



# **Micropollutants in Biosolids**

#### **On-going Research In North America**

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Neptune workshop: Technical Solutions for Nutrient and Micropollutants Removal in WWTPs Université Laval, Québec, March 25-26, 2010 Presentation Outline

- Historical focus of MPs in biosolids in Canada
- Some Canadian results
- Ongoing studies in Canada
- Studies in U.S./North America
- Brief summary

## Historical Context in Canada

- Prior to mid-1970s, focus was on metallic pollutants and potential uptake in crops & livestock after biosolids amendment
- Mid-late 1970s, PCBs investigated for translocation from biosolids-amended soils to plant crops
- 1980s-1990s focused on "priority pollutants", including solvents, pesticides, PAHs, phenolics and chlorinated aromatics

   Publications of Webber and colleagues



 Starting about 2002-03, Dr. H-B (Bill) Lee and colleagues of Environment Canada began reporting concentrations of EDCs & PPCPs in biosolids

- Mostly anaerobically digested solids

 Ca. 2005, Trent University (Dr. C. Metcalfe and colleagues) start reporting concentration data for PPCPs in biosolids



|           | BPA Concentration (ng/g TS) |                    |  |  |
|-----------|-----------------------------|--------------------|--|--|
|           | Raw Sludge                  | Digested Biosolids |  |  |
| # WWTPs   | 11                          | 21                 |  |  |
| # Samples | 12                          | 23                 |  |  |
| Median    | 280 🔶                       | 555                |  |  |
| Range     | 130-39,800                  | 130-11,100         |  |  |

#### (Lee and Peart, 2002)

## **Alkylphenol Ethoxylates**

Neptune

|                          |                            | <u>(Lee and</u>    | <u>i Peart, 2002</u> |  |
|--------------------------|----------------------------|--------------------|----------------------|--|
| Compound                 | Concentration<br>(µg/g TS) |                    | Removal              |  |
|                          | Median<br>raw              | Median<br>digested | (%)                  |  |
| 4-nonylphenol (NP)       | 91.5                       | 413                | -351% *              |  |
| NP mono-EO (NP1EO)       | 61.9                       | 83.7               | -35%                 |  |
| NP di-EO (NP2EO)         | 35.2                       | 24.8               | 29%                  |  |
| NP tri-EO (NP3EO)        | 13.8                       | 6.9                | 50%                  |  |
| Higher NP-EOs NP(4-17)EO | 43.5                       | 11.3               | 74%                  |  |
| 4-tert-octylphenol       | 4.75                       | 10.4               | -119% *              |  |



|           | Triclosan Concentration<br>(ng/g TS) |                    |  |
|-----------|--------------------------------------|--------------------|--|
|           | Raw Sludge                           | Digested Biosolids |  |
| # WWTPs   | 11                                   | 21                 |  |
| # Samples | 12                                   | 23                 |  |
| Median    | 10,600 🗲                             | 14,450             |  |
| Range     | 3,430-17,900                         | 900-28,200         |  |

#### (Lee and Peart, 2002)

### Fragrance Compounds (Lee et al., 2003)

| Compound           | Median Conc'n<br>(ng/g TS) |          | Removal |
|--------------------|----------------------------|----------|---------|
|                    | Raw                        | Digested | (70)    |
| Galaxolide (HHCB)  | 11,850                     | 14,500   | -22.4   |
| Tonalide (AHTN)    | 8010                       | 12300    | -53.7   |
| Celestolide (ADBI) | 175                        | 320      | -82.9   |
| Phantolide (AHDI)  | 110                        | 120      | -9.1    |
| Traesolide (ATII)  | 1345                       | 1870     | -39.0   |
| Musk Xylene (MX)   | 19                         | 3.3      | 82.6 *  |
| Musk Ketone (MK)   | 145                        | 4.5      | 96.9 ★  |

Neptune FP6 Project

# Fragrance Compounds (Yang & Metcalfe, 2005)

| Compound           | Median Conc'n<br>(ng/g TS) |          | Removal<br>(%) |
|--------------------|----------------------------|----------|----------------|
|                    | Raw                        | Digested |                |
| Galaxolide (HHCB)  | 3,300                      | 6,790    | -106           |
| Tonalide (AHTN)    | 720                        | 1,350    | -88            |
| Celestolide (ADBI) | 23.6                       | 51.2     | -117           |
| Phantolide (AHDI)  | 20.1                       | 33.8     | -68            |
| Traesolide (ATII)  | 199                        | 413      | -108           |
| Musk Xylene (MX)   | 76.6                       | 95.1     | -24            |
| Musk Ketone (MK)   | 39.8                       | 53.0     | -33            |



|                                | Concentration (ng/g TS) |                                 |           |   |
|--------------------------------|-------------------------|---------------------------------|-----------|---|
| Carbamazepine or<br>Metabolite | Raw sludge              | Anaerobic<br>digested<br>sludge | Removal % |   |
| Carbamazepine                  | 69.6 ±2.2 <sup>a</sup>  | 258 ±4.7                        | -271%     | 1 |
| 2-hydroxy-<br>carbamazepine    | 1.9 ±1.1                | 3.4 ±0.9                        | -79%      |   |
| 3-hydroxy-<br>carbamazepine    | 1.6 ±0.8                | 4.3 ±0.9                        | -169%     |   |

(n=3)

Miao et al. (2005)

<sup>a</sup> mean ± standard deviation



- Canada
  - CCME
  - Environment Canada
  - Water Environment Association of Ontario
- U.S. North America
  - Water Environment Federation
  - Water Environment Research Foundation
  - U.S. EPA

**Nepture:** CCME – Who They Are & Mandate

- Canadian Council of Ministers of the Environment represents the 14 Environment Ministers of the Canadian federal government, 10 Provinces and 3 Territories
- CCME is responsible for the coordination of national environmental issues, such as:
  - harmonization of municipal effluent compliance,
  - acid rain policy, and
  - Codes of Practice (e.g. petroleum storage tanks).



- Recognized that many academic institutions and agencies are involved in MP research
- Research funding is scarce, so duplication of efforts needs to be avoided
- Hosted a 1-day workshop Dec. 2 in Ottawa
- Sixty participants, attended, representing:
  - science research associations,
  - NGO's,
  - industry/business,
  - academia and
  - government



- Science and research co-ordination activities of a new national body
  - Based on your organization's needs, what would be useful functions for a research coordination body?
    - Information co-ordination and dissemination;
    - Research priority-setting activities;
    - Active identification of collaboration opportunities;
    - Funding research in identified priority areas;
    - Other functions
  - What do you see as the current challenges to undertaking the above activities?



- Governance and Funding
  - What would be the most workable governance structure for a research co-ordinating body?
  - Should the identified functions be undertaken by one body, or could they be divided among several organizations? New organization or existing?
  - Funding Options (who provides the funding?)

General consensus was to keep this process moving forward and not let it remain an academic exercise.



- No focused study has been completed yet on an inventory of MPs in Canadian biosolids
- Complete a targeted sampling program which will provide a basis for CCME to evaluate and manage the risks associated with MPs in biosolids with respect to:
  - managed land application,
  - land reclamation,
  - production of commercial soil amendments and
  - energy production



CCME Efforts: Concentrations of MPs and Effects of Biosolids Treatment

- Year 1: literature review of micropollutants in raw sludges and treated biosolids, and removal through treatment processes (complete)
  - 121 pages
  - Posted at:

http://www.ccme.ca/assets/pdf/pn\_1440\_

contam\_invt\_rvw.pdf



CCME Efforts: Concentrations of MPs and Effects of Biosolids Treatment

- Year 2: field study of 11 sites across Canada using different techniques for producing solids for land amendment
  - Anaerobic digestion
  - Autothermal aerobic digestion
  - Composting
  - Alkaline stabilization
  - Heat drying
  - Dewatering by filter presses and by geotextile bags
  - 57 Pharmas and PCPs, fragrances, alkylphenols, metals
  - Draft Report review complete and revisions in progress
  - Report posted on CCME website in April or May (?)

Environment Canada Efforts

- Authorized by Chemicals Management Plan under Canadian Environmental Protection Act (CEPA)
- Monitoring of wastewater samples and solids for MPs
  - Alkylphenols, parabens, pharmaceuticals,
  - Perfluorinated organics, brominated flame retardants (PBDES + 4 others)
  - Volatile methyl siloxanes (D4, D5, D6)
  - 18 metals/metalloids

Environment Canada CMP Study

- 20 WWTPs across Canada
- Primary, secondary, advanced, and lagoon treatment
- About 10% of the Canadian population
- Two campaigns
  - Cold temperatures (January to May) ongoing
  - Warm temperatures (June to November) over
- Contact Shirley Anne Smyth
  - (905) 336-4509; ShirleyAnne.Smyth@ec.gc.ca



- Water Environment Assoc'n of Ontario
- Literature review of micropollutants (including pathogens) in biosolids and <u>fate</u> and effects in terrestrial environment
- Update of a review completed in 2001
- 200+ page report completed Dec. 2009
- Peer-reviewed by North American experts to confirm accuracy and completeness
- Will be posted on WEAO website (<u>www.weao.org</u>) after public consultation

# Water Environment Federation

- Microconstituent Community of Practice (COP)
- Chair: Joe Cleary, HydroQual, Inc.
- Issue Leaders
  - Occurrence: Dr. Tom Granato, Metro Chicago
  - Analytical: Akin Babatola, Santa Cruz, CA
  - Treatment: Rich Edwards, Pfizer, Inc.
  - Fate and Transport: Dr. Mary Buzby, Merck & Co.
  - Communications: Linda MacPherson, CH2M-Hill
     Effects: Diana Eignor, U.S. EPA
- WEF Liaison: Bonnie Bailey

WEF Microconstituent COP Activities

- Development of webcasts
- Development of Technical Practice Updates (TPUs)
- Sessions in WEFTEC10 Technical Program
- Development of Bi-annual Joint Micro-Con and Industrial Water Quality Conference
- Development of a collaborative outreach program with other organizations (e.g. SETAC, AWWA) to work together on topics of common interest.



- Oct 2009, Joint Webcast with the Society of Environmental Toxicology and Chemistry (SETAC): <u>Microconstituents in the Aquatic</u> <u>Environment: Impacts for Wastewater</u> <u>Utilities</u>
- Dec 2009, Microconstituents in <u>Biosolids</u>
- 2010(?), Microconstituents in <u>Water Reuse</u>



- Available at www.wef.org
  - Analytical Methods and Monitoring Technologies for MCs
  - Current Regulatory Framework for MCs in Water
  - Effects of Wastewater Treatment on MCs
  - MCs in Biosolids
  - Nanoparticles
  - Source Control of MCs
  - Sources of MCs and EDCs
  - MCs in the Water Environment A Resource Paper for Public and Press



- Contaminants reviewed were pharmaceuticals, brominated flame retardants, plastics & plasticizers, musks & fragrances
- Regulatory status in U.S. and EU
- Methodologies for assessing MCs in biosolids
- Summarized current research status and future directions



- Fate of Estrogenic Compounds during Municipal Sludge Stabilization & Dewatering – WERF Project #04-HHE-6
- WERF Project Manager Alan Hais
   (703) 684-2470; ahais@werf.org
- Work led by AECOM and US Geological Survey
  - Study is in progress



- Establish identity, characteristics, concentrations, temporal/seasonal variations and estrogenic potency in biosolids during different treatment processes
- Examine effect of sludge treatment and dewatering processes on estrogenic compounds and estrogenic activity
- Targeted full- and pilot/bench-scale studies
- Phase II will examine other trace contaminants



- U.S Targeted National Sewage Sludge Survey (TNSSS)
- Released in 2009
  - 74 POTWs, >1 MGD, secondary treatment or better in contiguous U.S.
  - Tested for 25 metals, 11 PBDEs, 97 pharmaceuticals, steroids and hormones (and others) in 84 samples
- Project contact Rick Stevens
  - (202) 566-1135
  - stevens.rick@epa.gov



- Of 72 pharmaceuticals, 3 (ciprofloxacin, diphenhydramine and triclocarban) were found in all 84 samples; 9 were found in at least 80 of the samples
- 10 of 11 PBDEs were found in all samples
- 17-α-ethinyl-estradiol not found in any samples
- Reference:

www.epa.gov/waterscience/biosolids/tnsss-overview.html



- Concentrations and potential effects of MPs in biosolids are a concern in North America because land application is an important management alternative
- Substantial research effort is going on in Canada and the U.S.
- Occurrence of MPs in biosolids is becoming better characterized
  - Fate and effects of MPs in terrestrial environment are generally not well understood



- Thank you for your attention!
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