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LAND APPLICATION OF MUNICIPAL BIOSOLIDS: MANAGING THE FATE AND TRANSPORT OF CONTAMINANTS OF EMERGING CONCERN

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ABSTRACT

Municipal biosolids provide organic matter to soil, and nutrients essential for crop growth. Some contaminants of standing and emerging concern such as pharmaceutical and personal care products (PPCPs), hormones, brominated flame retardants, and highly persistent perfluoroalkyl acids are not fully removed in the waste treatment process; and thus, they are often found in resultant biosolids applied to land. This paper provides an overview of selected research led by Agriculture and Agri-Food Canada on monitoring and predicting the fate and transport of these noted contaminants in field soil-hydrological environments following land applications of dewatered and liquid municipal biosolids. Different land application practices were examined in the context of their potential to reduce environmental exposure. Field studies of liquid/slurry municipal biosolids demonstrated that in macroporous soils contaminants of all types can rapidly reach shallow groundwater and tile drainage systems. Nevertheless, loads of contaminants in subsurface (tile) drainage can be significantly reduced if an aeration-based pre-tillage is employed. For dewatered municipal biosolids, directly injecting biosolids into subsoil had an indifferent effect upon water contamination, when compared with traditional surface application methods. For very high single applications of dewatered municipal biosolids to land, compounds such as the antifungal miconazole, the PBDE congener BDE 209, and perfluorooctanoic acids, for example, can persist in biosolid aggregates. Yet, for modestly macroporous soils, most of these compounds will not enter critical subsurface water receptors.

1. INTRODUCTION

The municipal sewage treatment process does not fully eliminate contaminants of emerging concern (CEC) (Clarke and Smith, 2011; Verlicchi and Zambello, 2015) such as pharmaceutical and personal care products (PPCPs), hormones, polybrominated diphenyl ethers, and perfuoroalkyl acids in biosolid residuals and wastewaters (Hernando et al., 2006; McLellan and Halden, 2010; Nieto et al., 2010); Venkatesan and Halden, 2014; Alder and van der Voet, 2015. In many countries and more localized jurisdictions, land application of municipal biosolids (treated sewage sludge) is routinely conducted (Joshua et al., 1998; USEPA, 1999; European Commission, 2001; Mantovi et al., 2005; Schut, 2005; Kelessidis and Stasinakis, 2012) as a means to provide nutrients and organic matter for crop growth and reduce disposal burdens. In Canada, the amounts of municipal biosolid mass that can be applied to land is often governed by heavy metal limits; limit criteria that can vary among provincial jurisdictions (CCME, 2010). CECs resulting from and application of municipal biosolids to agricultural field soils have been detected in groundwater and subsurface drainage networks (Barnes et al., 2008; Lapen et al., 2008, Edwards et al., 2009) and in surface runoff (Pedersen et al., 2005; Topp et al., 2008; Sabourin et al, 2009); also many CECs have been shown to persist in soil (Kinney et al., 2006; Gottschall et al., 2012; 2013; 2017). There is also evidence of CECs being absorbed and translocated in plants (Boxall et al., 2006; Stahl et al., 2009; Wu et al., 2010; Picó et al., 2017).

This paper summarizes a suite of studies conducted in Ontario Canada by Agriculture and Agri-Food Canada, that examine CEC persistence in soil and hydrological exposure pathways associated with land application of municipal biosolids; with consideration of: biosolid type (LMB, liquid municipal biosolids; DMB, dewatered municipal biosolids), land application rates, and methods of land application.

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2. METHODS AND MATERIALS

2.1 Study sites

Table 1 documents for selected studies, the field sites and generalized experimental details, including type/amount of municipal biosolids applied, land application methods, environmental endpoints monitored, and study duration. The Winchester study site consisted of six treatment plots and two control plots, each 100 m in length x 15 m in width. All plots were tile drained (artificial subsurface drainage). The Ottawa study site was located on an experimental agricultural field. The field consisted of four independently tile drained plots, approximately 3 ha each, of which only two plots were used for the experiment (one treatment and one control/reference). The London, Ontario study consisted of 25-30 plots of 2 m^2. For all study sites, no previous biosolids applications had taken place before the experiments summarized here. Table 2 lists the various types of CECs monitored for each study with selected compounds identified.

3. RESULTS AND DISCUSSION

3.1 LMB applications

3.1.1 Winchester

Selected PPCPs monitored were: triclosan (antibacterial), sulfapyridine, sulfamethoxazole (antimicrobials), cotinine (nicotine metabolite), atenolol (beta blocker), carbamazepine (anticonvulsant), fluoxetine (antidepressant), acetaminophen, naproxen, ibuprofen (analgesics), gemfibrozil (lipid regulator). PPCPs moved rapidly (within minutes) to tile drains (~0.8m depth) following LMB ap-

TABLE 1: Summary of selected municipal biosolid CEC studies. All municipal biosolids that were surface applied, with the exception of the AerWay® approach, were subjected to soil incorporation via light tillage implement within 24hrs post application.

Study Site	Biosolid Type and Application Rate*	Application Method(s)	Environmental Monitoring	Duration of Study	References
Winchester	LMB, 93,500 L ha^{-1}	Surface apply over aerated (AerWay® SSD) soil vs. surface apply on no-till soil followed by incorporation (to ~0.10 m)	Subsurface drainage (tile)	40 days post-application	Lapen et al., (2008); Gottschall et al., (2010)
Winchester	DMB, 8 Mg dry weight dw ha^{-1}	Direct DMB injection (to ~0.11 m) using the Terratec Environ. Ltd. direct injection system vs. surface apply on no-till soil followed by incorporation (to ~0.10 m)	Subsurface drainage (tile), groundwater, soil	~6 months post-application	Edwards et al., (2009)
Ottawa	DMB, 22 Mg dw ha^{-1}	Surface apply on no-till soil followed by incorporation (to ~0.10 m)	Subsurface drainage (tile), groundwater, soil, wheat grain	~1 year post-application	Gottschall et al., (2012; 2013, 2017)
London	LMB, 93,500 L ha^{-1}	Injection (to ~0.10 m) vs. surface apply followed by incorporation (to ~0.15 m)	Surface runoff	~9 months post-application	Topp et al., (2008)
London	DMB, 8 Mg dw ha^{-1}	Surface apply on no-till soil followed by incorporation (to ~0.15 m)	Surface runoff	~1 month post-application	Sabourin et al., (2009)

**LMB = liquid municipal biosolids, DMB = dewatered municipal biosolids*

TABLE 2: Selected CEC (classes) and measurement targets.

Contaminant Class	CEC	Measurement Targets	Study Sites; References
Polybrominated Diphenyl Ethers (PBDEs)/Other Brominated Flame retardants (BFRs)	BDE-47 BDE -99 BDE-153 BDE-154 BDE-183 BDE-209 Decabromodiphenyl ethane (DBDPE) 1,2-Bis(2,4,6-tribromophenoxy)ethane (BTBPE)	Subsurface drainage (tile), groundwater, soil, biosolid residues, wheat grain	Winchester, Ottawa; Gottschall et al., (2010; 2017)
Perfluoroalkyl Acids (PFAAs)	Perfluorooctanoic acid (PFOA) Perfluorooctane sulfonate (PFOS)	Subsurface drainage (tile), groundwater, soil, biosolid residues, wheat grain	Winchester, Ottawa; Gottschall et al., (2010; 2017)
Hormones and Fecal Sterols	Estrone Testosterone Desogestrel Androsterone Progesterone Coprostanol Cholesterol Cholestanol	Subsurface drainage (tile), groundwater,soil, biosolid residues, wheat grain	Ottawa; Gottschall et al., (2013)
PPCPs: Antidepressants Analgesics Lipid regulators Antimicrobials Beta blockers	Fluoxetine Ibuprofen Gemfibrozil Sulfamethoxazole Atenolol	Subsurface drainage (tile), groundwater, soil, biosolid residues, wheat grain	Winchester, Ottawa, London; Lapen et al., 2008); Gottschall et al., (2012); Topp et al. (2008); Sabourin et al., (2009)

plication, with surface spreading of LMB resulting in significantly higher ($p<0.05$) tile loads of PPCPs than surface spreading of LMB immediately preceded by aeration-based tillage. Maximum concentrations of PPCPs were detected exclusively where LMB was surface spread over no-till soil, ranging from 267 ng L^{-1} for atenolol, to 4117 ng L^{-1} for ibuprofen; sulfapyridine was only detected once above limits of quantitation (22.4 ng L^{-1}, at a surface spread over no-till plot). By aerating the soil using the AerWay® SSD system, application induced loads of PPCPs to tile drains via soil preferential flow paths (macropores) were critically reduced. For some of the more persistent PPCPs, there may be more parity in loading among the two land application methods over the longer term. But high concentration PPCP pulses during and immediately following land application were clearly dampened by the soil aeration methods deployed in the study. Figure 1 shows mass export of selected PPCPs.

Major polybrominated diphenyl ethers (PBDEs) monitored for this experiment were: BDE-47, -99, -153, -154, -183, and -209; Perfluoroalkyl acids (PFAAs) monitored included perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA). These compounds have human and environmental health implications (Thibodeaux et al., 2003; Costa and Giordano, 2007). Maximum concentrations in tile drainage ranged from 6-320 ng L^{-1} following LMB application, and all maximum values, like the PPCPs, were observed for the surface spread over no-tilled plots. Mass loads for PBDEs were significantly higher ($p<0.05$) for surface spreading vs. control/reference plots (where no amendment was applied), but there were no significant analyte load differences between surface spreading (over no-till) and aeration tilled plots. For PFAAs, only PFOS and PFOA were found above detectable limits in subsurface tile drainage, with maximum concentrations of 17 and 12 ng L^{-1}, respectively, on a surface spread and aeration plot, respectively.

3.1.2 London

The selected PPCP compounds monitored were: atenolol, carbamazepine, cotinine, gemfibrozil, naproxen, ibuprofen, acetaminophen, sulfamethoxazole and triclosan. Surface runoff (generated by rainfall simulator) for plots where LMB was injected (~0.1 m depth in soil), rarely had concentrations of PPCPs above limits of quantitation, while runoff from the surface spread plots ranged from 70-1477 ng L^{-1} (atenolol (70), carbamazepine (221), cotinine (83), gemfibrozil (597), acetaminophen (114), ibuprofen(1477), naproxen (509), triclosan (258)) 1 day post-application, generally declining thereafter following first order kinetics, with K (d^{-1}) values ranging from 0.023 for triclosan to 0.346 for sulfamethoxazole. Carbamazepine and triclosan were still detected from runoff events 266 days post-application. Results show that injection of biosolids prevents surface runoff of PPCPs, and that concentrations of selected compounds in runoff from surface applied amendment (followed by 'soil incorporation') could produce concentrations in toxicologically important ranges; notwithstanding cumulative inputs to downstream receptors. However, in terms of reducing inputs to subsurface drainage, injection into discrete furrows may augment loads of contaminants to subsurface tile drains in relation to application to surface application on tilled soil (Akhand et al., 2008). Figure 1 shows mass export of selected PPCPs.

3.2 DMB applications

3.2.1 Winchester

PPCPs monitored that were selected for discussion herein included: acetaminophen, fluoxetine, ibuprofen, gemfibrozil, naproxen, carbamazepine, atenolol, sulfamethoxazole, cotinine, triclosan, and triclocarban. There were no significant differences ($p>0.05$) in PPCPs loads in tile drainage among surface spread and directly injected DMB (0.05 m diam. injected continuously to a depth of ~ 0.11 m in soil) plots, although late study period (>100 days post-application) average loads were consistently higher from tiles of injected plots, but they were not different significantly ($p>0.05$). This was likely due to PPCPs in the injected DMB being more protected from photodegradation, higher soil temperatures, oxygen, and the more biologically active surface soils than the surface spread DMB which was more exposed to environmental elements and microorganisms in the surface soils. Hence, there may have been more persistence in PPCPs in the injected biosolids, in relation to those in biosolids spread on surface and lightly incorporated into soil. Maximum PPCP concentrations in subsurface tile drainage did not exceed many literature based aquatic toxicity thresholds except for one triclosan tile water sample from the surface spread plot. Surface spreading and direct injection of DMB resulted in lower concentrations of PPCPs in subsurface drainage than application of LMB, although PPCPs appeared to be more persistent in soil, especially for directly injected materials. However, by directly injecting DMB, problems associated with vector attraction and odour are minimized; without, as we have shown, increased liability of transport to subusurface hydrological receptors. Figure 1 shows mass export of selected PPCPs.

3.2.2 London

Selected compounds monitored were atenolol, carbamazepine, cotinine, caffeine, gemfibrozil, naproxen, ibuprofen, acetaminophen, sulfamethoxazole, triclosan and triclocarban. Maximum concentrations in surface runoff generated by rainfall simulator ranged from undetectable (gemfibrozil) to 110 ng L^{-1} (triclosan), and time to reach maximum concentration varied from 1 to 36 days post-application. The compounds with the least mass exported (<1% of that applied) had log Kow (octanol-water partition coefficient) values of ≥3.18 (triclocarban, triclosan, sulfamethoxazole, ibuprofen, naproxen and gemfibrozil), while those with >1% mass exported had log K_{ow} values of ≤2.45 (acetaminophen, carbamazepine, caffeine, cotinine, atenolol), indicating log K_{ow} may be a factor in determining runoff potential of these PPCPs. Figure 1 shows mass export of selected PPCPs.

3.2.3 Ottawa

The biosolids applied to the Ottawa site represented high instantaneous surface application rates of 22 Mg dw

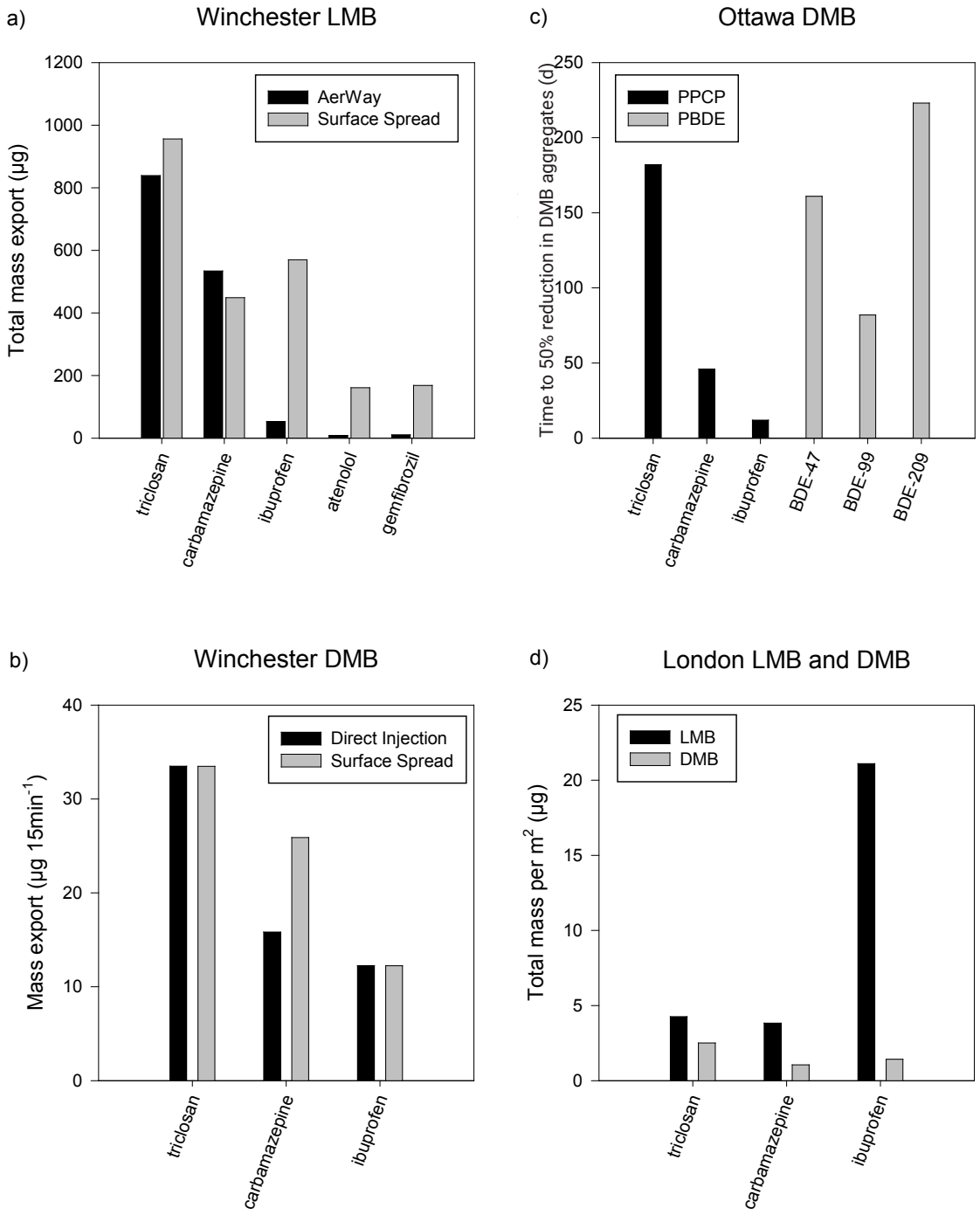

FIGURE 1: Selected results from biosolid studies: a) study period (40 d) PPCP mass load estimates (µg) in subsurface tile drainage associated with AerWay® LMB and surface+incorporate LMB application, b) maximum mass export (µg per 15 minutes) in subsurface tile drainage for selected PPCPs associated with DMB direct injection and DMB surface application, c) time to 50% reduction in concentration of CEC in DMB aggregates that were in soil following land application and, d) total mass per m2 (µg) of selected PPCPs in surface runoff from LMB (surface apply) and DMB (surface apply) applications (note: no PPCP concentrations for LMB injection surface runoff were above detectable limits).

ha^{-1}. Hormones (androsterone, desogestrel, estrone) were only detected on two occasions, up to ~2 months post-application in tile drainage (2-34 ng L^{-1}), but were not detected in groundwater (2 m depth). Sterols were detected up to ~1 yr post-application in tile drainage and sterol ratios were indicative of biosolid-borne contamination. The limited transport of hormones and sterols to subsurface tile drainage networks may be attributed to a combination of the hydrophobicity of these compounds and more limited macroporosity of the field soil (in relation to the Winchester study soils). The transitory contamination from hormones and sterols appears unlikely to result in any significant pulse exposure risk in subsurface drainage and groundwater, even at the high application rates examined.

Over 80 PPCPs were monitored, but only carbamazepine, ibuprofen, acetaminophen, triclosan, triclocarban, venlafaxine, and citalopram were detected in subsurface drainage (with concentrations ranging from 5–74 ng L^{-1}). No PPCPs were detected in groundwater >2m and those detected at 2m depth (ibuprofen, triclosan, triclocarban, venlafaxine) were only detected on one occasion within one month after DMB application. PPCPs persisted in DMB aggregates (intact DMB within the soil) up to ~1 yr post-application, however (Figure 1). But these persistent PPCPs were not critically detected in subsurface tile drainage and groundwater receptors. No PPCP was detected in wheat (grain) grown following land application.

PBDEs, other BFRs, and PFAAs were detected in subsurface drainage and 2m groundwater for up to ~1 yr post-application. Several compounds in subsurface drainage were detected at significantly higher ($p<0.05$) concentrations than reference plot (no DMB applied)/pre-application (DMB plot) concentrations (BDE-47, -100, and -153). PBDEs and PFAAs persisted up to ~1 yr post-application in DMB aggregates within the soil as well. Several PBDEs in DMB aggregates had concentration reductions >90% after 1 yr post-application, following an exponential decay pattern (Figure 1). No PBDEs or other BFRs were found in wheat grain. Although a considerable PBDE and PFAA load was applied at time of biosolid application (22 Mg dw ha^{-1}), only subsurface drainage showed significant increases of PBDEs relative to pre-application levels, and detection of PBDEs and PFAAs in subsurface drainage, groundwater, and soil indicated that atmospheric deposition was likely an important source of these compounds. In addition, post-application levels of PBDEs and PFAAs in the soil remained largely within background soil levels derived from the literature.

4. CONCLUSIONS

Transport of CECs from biosolids (liquid and solid) was tempered by limited macroporosity of soil, as well as application techniques that disrupted preferential flow paths to subsurface water resource receptors. Pre-tillage is crucial in this regard, and in cases where transport to subsurface drainage was rapid, as with the LMB application at Winchester, CEC concentrations peaked only briefly, and rarely exceeded concentrations typical of effluents associated with many waste water treatment plants. Further, although many CECs were detected in soil and water for extended periods following, in particular, dewatered biosolid applications, concentrations did not typically exceed many documented acute or chronic toxicity thresholds, and compounds were shown to dissipate considerably over time (i.e PBDEs, PPCPs). Nevertheless, cumulative effects and impacts of transformation products and metabolites needs better experimental documentation (Mompelat et al., 2009) in regard to the fate and transport pathways associated with land applied biosolids. Further, the implication of nonextractable residues on dissipation kinetics (Boxall et al., 2012) needs to be more succinctly examined, since dissipation could be very strongly linked to, among many things, the nature and mode of land application method.

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