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Short-Term Legacy Effects of Feedlot Manure Amendments on Water-Extractable Organic Carbon in a Clay Loam Soil Profile

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Short title: legacy manure effects on water-extractable carbon

Key words: legacy effects, feedlot manure, manure type, bedding material, water-extractable organic carbon, leaching potential

Abstract

Limited research exists on legacy effects of land application of feedlot manure on accumulation, redistribution, and leaching potential of water-extractable organic carbon (WEOC) in soil profiles. We sampled a clay loam soil at six depths (0-1.50 m) in 2016 two years after the last application (2014) of 17 continuous annual manure applications (since 1998). The amendment treatments were stockpiled (SM) or composted (CM) feedlot manure containing straw (ST) or wood-chip (WD) bedding at three application rates (13, 39, 77 Mg ha⁻¹ dry basis). There was also an unamended control (CON) and inorganic fertilizer (IN) treatment. The soil samples were analyzed for concentrations of WEOC. The total mass or accumulation of WEOC in the soil profile was greater ($P \leq 0.05$) by 1.2 to 3.3 times for the CM-ST-77 treatment compared to 12 of 14 other treatments, and it was significantly greater for amended than CON or IN treatments. The total WEOC mass was 14 to 20% greater for CM-ST than CM-WD, SM-ST, and SM-WD treatments; and it was 16 to 22% greater for CM than SM at the 39 and 77 Mg ha⁻¹ rates. The 77 Mg ha⁻¹ rate of the four manure type-bedding treatments had the significantly greatest (by 37-527%) concentrations of WEOC at the six depths compared to other treatments, suggesting greater redistribution and leaching potential. Significant manure effects occurred on soil WEOC two years after manure was last applied following 17 continuous applications, and indicated an increased risk of leaching potential at the higher application rate.

Introduction

Discontinued long-term application of feedlot manure to cropland may result in legacy (residual) treatment effects on water-extractable organic C (WEOC) in the soil profile. Accumulation and redistribution of WEOC in the soil profile may increase the labile pool of C and also increase the leaching potential. The WEOC in soil is important because its readily degraded compounds may influence biological, chemical, and physical processes in soil, transport of metals and pesticides, mineral weathering, greenhouse gas production, C balance, and water quality (Chantigny 2003; Kindler et al. 2011; Lu et al. 2011; Zhao et al. 2012). Degradation of organic C is generally greater for composted (CM) than stockpiled (SM) feedlot manure types during processing of manure prior to land application (Larney et al. 2006). Straw (ST) is more soluble than wood (WD) in water, and ST is also decomposed or mineralized faster than WD bedding because of its lower C:N ratio (Miller et al. 2013). Therefore, concentrations of WEOC in amended soils during the legacy period might be greater for less decomposed feedlot manure types (SM>CM), and greater for more soluble and decomposable ST than WD bedding material (ST>WD). Miller et al. (2011) studied accumulation and redistribution of chloride, nitrate, and soil test P in soils profiles under these feedlot amendments, but not WEOC.

Numerous abiotic and biotic factors control the concentration of extractable organic C (EOC), WEOC, and dissolved organic C (DOC) in soils (McGill et al. 1986; Kalbitz et al. 2000; Chantigny 2003). The EOC is extracted from soil using various reagents, WEOC is a subset of EOC and is extractable with water, and DOC is in the “in situ” component in the soil solution or pore water generally sampled with lysimeters or in leachate (Chantigny 2003). Soil pH, phosphate, temperature, wet-dry cycles, water content, freeze-thaw cycles, liming, and organic fertilization generally have a strong positive influence on DOC (Kalbitz et al. 2000). Biological factors such as microbial activity and proportion of fungi mostly have a positive influence on WEOC or DOC. In contrast, Al and Fe oxides, clay content, and ionic strength all have a strong

negative effect. Soil pH and ionic strength determine the solubility of organic matter, whereas Al- and Fe-oxides determine sorption-desorption processes (Chantigny 2003). The latter author reported that the production and consumption of WEOC depends mainly on the microbial activity and the equilibrium with the solid phase of soil organic matter. McGill et al. (1986) stated that production of water-soluble organic C in soil was controlled by desorption from soil colloids, dissolution from litter, exudation, sloughing, and exfoliation from plant roots, and hydrolysis of insoluble soil organic polymers.

Long-term application of manure to cropland may initially cause an accumulation of WEOC in soil because these soluble compounds are generally present in the amendments (Chantigny 2003; Hao et al. 2014). In a review paper, Chantigny (2003) reported that many short-term studies have reported an immediate and significant increase in WEOC in soil amended with crop residues or manure compared to unamended soils. But the WEOC appeared to be readily decomposed, was not persistent, and did not leach to deeper soil layers because of rapid decomposition. Chantigny (2003) also noted that longer-term studies have found an increase in WEOC for organic farming or organically-amended soils compared to conventional farming using inorganic fertilizers, which was attributed to long-term repeated additions of organic matter (Zsolnay and Gorlitz 1994; Leinweber et al. 1995).

Some more recent studies have reported both accumulation and redistribution of EOC or WEOC in soils (Gonet and Debska 2006), whereas others found evidence of accumulation but not redistribution or EOC/WEOC or DOC to depth (Angers et al. 2006; Long et al. 2015; He et al. 2017). Angers et al. (2006) reported accumulation of WEOC in soils (Quebec) amended with liquid and solid cattle manure at the 0 to 0.10 m depth but not 0.10 to 0.30 m depth compared to unamended control and mineral fertilizer, and attributed no redistribution to decomposition and adsorption. Gonet and Debska (2006) found greater accumulation of EOC at both the 0 to 0.25 m and 0.25 to 0.50 m depths for a soil in Poland amended with cattle slurry application

compared to mineral fertilizer. In a lysimeter study in China, Long et al. (2015) reported greater DOC in the surface soil for liquid pig manure compared to unamended control, but not at deeper depths to 90 cm. He et al. (2017) found that poultry litter application to five cultivated soils in Texas increased WEOC in the surface (0-15 cm) soil compared to the unamended control; but they did not sample deeper depths.

We are unaware of any studies that have focused on the legacy effects of discontinued application of feedlot manure amendments on accumulation, redistribution, and leaching potential of WEOC in soils. The overall objective of our study was to examine the short-term legacy effects of different feedlot amendments on accumulation, redistribution, and leaching potential of WEOC in the soil profile. The first specific objective was to determine if significant legacy effects persisted two years after the last application of 17 yr of continuous annual manure applications. The second specific objective was to determine if accumulation and redistribution of WEOC would be greater for less decomposed SM than CM, greater for more water-soluble and decomposable ST than WD bedding, greater with increased application rates, and greater for amended than unamended treatments.

Materials and Methods

A long-term field experiment (randomized complete block design) was started in the fall of 1998 at Lethbridge, in southern Alberta. A detailed description of the site and experimental design has been already reported (Miller et al. 2004; 2009). The clay loam soil is classified as a Dark Brown Chernozem. Briefly, a factorial design of two manure types (composted, CM; stockpiled, SM), two bedding materials (straw, ST; wood chips, WD), and three application rates (13, 39, and 77 Mg ha⁻¹ dry wt.) was evaluated (12 treatments), plus an unfertilized control (CON), and an inorganic fertilizer (IN) treatment (100 kg N ha⁻¹ as NH₄NO₃ and 17 kg P ha⁻¹ as triple super-phosphate), resulting in fourteen treatments (25 x 6 m plot size) with four replications. No

inorganic fertilizer was co-applied with the organic amendments. All amendments and inorganic fertilizer were annually applied in the fall from 1998 to 2014 (17 yrs). No feedlot amendments were applied after the last application in 2014, but inorganic fertilizer was still annually applied to the IN treatments. The manure was incorporated to a depth of 0.20 m. The abbreviations used to refer to the amendment treatments in this study follow the format of manure type-bedding-rate (e.g., CM-ST-13, etc.). Irrigated barley (*Hordeum vulgare*) for silage was grown on the plots in all years.

Soil samples were taken from six depths (0-0.15, 0.15-0.30, 0.30-0.60, 0.60-0.90, 0.90-1.20, 1.20-1.50 m) in the fall of 2016 (legacy phase). This sampling was two years after the last application (2014) of 17 continuous annual manure applications (since 1998). Soil sampling was conducted at one sampling location per treatment plot using a Giddings (Giddings Machine Co., Windsor, CO, USA) coring machine mounted on the back of a truck. Soil samples were air-dried and then ground through a 2-mm sieve.

Water-extractable organic C (WEOC) was determined on 1:5 extracts [10 g air-dried soil:50 mL deionized cold (20°C) water]. Samples were shaken for 1 h, filtered through Q2 filter paper (Fisher Scientific, Hampton, NH, USA), and then filtered through 0.45-µm syringe filters (Chromatographic Specialties Inc., Brockville, ON, Canada). The extracts were then analyzed for non-purgeable organic C (WEOC) using a Shimadzu TOC-VCSH Organic Carbon Analyzer (Shimadzu Scientific, Inc., Columbia, MD). Amendment samples (air-dried and ground to < 2-mm) were also taken during fall manure application in four years (2008, 2010, 2012, 2014) and analyzed for WEOC on 1:5 extracts (6 g air-dried amendment:30 mL deionized water). There were 15 subsamples for each type of amendment for each year. About 2 to 3 g were taken from each subsample to make a composite sample for each amendment for each year. This process of making a composite sample was repeated four times for the four organic amendments for a total of 16 samples for each year. The composite sample was then used for the analysis.

Total C (organic and inorganic C) was determined for the same six depths in the soil profile from soil samples collected in the fall of 2013. Although these samples were collected 3 yr prior to the 2016 sampling in this study, total C should remain relatively constant over this time frame. Soil samples were fine-ground ($<150\ \mu\text{m}$) and then total C determined using the Dumas automated combustion technique (Nelson and Sommers 1996) using a CNS analyzer (Carlo Erba, Milan, Italy). The percent of total C that was WEOC was then determined.

Bulk densities of the surface 0 to 0.15 m depth were obtained from undisturbed soil cores obtained by hand in the fall of 2014. Soil bulk density of the deeper five depths (0.15-0.30, 0.30-0.60, 0.60-0.90, 0.90-1.20, 1.20-1.50 m) was determined from soil cores obtained at 11 locations adjacent to the experiment plots in 1999 using a truck-mounted Giddings coring machine. Soil bulk densities below the depth of manure incorporation ($> 0.20\ \text{m}$) should remain relatively constant over time. Soil bulk density values were used to determine the mass (kg ha^{-1}) of WEOC at each depth and total mass (Mg ha^{-1}) in the soil profile. Precipitation and pan evaporation during the experiment (1999-2016) were determined from the AAFC weather station located at the Lethbridge Research and Development Center. Pan evaporation was not available for 2015 and 2016.

Statistical Analysis

A MIXED model analysis (SAS Institute Inc. 2005) was conducted using treatment as the fixed effect in the model and replicate as the random effect. The UNIVARIATE procedure with normal plot option was utilized to test the normality of the sample and if a logarithmic transformation was required. Main treatment and interaction effects were evaluated using a least significant difference (LSD) test or Tukey-Kramer test (≥ 5 means). A probability level of $P \leq 0.05$ was considered significant for F statistic values and means comparisons.

Two Mixed model analyses were conducted. The first analysis examined the effect of all 14 treatments on the dependent variables, including the unamended CON and IN treatments. This analysis was conducted for the total mass of WEOC in the soil profile (0-1.50 m), as well as for each of the six depths. An Estimate statement was also used to compare unamended CON or IN treatments versus the amended treatments. The second analysis examined the effect of the three treatments factors of manure type (SM, CM), bedding material (ST, WD), and application rate (13, 39, 77 Mg ha⁻¹) on the concentration of WEOC in the soil profile. The CON and IN treatments were omitted for this second analysis because there was only one level of these factors.

Results

Weather During the Experiment (1999-2016)

Annual precipitation at Lethbridge from 1999 to 2016 ranged from 178 mm to 637 mm or 44% to 157% of the long-term (30-yr) average of 406 mm. Precipitation from January to August of 2016 prior to soil sampling in September was 275 mm or 90% of the long-term average (306 mm) for this period. Annual pan evaporation from 1999 to 2014 ranged from 1278 to 1864 mm or 98% to 143% of the long-term average (1299 mm).

Total C and WEOC in Amendments

The total C of the amendments has been previously reported and was greatest for SM-WD (340 g kg⁻¹), followed by CM-WD (269 g kg⁻¹), SM-ST (239 g kg⁻¹), and then CM-ST (191 g kg⁻¹) amendments (Miller et al. 2009). The WEOC concentration in the amendments applied was highest for SM-ST, followed by SM-WD, CM-ST, and then CM-WD (Table 1). The WEOC content was 49% greater for SM than CM when averaged across the two bedding materials; and it was 28% greater for ST than WD when averaged across the two manure types.

Total Mass of WEOC in the Soil Profile

The total mass of WEOC for the CM-ST-77 treatment in the soil profile (0-1.50 m) was significantly ($P \leq 0.05$) greater than 12 other manure treatments except for CM-WD-77 treatment (Figure 1). The estimate comparisons showed that total mass of WEOC in the soil profile was significantly greater for the amended treatments compared to unamended CON or IN treatments (data not shown). There was a significant manure type x bedding material interaction on total mass of WEOC in the soil profile (0-1.50 m), where mean values were 14 to 20% greater for CM-ST than the other three treatments (Table 2). A significant manure type x manure rate interaction also occurred (Table 2). Mean values at the 39 and 77 Mg ha⁻¹ application rates were 16 to 22% greater for CM than SM, but mean values were similar at the 13 Mg ha⁻¹ rate.

Concentration of WEOC at Depth in the Soil Profile

The estimate comparisons showed that WEOC concentration was significantly greater in amended than CON or IN treatments at four or five depths (Table 3). There were significant treatment effects (all 14 treatments) on WEOC concentration at each of the six depths (Table 3). The 77 Mg ha⁻¹ treatments generally had significantly greater (by 37 to 527%) WEOC than most of the other treatments across all six depths, and the percentage increases declined with depth. The concentration of WEOC exceeded the unamended CON for five of six depths for three treatments (CM-WD-77, SM-ST-77, SM-WD-77), the top 3 depths for two treatments (CM-ST-39, CM-ST-77), the top two depths for three treatments (CM-WD-39, SM-ST-39, SM-WD-39), and no depths for four treatments (CM-ST-13, CM-WD-13, SM-ST-13, SM-WD-13).

The depth distribution of WEOC in the soil profile (0-1.50 m) for all 14 treatments is shown in Figure 2. Concentrations were greater for amended than unamended CON and IN treatments, particularly at the 39 and 77 Mg ha⁻¹ rates for the 0 to 0.60 m depth. Maximum

accumulation of WEOC for the amended treatments generally occurred in the surface (0-0.15 m depth) soil. The one exception was for CM-ST-77, where maximum accumulation occurred at the 0.15-0.30 m depth.

Significant treatment factor effects occurred on WEOC concentration at all six depths in the soil profile (Table 4). There was a significant bedding material x manure rate effect at the 0 to 0.15 m depth, where mean values were 32% greater for WD than ST at the 77 Mg ha⁻¹ rate (Table 5). At the 0.15 to 0.30 m depth, there were significant two-way interactions for all three treatment factor combinations (Table 4). For the manure type x bedding material interaction, mean values were 28 to 36% greater for CM-ST than other three treatments (Table 5). For the manure type x manure rate interaction, mean values were 21 to 22% greater for CM than SM at the 39 and 77 Mg ha⁻¹ rates (Table 5). For the bedding material x manure rate interaction, mean values were 20 to 35% greater for ST than WD at the two highest rates (Table 5).

There was a significant manure rate effect at the 0.30 to 0.60 m depth, where mean values were 64 to 137% greater at the 77 Mg ha⁻¹ rate than 39 and 13 Mg ha⁻¹ rates (Table 4). A significant manure rate effect occurred at the 0.60 to 0.90 m depth, where mean values were 48 to 77% greater for 77 than 39 and 13 Mg ha⁻¹ rates (Table 4). A significant manure rate effect also occurred at the 0.90 to 1.20 m depth, where mean values were 37 to 48% greater at 77 Mg ha⁻¹ rate than the two lower rates. At the 1.20 to 1.50 m depth, there were significant manure type x bedding material and manure type x manure rate effects (Table 4). For the manure type x bedding material effect, mean values were 12% greater for SM-WD than SM-ST (Table 5). For the manure type x manure rate effect, mean values were 23% greater for SM than CM at the 77 Mg ha⁻¹ rate (Table 5).

Relationship of WEOC with Total C in Soil

The percentage of WEOC as a fraction of total C ranged from 0.1 to 3.5% (mean of 0.5%) when averaged over all treatments and depths (data not shown). The percentage values ranged from 0.4 to 0.9% for the amended treatments, and were 0.3% for the CON and IN treatments. The WEOC fraction was similar (0.6%) for CM and SM manure types, and also for ST and WD (0.5-0.6%) bedding materials. For the three application rates, the percentage of WEOC was 0.5, 0.6, and 0.7% for the 13, 39, and 77 Mg ha⁻¹ rates, respectively. The WEOC fraction ranged from 0.3 to 1% for the 0 to 0.15 m depth, and from 0.4 to 3.5% for the 0.15 to 0.30 m depth, and was < 2% at greater depths.

The correlation coefficients (*r*) between WEOC and total C ranged from 0.42 (*P*=0.08, *n*=19) to 0.86 (*P* < 0.0001, *n*=20) for the 12 amended treatments, and was -0.25 (*P*=0.29, *n*=20) for the CON and 0.50 (*P*=0.03, *n*=20) for the IN treatment (data not shown). Correlation coefficients were greater for SM (*r*=0.77, *P* < 0.0001, *n*=115) than CM (*r*=0.65, *P* < 0.0001, *n*=124), and for WD (*r*=0.82, *P* < 0.0001, *n*=120) than ST (*r*=0.60, *P* < 0.0001, *n*=119). Correlations (*P* < 0.0001) were 0.61, 0.70, and 0.67 for the 13, 39, and 77 Mg ha⁻¹ rates, respectively. The *r* values were 0.85 (*P* < 0.0001, *n*=56) at the 0 to 0.15 m depth, 0.47 (*P*=0.0003) at the 0.15 to 0.30 m depth, 0.23 (*P*=0.09) at the 0.30 to 0.60 m depth, 0.12 (*P*=0.36) at the 0.60 to 0.90 m depth, and 0.33 (*P*=0.01) at the 0.90 to 1.20 m depth.

Discussion

Significant treatment effects on total WEOC concentrations in the soil profile (0-1.50 m) and at each of the six depths, as well as significantly greater total concentrations in amended than unamended CON or IN treatments, was evidence that legacy treatment effects still persisted two years after the last application of 17 continuous annual manure applications. In a review paper, Chantigny (2003) reported that persistence of WEOC in soil is generally short-lived immediately after application of organic amendments but may persist in longer-term studies,

and persistence was increased by greater application rates. This was consistent with our findings where we found that the treatment with significantly greatest total WEOC mass in the soil profile (0-1.5 m) or concentrations of WEOC at each of the six depths was always associated with the highest application rate of 77 Mg ha⁻¹.

Our finding of significant legacy effects on WEOC was generally consistent with previous findings on other biochemical and microbial properties (Ginting et al. 2003; Wyngaard et al. 2016; Zhang et al. 2018). For our study, we speculate that significant legacy effects still persisted two years after 17 continuous applications were terminated because the soil microbial community was still responding to the long-term additions of organic matter and nutrients. Organic amendments can sustain soil microbes for long periods of time because organic fertilizers release nutrients gradually over time (Zhang et al. 2018).

Significantly greater total mass of WEOC in the soil profile for CM than SM with ST or WD bedding, and significantly greater total mass for CM than SM at 39 and 77 Mg ha⁻¹ rates, indicated greater accumulation of WEOC for more decomposed CM than SM. For the treatment factor analysis at each of the six depths, the expectation of greater concentrations and redistribution of WEOC for SM than CM was generally not supported for the manure type x bedding material and manure type x manure rate interactions, and was not consistent with concentrations in the amendments. The one exception was for the manure type x manure rate interaction at 1.20 to 1.50 m depth, where concentration of WEOC was significantly greater for SM than CM at the 77 Mg ha⁻¹ rate. This latter finding was consistent with Li et al. (2016) who reported greater leaching of non-purgeable organic C in leachate from soil cores amended with fresh compared to composted beef feedlot manure.

We expected greater total mass and concentrations of WEOC for less decomposed SM than CM because WEOC was 49% greater for the less decomposed SM than CM amendment.

Miller et al. (2012) reported similar WEOC in surface (0-15 cm) soil (fall sampling) of CM- and SM-amended soils for 3 of 4 yr, and significantly greater WEOC for CM than SM in one of 4 yr, after 9 to 12 continuous applications of amendments. The finding of generally similar or greater soil WEOC for CM than SM in our study might be explained by recent research that reported that WEOC may contain both labile and stable forms of soil organic C (Poeplau et al. 2018). The latter authors also concluded that it may be difficult to use WEOC as a soil organic C fraction with distinct turnover rate. In addition, the slightly greater pH of the surface (0-0.15 m) soil for CM (7.22 ± 0.03) than SM (7.12 ± 0.03) treatment after 15 continuous applications (Miller et al. 2016) may have contributed to similar or greater WEOC for CM than SM. Soil pH generally has a strong positive influence on DOC in soil (Kalbitz et al. 2000).

Significantly greater total mass of WEOC for ST than WD for composted manure indicated greater accumulation for more soluble and decomposable ST than WD bedding, and was consistent with greater WEOC concentration for ST than WD for composted manure amendment. In contrast, similar total mass of WEOC for ST and WD for stockpiled manure was not consistent with greater WEOC concentration for ST than WD for stockpiled manure amendment. The expectation for greater concentration and redistribution of WEOC for more soluble and decomposable ST than WD at each depth was generally not supported. The exceptions were at the 0.15 to 0.30 m depth where greater WEOC concentration occurred for ST than WD with composted manure, and greater concentration for ST than WD at the 77 Mg ha⁻¹ rate. The relative difference in electrical conductivity (ionic strength) was greater for ST than WD with composted than stockpiled manure (Miller et al. 2016), which may have been a contributing factor to significant bedding effect for CM but not SM.

A positive response of total mass and concentrations of WEOC in the soil profile to application rate was consistent with greater inputs of amendments with relatively high WEOC concentrations (3.9-7.4 g kg⁻¹). Our finding was also consistent with other soil profile studies of

WEOC (Kalbitz et al. 2000; Chantigny 2003;; Liu et al. 2013). Miller et al. (2017) found that total C in surface soil after 17 continuous applications was also significantly increased with each successive application rate.

The CM-ST-77 treatment had significantly greater total mass of WEOC in the soil profile compared to 12 of 14 total treatments. This finding was not consistent with either the trend in WEOC concentration (SM-ST>SM-WD>CM-ST>CM-WD) or total C (SM-WD>CM-WD>SM-ST>CM-ST) of the amendments. Mixing of the amendment with the soil, and subsequent organic matter-mineral interactions, may have obscured the amendment effect on total mass of WEOC in the soil profile. The pH of the surface soil for the CM-ST-77 treatment (7.40) was higher than 12 other treatments (6.94-7.35) after 15 continuous applications (Miller et al. 2016). This higher pH may have contributed to greater total mass of WEOC for CM-ST-77.

Significantly greater (by 37-527%) concentrations of WEOC for the 77 Mg ha⁻¹ rate of the four manure type-bedding treatments than certain other treatments (including CON and IN) at the six depths was clear evidence for greater redistribution and increased risk of leaching potential. This was also consistent with significantly greater WEOC concentrations for amended than CON or IN treatments at the top four or five depths. This suggested that increased risk of leaching of WEOC at depth is induced by repeated and higher applications over several years. Some studies have reported both accumulation and redistribution of EOC or WEOC in soils (Gonet and Debska 2006), whereas others found evidence of accumulation but not redistribution of EOC/WEOC or DOC to depth (Angers et al. 2006; Long et al. 2015; He et al. 2017).

Concentrations of WEOC at the 0 to 0.15 m depth for the amended treatments ranged from 208 to 499 mg kg⁻¹, and were significantly and considerably lower for the unamended CON (79.6 mg kg⁻¹) and IN (87.8 mg kg⁻¹) treatments. Since WEOC in soil seldom exceeds 200 mg

kg⁻¹ (McGill et al. 1986), the concentrations in the surface soils of our amended treatments could be classified as extremely high. The increase in soil WEOC from the amendments compared to the CON ranged from +128 to +411 mg kg⁻¹. This increase was consistent with Chantigny (2003) who reported (in a review paper) that land application of organic amendments (dairy manure or farmyard manure) caused an increase of +7 to +422 mg kg⁻¹ in EOC and/or WEOC concentrations.

Relationship of WEOC with Total Carbon in Soil

The range (0.1-3.5%) in WEOC as a fraction of total C in our soil was generally consistent with previous studies that reported that WEOC generally represents < 4% of the total C or total organic C in agricultural soils (McGill et al. 1986; Zsolnay 1996; Angers et al. 2006; Hamkalo and Bedernichek 2014; Benbi et al. 2015; Awale et al. 2017). The percentage values in our study may have been slightly underestimated at deeper depths because of increasing inorganic C. The percentage of WEOC (of total C) in SM and CM with ST bedding is 6 to 7% (Miller et al. 2012), and in feedlot manure in southern Alberta it is 6% (Hao et al. 2014). The considerably lower percentage of WEOC in the soil than amendment was likely due to rapid decomposition of soluble C in the soil (Chantigny 2003).

Positive and generally significant correlations between WEOC and total C for the amended treatments ($r=0.42-0.86$), and a negative (nonsignificant) correlation for the unamended CON ($r=-0.25$), suggested that the source of the WEOC in the soil of the amended treatments was likely the organic matter in the amendments, and not native soil organic matter. This finding was consistent with more labile C in feedlot manure than in the soil because manure-C has undergone less decomposition than soil-C. In a meta-analysis of 42 research articles, Maillard and Angers (2014) observed a dominant effect of cumulative manure-C input on soil organic C response that explained at least 53% of the variability in soil organic C stock

differences compared to unamended or inorganic fertilized reference treatments. They also reported that the fraction of applied organic matter that was transformed into soil organic matter or global manure-C retention coefficient was 12% over 18 yr. A stronger positive correlation with WEOC and total C for SM ($r=0.77$) than CM ($r=0.60$), and for WD ($r=0.82$) than ST ($r=0.60$), suggested a closer and stronger relationship between these two C pools for SM than CM and for WD than ST. As expected, correlations were strongest in the surface (0-0.15 m) soil ($r=0.85$) with the highest soil organic matter content, and then r values ($r < 0.47$) decreased with depth. Positive and significant correlations ($r=0.76$ to 0.80) have been reported between WEOC and total organic C in arable soils (Hamkalo and Bedernichek 2014; Haney et al. 2014; Awale et al. 2017). But some have reported poor correlations ($r=0.24$) in other arable soils (Benbi et al. 2015).

Conclusions

A shift by feedlot producers from long-term continuous to short-term discontinued applications of these feedlot manure amendments may still cause significant treatment effects two years later. A shift by feedlot producers from more traditional to less common manure management practices such as SM to CM manure type and ST to WD bedding will likely have no or little effect on WEOC in the soil, but there were some exceptions. In contrast, a shift to higher application rates will most likely have the strongest effect and increase accumulation and redistribution of WEOC in the soil, which may increase the leaching potential. Future research could study water-soluble C in pore-water within and below the root zone to confirm if leaching is occurring to deeper depths, and also investigate leaching of water-soluble organic nitrogen.

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Figure Captions

Figure 1. Total mass (Mg ha^{-1}) of water-extractable organic C (WEOC) in 2016 (2 yr after 17 continuous annual manure applications) in the soil profile (0-1.50 m depth) for composted manure with straw bedding (CM-ST), composted manure with wood-chip bedding (CM-WD), stockpiled manure with straw bedding (SM-ST), and stockpiled manure with wood-chip bedding (SM-WD) in relation to application rates of 0 (unamended control), 13, 39, and 77 Mg ha^{-1} (dry wt.). The total mass of WEOC for the inorganically-fertilizer treatment (IN) is not shown, and was 0.96 Mg ha^{-1} .

Figure 2. Concentrations of water-extractable organic C (WEOC) in 2016 (2 yr after 17 continuous annual manure applications) at six depths from 0 to 1.50 m for composted manure with straw bedding (CM-ST) (a), stockpiled manure with straw bedding (SM-ST) (b), composted manure with wood-chip bedding (CM-WD) (c), and stockpiled manure with wood-chip bedding (SM-WD) (d). The line graphs are 13, 39, and 77 Mg ha^{-1} application rates of amendment, as well as inorganic fertilizer (IN) and unamended control (CON) treatments.

Table 1. Concentrations of water-extractable organic carbon (WEOC) in the four feedlot (beef cattle) amendments applied to manured plots. The concentrations averaged over the two manure types and bedding materials are also shown.

Amendment ^a	WEOC
	g kg ⁻¹
SM-ST	7.4±0.9
SM-WD	5.4±0.8
CM-ST	4.6±0.6
CM-WD	3.9±0.5
SM	6.4±0.7
CM	4.3±0.4
ST	6.0±0.7
WD	4.7±0.5

^aSM, stockpiled feedlot manure; CM, composted feedlot manure; ST, straw bedding; WD, wood-chip bedding

Table 2. Influence of manure type, bedding, and application rate on total mass per hectare of water-extractable organic C (WEOC) in the soil profile (0-1.50 m)

Treatment ^a	WEOC
	Prob > F
Type (T)	0.0014
Bedding (B)	0.0523
Rate (R)	<0.0001
TxB	0.0266
TxR	0.0453
BxR	0.1532
TxBxR	0.1290
TxB	Mg ha ⁻¹
CM-ST	2.26±0.22a
CM-WD	1.98±0.18b
SM-ST	1.88±0.16b
SM-WD	1.89±0.17b
TxR	
CM-13	1.35±0.05a
SM-13	1.36±0.06a
CM-39	2.12±0.11a
SM-39	1.74±0.08b
CM-77	2.93±0.14a
SM-77	2.53±0.09b

Note: Values (total mass) are the mean±standard error. Means within a column (within manure type, bedding, or bedding x rate interaction) not sharing a lowercased italic letter differ significantly at the $P \leq 0.05$ level (LSD test).

^aSM, stockpiled feedlot manure; CM, composted feedlot manure; ST, straw bedding; WD, wood-chip bedding; 13, 39, and 77 Mg ha⁻¹ application rates (dry basis)

Table 3. Influence of all treatments on concentration of water-extractable organic carbon (WEOC) at the six depths throughout the soil profile

Treatment ^a	0-0.15 m	0.15-0.30 m	0.30-0.60 m	0.60-0.90 m	0.90-1.20 m	1.20-1.50 m
	mg kg ⁻¹					
CM-ST-13	183±13.7 ^{ef}	159±16.2 ^{ef}	69.2±7.3 ^{de}	39.7±4.7 ^c	24.1±2.3 ^{bc}	17.7±0.5 ^c
CM-ST-39	340±21.7 ^{bcd}	329±22.9 ^b	117±5.0 ^{bc}	52.8±3.1 ^{abc}	29.4±2.4 ^{abc}	21.4±1.3 ^{bc}
CM-ST-77	471±72.9 ^{ab}	522±67.1 ^a	192±12.8 ^a	55.7±4.7 ^{abc}	30.1±2.3 ^{abc}	22.4±1.9 ^{abc}
CM-WD-13	208±20.6 ^{def}	138±13.3 ^{ef}	75.2±6.5 ^{cde}	36.4±3.7 ^c	19.8±0.9 ^c	17.0±0.9 ^c
CM-WD-39	329±20.0 ^{cd}	217±21.5 ^{de}	102±6.1 ^{cd}	42.0±4.1 ^{bc}	23.7±2.7 ^{bc}	18.0±2.0 ^{bc}
CM-WD-77	499±4.9 ^a	334±13.0 ^{bc}	174±14.4 ^a	74.4±4.7 ^a	37.2±3.3 ^a	20.4±0.4 ^{bc}
CON	79.6±1.9 ^f	86.6±5.3 ^f	62.6±3.4 ^{de}	34.8±2.7 ^c	23.1±0.9 ^{bc}	18.5±0.6 ^{bc}
IN	87.8±3.3 ^f	87.5±2.0 ^f	45.3±12.4 ^e	35.1±6.3 ^c	20.4±2.0 ^c	18.6±1.0 ^{bc}
SM-ST-13	170±16.3 ^{ef}	146±4.2 ^{ef}	71.1±7.4 ^{cde}	36.1±6.1 ^c	21.8±1.7 ^c	17.0±0.7 ^c
SM-ST-39	308±37.0 ^{cde}	240±9.8 ^{bcde}	99.6±9.4 ^{cd}	40.5±5.9 ^{bc}	20.4±4.4 ^c	16.2±1.4 ^c
SM-ST-77	362±14.8 ^{abc}	328±4.5 ^{bcd}	160±15.7 ^{ab}	65.8±5.6 ^{ab}	32.5±1.4 ^{abc}	24.5±0.6 ^{ab}
SM-WD-13	213±18.3 ^{cdef}	142±3.6 ^{ef}	77.9±7.0 ^{cde}	42.0±3.1 ^{bc}	25.9±1.9 ^{abc}	17.5±0.7 ^c
SM-WD-39	313±31.5 ^{cde}	223±10.6 ^{cde}	103±10.3 ^{cd}	45.6±6.2 ^{bc}	22.9±2.7 ^{bc}	20.2±2.1 ^{bc}
SM-WD-77	497±31.1 ^a	306±14.3 ^{bcd}	172±8.6 ^a	76.4±8.4 ^a	35.8±4.9 ^{ab}	28.1±2.1 ^a
Prob > F	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Estimate ^b						
CON vs AMEND	-245 ^{***}	-170 ^{***}	-55.1 ^{***}	-15.8 ^{**}	-3.8NS	-1.6NS
IN vs AMEND	-236 ^{***}	-170 ^{***}	-72.3 ^{***}	-15.5 ^{**}	-6.6 [*]	-1.5NS

Note: Values (concentrations) are the mean±standard error. Means within a column not sharing a lowercased italic letter differ significantly at the P ≤ 0.05 level (Tukey-Kramer test)

^aSM, stockpiled feedlot manure; CM, composted feedlot manure; ST, straw bedding; WD, wood-chip bedding; 13, 39, and 77 Mg ha⁻¹ application rates (dry basis)

^bEstimate values are negative when treatments 1 < treatments 2, and are positive when treatments 1 > treatments 2. For example, a negative estimate value for CON vs MAN indicates that mean value for MAN is greater than CON. Estimate values indicate significant differences at 0.05 (*), 0.01 (**), and 0.001 (***) levels. NS, not significant (P > 0.05 level).

Table 4. Influence of treatment factors on concentration of water-extractable organic carbon (WEOC) at the six depths throughout the soil profile

Treatment Factor	Depth (m)					
	0-0.15 m	0.15-0.30 m	0.30-0.60 m	0.60-0.90 m	0.90-1.20 m	1.20-1.50 m
	Prob > F					
Type (T)	0.2496	<0.0001	0.1830	0.7691	0.5873	0.6876
Bedding (B)	0.0004	<0.0001	0.8866	0.1500	0.4678	0.4539
Rate (R)	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
TxB	0.2166	0.0005	0.1450	0.3494	0.1850	0.0324
TxR	0.9107	0.0098	0.3764	0.3747	0.1929	0.0061
BxR	0.0015	0.0241	0.6554	0.0558	0.1948	0.7064
TxBxR	0.9922	0.1292	0.5338	0.2484	0.2054	0.6334
Rate effect ^a	mg kg ⁻¹					
13	NA	NA	73.3±3.3c	38.5±2.1b	22.9±1.0b	NA
39	NA	NA	106±3.8b	46.0±2.5b	24.7±1.6b	NA
77	NA	NA	174±6.6a	68.1±3.4a	33.9±1.6a	NA

Note: Values (concentrations) are the mean±standard error. Means within a column not sharing a lowercased italic letter differ significantly at the $P \leq 0.05$ level (LSD test). NA, not applicable because of significant interaction effect (see Table 5)

^a13, 39, and 77 Mg ha⁻¹ application rates (dry basis)

Table 5. Two-way treatment interaction effects on concentration of water-extractable organic carbon (WEOC) at the six depths throughout the soil profile

Treatment factor Interaction ^a	Depth (m)					
	0-0.15	0.15-0.30	0.30-0.60	0.60-0.90	0.90-1.20	1.20-1.50
mg kg ⁻¹						
Type x Bedding						
CM-ST	NA ^b	305±37.1a	NA	NA	NA	20.6±0.9ab
CM-WD	NA	229±25.8b	NA	NA	NA	19.4±1.1ab
SM-ST	NA	238±24.8b	NA	NA	NA	19.5±1.3b
SM-WD	NA	224±20.9b	NA	NA	NA	21.9±1.6a
Type x Rate						
CM-13	NA	148±10.5a	NA	NA	NA	17.4±0.5a
SM-13	NA	144±2.7a	NA	NA	NA	17.2±0.5a
CM-39	NA	279±24.8a	NA	NA	NA	21.2±1.5a
SM-39	NA	230±7.5b	NA	NA	NA	18.5±1.5a
CM-77	NA	388±29.7a	NA	NA	NA	21.4±1.0b
SM-77	NA	317±8.1b	NA	NA	NA	26.3±1.2a
Bedding x Rate						
ST-13	181±10.5a	153±8.2a	NA	NA	NA	NA
WD-13	212±11.4a	140±6.4a	NA	NA	NA	NA
ST-39	328±18.6a	296±21.6a	NA	NA	NA	NA
WD-39	323±16.0a	220±11.1b	NA	NA	NA	NA
ST-77	378±14.4b	385±30.0a	NA	NA	NA	NA
WD-77	498±14.6a	320±10.4b	NA	NA	NA	NA

Note: Values (concentrations) are the mean±standard error. Means within a column not sharing a lowercased italic letter differ significantly at the P ≤ 0.05 level (LSD test)

^aSM, stockpiled feedlot manure; CM, composted feedlot manure; ST, straw bedding; WD, wood-chip bedding; 13, 39, and 77 Mg ha⁻¹ application rates (dry basis)

^bNA, not applicable because no significant interaction effect

Figure 1. tab2 to graph2.wmf

Figure 2. WEOC 2016.draw