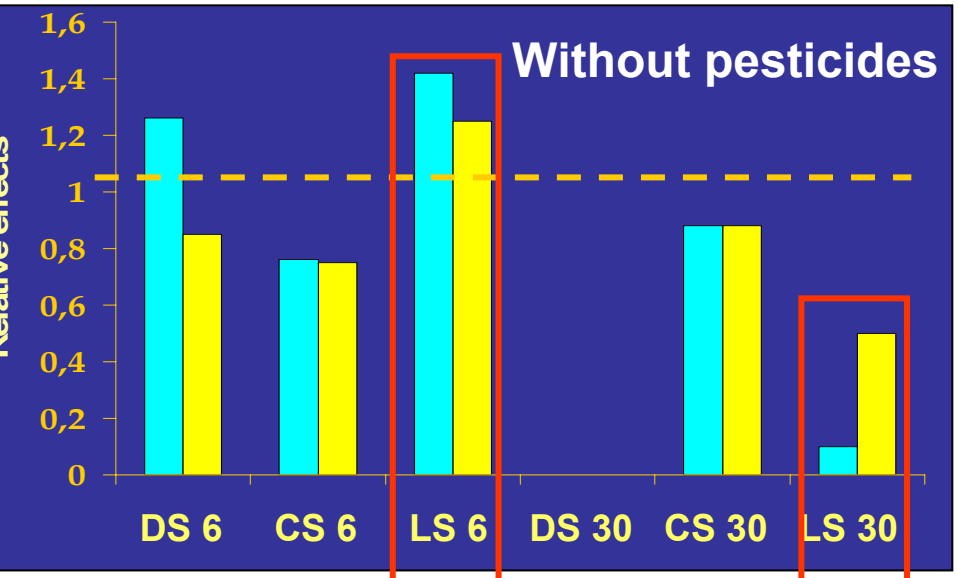


- Sludge are fertilizers at low dose
- Dried sludge inhibits plant development at high dose (fungal proliferation)

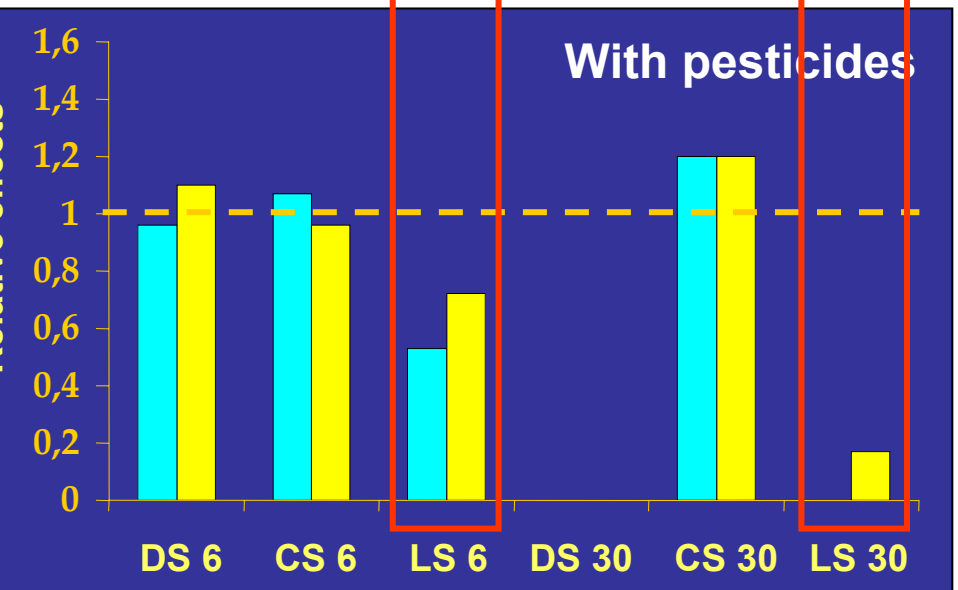
- Spiked sludges are phytotoxic at high dose
- Phytotoxicity could be due to pesticides

9 – Sludge are toxic for springtails

F. candida



Reproduction, survival

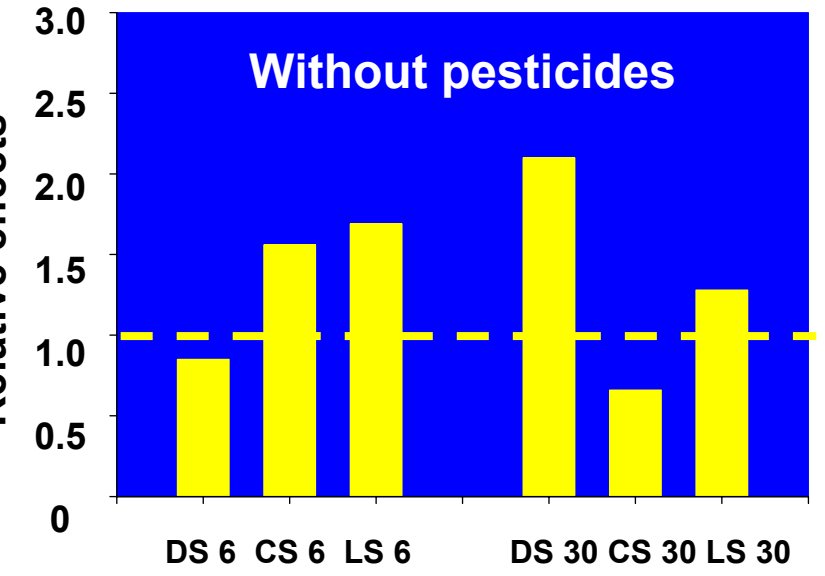


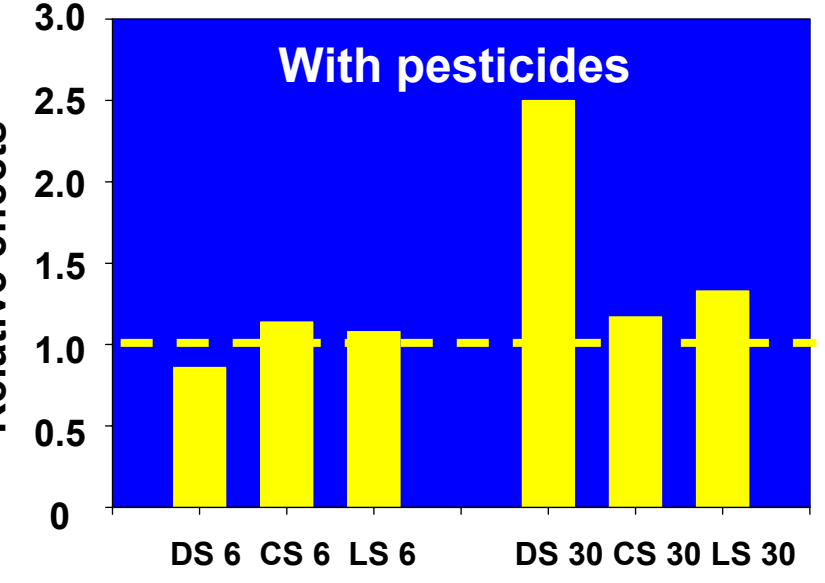
- No effect at low dose
- Dried sludge inhibits sprintails life at high dose (fungal proliferation)
- Limed sludge inhibits sprintails survival at high dose (pH?)

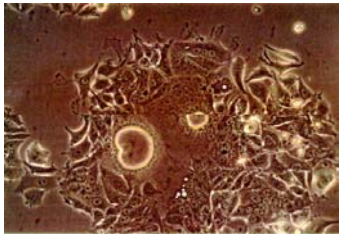
- Limed sludge strongly inhibits springtail survival at high dose

- Toxicity could be due to pesticides

10 – Leachates induce no clear estrogenic effects







MELN cells

- Dried sludge only induced significant estrogenic effects on cells at high dose
- That effect seems strengthened by pesticides (nonylphenol ?)

Conclusions and perspectives

- Urban sewage sludge contain pesticides at relatively high concentrations

- The persistence of pesticides entering the soil through sludge is increased

- Eco-toxic effects have been evidenced at the high dose of sludge (worst case), and not in an agronomic reality

- The effects of sludge are in general higher than these of added pesticides

- The process in WWTPs must be improved to degrade organic pollutants

Acknowledgements

- **The colleagues of the 4 research institutes**
  scientists, research assistants, technicians, administrative staff

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ADEME