Biosolids Trace Chemicals Research Happening in This Region
Gordon Price discusses research he started in 2008
NEBRAMail, December 29, 2014

Introduction

In NEBRA's region, there is only one researcher looking closely at key questions about microconstituents in biosolids: Gordon Price at Dalhousie University's Truro, NS agricultural campus. Microconstituents – trace chemicals such as pharmaceuticals and personal care products (PPCPs) and other emerging substances of concern (ESOCs) – and their presence and impacts in the environment have held public attention for more than a decade. While research to date has found that these trace chemicals are unlikely to present significant risk to public health and the environment via their presence in biosolids, more research is needed. The Water Environment Research Foundation (WERF) in the U. S. has been conducting a review of the state of the science and developing research plans. Meanwhile, a research agenda in Canada, funded in part by the Canadian Water Network (CWN), has been advancing several projects assessing the presence, fate, and potential impacts of microconstituents in biosolids.

The field study sites in Truro overseen by Dr. Price have received biosolids since 2008. For 2013 and 2014, the CWN has provided funding for the Dalhousie-led research team to continue its biosolids research with a focus on "impacts of alkaline stabilized biosolids application on fate and transport of emerging substances of concern in agricultural soils, plant biomass, and drainage water" (details).

An initial part of Dr. Price's research was looking at the nitrogen and pH values and dynamics of Halifax lime-treated biosolids applied to the agricultural test plots he established in Truro, NS. (See presentation to the SSSA conference, 2011.)

As an example of initial findings from Dr. Price's microconstituents work, in fall 2014, the *Canadian Journal of Soil Science* published a paper focused on triclosan (TCS) and its apparent aerobic microbial degradation in soil (see graphs). "A rapid decrease in triclosan concentration was observed during the first few weeks of the incubation study, with concentrations decreasing from 92 +/- 26 to 20 +/- 2 ng g-1 (average 78% decrease) after four months. The field results indicate that triclosan in Fall-applied may persist over winter. However, a rapid decrease in triclosan concentration during the spring and summer months led to levels lower than predicted following the Spring application, and levels below our reporting limit (up to 85% decrease) by the end of the study" (see graphs, below).

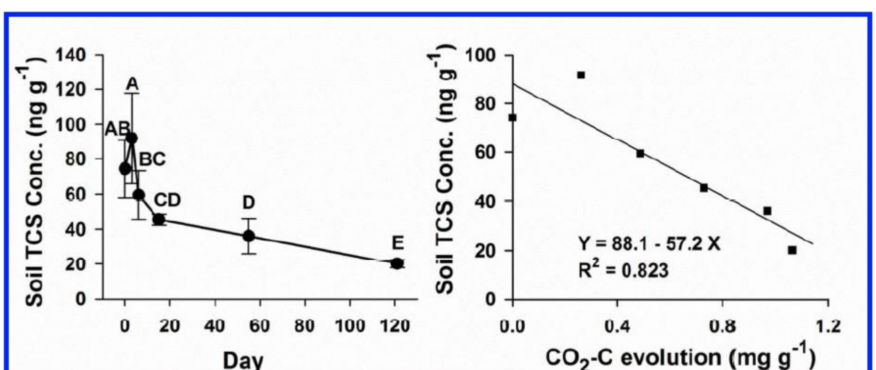

Fig. 3. Left: Triclosan concentration over time from incubation soil amended with the equivalent of 42 t ATB ha^{-1}. Error bars represent 95% Confidence Interval, and days with the same letter are not significantly different (α=0.05). Right: Relationship between triclosan concentration and the cumulative evolved CO_2-C from biosolid-amended soil. This supports the idea of aerobic microbial degradation as a likely removal pathway for triclosan.

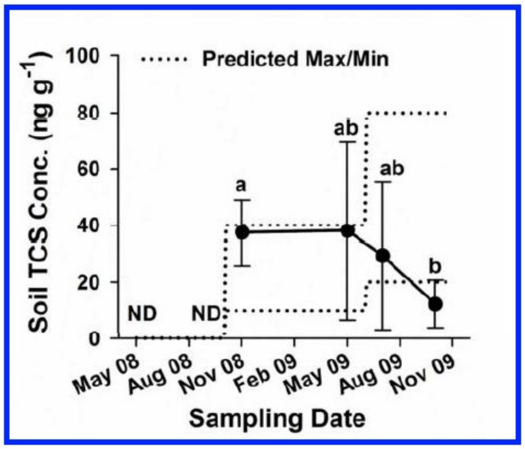

Fig. 5. Measured and predicted soil triclosan concentrations in the top 15 cm of field plots receiving annual biosolid applications. Application rate was 21 t ha^{-1} in Oct. 2008 and again in June 2009. No triclosan was detected in the May 08 and Sept. 08 soil samplings. Predicted concentrations are based on the range of triclosan concentrations measured in ATB (Figure 2). Error bars show 95% C.I. (d.f.=8). ND means not detected (concentrations below linear calibration range).

The Truro study site

Dr. Price set up the Truro land application research site in 2008, and, during that growing season, background data were collected. In the fall of that year, lime-treated biosolids from Halifax were applied at one-half the recommended rate. Another half-rate application was applied in the spring of 2009, just before corn was planted.

Beginning in 2010, a portion of the treated area received no further biosolids applications. (It did receive standard starter fertilizer applied with the corn seed each year.) These plots are measuring the impacts of just what had been applied the one time. "If we detect something, how far does it ripple out?" said Dr. Price.

The remainder of this study area has received annual spring biosolids applications at the standard rate of 7 tonnes/ha. In 2010, 2011, and 2012, corn was grown on this study area, using standard chemical starter fertilizer as well as biosolids. In 2013 and 2014, the crop was barley – and no starter fertilizer was applied. The plan for 2015 is to plant corn again.

As they were conducting the initial pH and nutrient analyses, Dr. Price's team developed their laboratory and partnerships with other labs to be able to measure microconstituents - trace chemicals - in biosolids, soils, and plants. However, they continue to monitor nutrients, metals, pH, and crop quality and yield.

"Our first few years of studying trace chemicals was just trying to do a broad scope survey of the various compounds that might be here," Dr. Price explained during a site visit in September. "We monitor and soil sample this site every 3 to 4 weeks from April through October or November. We've been doing that every year." Samples are analyzed and/or frozen and stored for future testing. A critical part of the research process has been developing and improving analytical methods, which are challenging because of the complexity of the biosolids and soil matrix and the low concentrations of chemicals being investigated.

Nearby are additional study sites. One field is segmented into plots, each of which has an individual tile drainage system that captures soil water and transmits it to a central building. "This allows us to apply a treatment to a plot and capture the tile drainage water from just that plot," Dr. Price explained. "We have nine cells and 3 replications: a control, a moderate application rate of biosolids, and a high rate of application. We're monitoring the water for several years, as well as taking soil and plant samples (rye was grown in 2013 and corn in 2014). We used to have to monitor rain events and come in and sample water manually. Now the system is automated to capture samples routinely."

Rob Jamieson, a colleague at Dalhousie, is modeling the biosolids - soil – plant system and trace contaminant fluxes in the soil/soil water system. Dr. Price explains: "Rob Jamieson's group has calibrated the model to the hydraulics of these test cells, so that measured concentrations of microconstituents found in these experiments can be related to expected real-world field concentrations. There is some variability inherent – soil compaction, etc. So each cell can be plugged into the model individually, so that it is not necessarily compared to other cells. The goal is to be able to project forward from changes in application rates, etc. to predict impacts on soil water and other parts of the system. We can then change soil characteristics and see the results and plug them into the model."

Another field contains monitoring wells that extend as far as the bedrock sandstone. These fields have not had biosolids applied in recent years. "So we will capture samples before application and then do a fall application of NViro soil and drill a multi-level groundwater sampling well and sample all wells and tile drainage through next spring and maybe next fall. This means we'll be capturing short-term movement of groundwater."

Research Goals

Dr. Price's goals for this research are extensive. As we tour the field sites, he touches on numerous interesting questions that he hopes to answer:

- "We're trying to assess the whole broader system – the agronomic and chemical dynamics in the site."
- A major focus is the trace chemicals: "We're trying to capture all the environmental pools: soil, water, and plant biomass (but not gases)."
- "Another interest is in mineralization of nitrogen and phosphorus."
- "I also wanted to see if we stress the system when we apply biosolids. Are we pushing greater mineralization because we are adding this organic matter source? Which means you might be stimulating broader microbial activity. Does that have an impact on these organic chemicals, which are not static? They're changing through the chemical environment and the microbial environment and/or the interaction of the two."
- "At another site, 80 km away, that's much larger in scale, we're looking at tillage vs. no till. That's a drained agricultural site, on a slope, and we can capture soil water there. Each plot is a hectare. Tillage was the factor we looked at. We used a standard agronomic rate of biosolids and watched for migration of chemicals into drainage water – pharmaceuticals particularly."

The Microconstituents Research

With regards to microconstituents, Dr. Price is focused on chemicals chosen based on quantities of sales: the top 20 chemicals based on production and use in pharmaceuticals, personal care products, etc. They include phthalates, because they are ubiquitous in plastics.

One student of Dr. Price is currently focused on phthalates. He explained: "There is a lot more work on TCC and TCS already; not as much on phthalates. We couldn't replicate other researchers' published methods of detections from standard control media like sand. So my student modified and developed a method. He is now working on the extraction part, doing validation series. A manuscript from this work is in progress. Testing the impacts of raw vs. treated solids will tell whether the treatment process somehow stabilizes or sorbs the microconstituents, making them leach less or not. The comparison also helps determine if the rate of leaching is linearly related to the concentration of the contaminant."

Another student is looking at sorption and desorption of three pharmaceuticals in mixture (most prior work has been done on individual chemicals). "We're just looking at biosolids alone initially, because these lime-treated biosolids have a big impact on pH, coming out of the plant at 11; when it gets to the field it is about 9.5; then it goes into the acidic environment of the soil. So we want to see if there is an impact of those changes in pH on the sorption and desorption. We're then looking at the capacity of the soil to sorb the organic chemicals over time, as a mixture. Is there competition between the chemicals for sorption sites? And what is the impact on desorption? The chemicals were chosen because they are common but have not been covered in the literature as much."

Bioassays
And his team has looked at biological health indicators. "We have done earthworm sampling at intensively disturbed plots over one year, spring and fall. We're hoping to do a fall sampling this year too. We found the worms tended to move to the places where biosolids were applied."

The earthworm work has included some metabolomics – looking a changes in metabolites as a way to understand stresses. "Think of this as a blood test," explains Dr. Price. "Can we take a test on earthworms and use it as an indicator of soil environmental health? For example, if a worm presents a certain metabolite profile, it may indicate some kind of stress - likely a chemical stress – but we likely won't know the specific compound of concern. But the metabolite signature will be a physiological response to a stressor. If this bioassay does find stress, we would then test chemicals one by one or in mixtures, to record the metabolic signatures of responses to these stresses (if any). This might then allow measures of metabolic signatures associated with particular chemicals in the field where biosolids are applied."

"And a colleague at Agriculture and Agri-Food Canada has sampled for nematodes three times a year for two years. They're looking at population dynamics of different types of nematodes that have different roles in the soil ecosystem."

Expanding the Research Team
In 2013, the Canadian Water Network funding helped Dr. Price expand the team to include more researchers:
- At Acadia University in Wolfville, NS, Anthony Tong is helping with analysis of microconstituents
- John Marimboh, also at Acadia, is regularly scanning for 28 trace metals in the archived monthly soil samples that extend back to 2008.
- Shiv Prasher at McGill University in Montreal has lysimeters collecting subsoil water in field soil columns in Quebec to which either untreated (raw) sludge or biosolids are applied. His team's interest is in testing for chemicals at different depths in the columns and, in particular, estrogenic compounds.
- Mehdi Sharifi at Trent University in Ontario is helping study the impacts of raw sludge or biosolids in three different reference soils, including the soil from the Dalhousie study plots. Dr. Price explains that "any degradation or kinetics studies in this project will use these three reference soils. We have 100 kg of each soil stored for this purpose." Dr. Sharifi's involvement includes one greenhouse study with corn looking at nutrient cycling and organic chemical movement (a student of Dr. Price is doing the lab analyses).

Looking Forward
Dr. Price fully expects this long-term project to be able to produce understanding and tools useful to the biosolids management profession. Already he has improved understanding of nitrogen dynamics from land application of the Halifax, lime-treated biosolids in Nova Scotia.

The models being developed are an expected output of the project: "The models we're developing could be applied to other soils – New England soils, for example, " says Dr. Price. "They can help ensure best management: if a projected, modeled scenario indicates a negative impact, then you know to change the management practice to avoid those impacts. It's about sustainability: How do we moderate the rate of application to work for each soil so that the soil can deal with it. We might find that different solids treatments may be more or less appropriate for a particular soil system."

With systems and research teams developed at considerable cost over many years, Dr. Price hopes to see this research work continue for several more. So does NEBRA. The field sites, laboratory capabilities, and researcher understanding of biosolids are investments that provide critical support for biosolids management in this region.