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EFFECTS OF RESIDUAL WASTE MATERIAL AS AGRICULTURAL SOIL
AMENDMENTS ON SOIL GREENHOUSE GAS FLUXES

BY
ASHLEY WAGGONER

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
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MASTER OF SCIENCE THESIS

OF

ASHLEY WAGGONER

APPROVED:

Thesis Committee:

Major Professor: José Amador

Rebecca Brown

Mark Stolt

Nasser H. Zawia

DEAN OF THE GRADUATE SCHOOL

UNIVERSITY OF RHODE ISLAND

2016

ABSTRACT

Anthropogenic causes of rising levels of greenhouse gases (GHG) such as CO₂, CH₄, and N₂O in the earth's atmosphere are increasingly of concern due to their effects on climate change. Much of the earth's carbon is retained in sinks created when atmospheric C is transformed into various forms of soil organic matter (SOM) and stored as stable, decay-resistant SOM for variable periods of time. When land is in its natural state, SOM is generally at a natural equilibrium of C inputs and outputs. Land use changes, particularly cultivation and conventional agricultural practices, have resulted in lower levels of SOM, leading to degraded, less productive soils. SOM is important to the physical, chemical and biological composition of the soil, and there has been a great deal of research addressing the impact of agricultural management practices on the retention of SOM and nutrients. One area of research is the use of organic waste materials as soil amendments to increase SOM. In addition to improving SOM, soil amendments also modify the physical, chemical, and biological properties of the soil, which affect the relative rates of production and consumption of GHG. The direct effects of the amendments are mediated by environmental factors that influence GHG flux, such as soil temperature and soil moisture. Organic wastes can be used as soil amendments either directly or after composting, providing an alternative to disposal in landfills or release into the environment as pollutants. The studies reported here examine the effects of residual waste materials (RWM) on GHG flux from agricultural soil. The amendments used included paper fiber with chicken manure (PF), dehydrated food waste (DFW), yard waste compost (YW), biosolids and yard waste compost (BIO), multisource compost (MC), and mineral fertilizer (MF). The first study measured GHG fluxes and assessed relationships between GHG fluxes and

soil properties from a field, in Kingston, RI, sown in sweet corn during the 2014 growing season. During this study, DFW and PF produced significantly higher maximum CO₂ fluxes than the control (CTL) (a field sown in buckwheat) in June. All other amended plots reached their maximum CO₂ flux in August, but none were significantly different from the CTL. Measurements for CH₄ and N₂O did not follow a temporal pattern, and were not significantly different from the CTL. No soil properties were significantly correlated with the change in GHG flux from all soils, but CO₂ and CH₄ fluxes for CTL and MC, respectively, were significantly correlated with active C and N₂O from PF with moisture content. The second study used microcosms to examine the effect of moisture and temperature on GHG flux and changes to soil properties in an amended agricultural soil. The flux values of CO₂, CH₄ and N₂O were significantly affected by interactions between temperature, moisture and amendment. Dehydrated food waste had highest CO₂ flux observed at 25°C and field capacity and consistently resulted in CO₂ flux values higher than unamended soil (CTL) at most moisture-temperature combinations, while MF and YW were always had the lowest CO₂ flux values. Net CH₄ flux was not affected by moisture differences among treatments, except at saturation and 25°C. Nitrous oxide production was most responsive to moisture, regardless of temperature and amendment type. Most amendments did not result in significantly increased GHG flux relative to CTL, and in some instances resulted in decreased flux for CH₄ and N₂O at moisture contents and temperatures likely to be encountered in the field. The exception was DFW, which produced the largest flux of CO₂ at 25°C, of N₂O at 20°C saturation, and of CH₄ at all moisture contents and temperatures. Overall, we found that the use of RWM as soil

amendments affected the magnitude, direction and timing of GHG flux and responds to differences in temperature and moisture. Generally, RWM – except for DFW – did not cause increased GHG fluxes of concern.

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I would also like to acknowledge my friends and family who offered me limitless support and encouragement and help during this whole process.

This research was funded in part by Rhode Island Agricultural Experiment Station and the University of Rhode Island International Center.

Preface

This thesis is organized in manuscript format in accordance with the University of Rhode Island Graduate School guidelines. It is divided into four sections: an introduction, two manuscripts (in preparation for submission), and a conclusion. Manuscript I is “Response of Agricultural Soil Greenhouse Gas Fluxes to Amendment with Residual Waste Materials in Sweet Corn Production” with authors A. Waggoner, R. Long, and J Amador. It is in preparation for submission to HortScience. Manuscript II is “Response of Agricultural Soil to Amendment with Residual Waste Materials: Effects of Temperature and Moisture on Greenhouse Gas Flux. It is in preparation for submission with authors A. Waggoner, R. Long, and J Amador to Journal of Environmental Quality.

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INTRODUCTION

Greenhouse gases (GHG) from anthropogenic sources, such as CO₂, CH₄, and N₂O, are increasingly concerning due to their effects on global climate change (IPCC, 2007). Release of increased levels of these GHG changes the global carbon (C) and nitrogen (N) cycles. Much of the earth's C is retained in carbon sinks, created when atmospheric C is transformed into various forms of soil organic matter (SOM) and stored for periods that range from days to millennia.

The C process begins when plants absorb atmospheric CO₂ and transform it to organic matter via photosynthesis. This carbon is transformed to SOM through the partial decomposition of plant litter and roots, or is ingested by heterotrophic organisms, which either release the C back into the atmosphere as CO₂, or metabolize the C into tissues and/or waste products. The C in these tissues and waste products is also eventually returned to the soil to decompose, where part of it is transformed into SOM. As organic carbon decomposes, the C is either released as CO₂ through microbial respiration, transported through the soil solution into aquatic environments as dissolved organic C (Cole et al., 2007), or becomes incorporated into soil aggregates where, through biogeochemical processes, it becomes stable, decay-resistant SOM. The residence time of SOM in the soil is controlled by the SOM and soils' physical and chemical composition, which determine the rate of microbial decomposition (Johnson et al., 2007; Hassan et al., 2014; Lal and Bruce, 1999; Li et al., 2013; Mondini et al., 2007).

Carbon can be sequestered in SOM when CH₄ oxidation is higher than CH₄ production in the soil (Segers, 1998; Smith et al., 2000). Methane is produced through

anaerobic respiration from methanogens using CO_2 , H_2 , and acetate created during the decomposition of organic C (Brady and Weil, 2002). This is common in environments such as wetlands and rice-paddies, or when aerobic environments are exposed to extended periods of rain and/or flooding (Segers, 1998; Smith et al., 2000). Additionally, CH_4 can be produced in anaerobic microsites found within aerobic soils. Within these mainly aerobic environments are also found methanotrophic bacteria that oxidize CH_4 , produced in the soil or diffused into the soil from the atmosphere, as an energy source. When CH_4 oxidation exceeds CH_4 production, the soil is considered to be a CH_4 sink.

The C and N cycles are tightly coupled due to the metabolic needs of organisms and plants for C and N (Brady and Weil, 2002; Sylvia et al., 2005). Nitrogen enters the soil in many forms, such as atmospheric N_2 gas that is converted to NH_4^+ through biological N fixation, organic N compounds from organic matter inputs, and mineralized forms from fertilizer created through the Haber–Bosch process. Once N is in the soil, it is susceptible to cycling between microorganisms and soils and can be released back to the atmosphere as N_2 , NO, and N_2O through immobilization, mineralization, nitrification, and denitrification. Immobilization and mineralization processes transform N between organic and inorganic forms, respectively. Nitrification converts inorganic N from NH_4^+ to NO_3^- , denitrification transforms NO_3^- to N_2 , and both can result in the production of N_2O as a byproduct of the process.

When land is in its native condition, the level of SOM is constant and C inputs and outputs are in equilibrium (Balesdent et al., 2000). Land use changes, particularly cultivation, and continued use of conventional agricultural practices, such as tillage,

fertilizer inputs and the removal of plant biomass result in lower levels of SOM, leading to degraded, less productive soils (Lal and Bruce, 1999). Tillage breaks soil aggregates, increases decomposition of recalcitrant SOM, and disrupts SOM accumulation (Balesdent et al., 2000). Removal of plant biomass causes decreased inputs of organic C, and fertilizer inputs accelerate microbial decomposition rates by removing nutrient limitations.

Soil organic matter is important for moisture and nutrient retention, soil structure and erosion control. Because of its importance to the physical, chemical and biological properties of soil, a great deal of research has revolved around the impact of soil management practices on the retention of SOM and nutrients (Balesdent et al., 2000; Six et al., 2002; Johnson et al., 2010; Loecke and Roberston, 2009). This has led to studies on the use of reduced and no-till techniques to decrease the net loss of carbon from the soil, as well as the use of organic wastes as soil amendments to increase SOM (Lal and Bruce, 1999; Johnson et al., 2007; Khorramdel et al., 2013; Lessard et al., 1997; Alvarenga et al., 2015).

When soil amendments are used to increase SOM, they also modify the physical, chemical, and biological properties of the soil. The increase in SOM causes changes to the soil structure due to its role in aggregate formation and stability (Annabi et al., 2007). The amendments are also often incorporated into the soil through tillage, which causes changes to moisture content, increasing drying, soil aeration and porosity. Amendment C:N ratio and the recalcitrance level of organic carbon affect decomposition rates, while the pH, electrical conductivity, and nutrient levels of the amendments affect chemical processes in the soil (Thangarajan et al., 2013).

Incorporation of amendments can also alter biological soil processes through shifts in microbial community composition, changes to density and changes to ecosystem functions (Johnson et al., 2007; Mondini et al., 2007; Khorramdel et al., 2013; Li et al., 2013). The changes induced by the addition of organic amendments affect the flux of GHG in soil (Johnson et al., 2007; Mondini et al., 2007; Li et al., 2013; Qiu et al., 2013).

Environmental factors also influence GHG flux. Soil temperature and soil moisture content alter the effects of soil amendments through effects on soil biogeochemical processes (Johnson et al., 2007; Li et al., 2013; Thangarajan et al., 2013; Hassan et al., 2014). Soil moisture content controls aeration because soil water directly competes with air for pore space, and acts as a barrier for diffusion of gases in the soil. Low soil moisture content provides more opportunity for gaseous exchanges between the soil and the atmosphere due to more open pore space. This allows for diffusion of O₂ into the soil for use in respiration by microorganisms and plant roots, and the release of CO₂ and N₂O from the soil (Brady and Weil, 2002). Conversely, high soil moisture content restricts the diffusion of O₂ into the soil, slowing the rate of aerobic microbial activity, and supports microbial production of precursors for CH₄ production and the establishment of anoxic microsites needed for denitrification and N₂O production (Linn and Doran, 1984; Conrad, 1996; Zhu et al., 2015). Soil moisture also affects soil temperature by altering the amount of energy needed to warm the soil.

Temperature also affects the rate of biological and chemical processes in the soil. At low temperatures (below 10°C), microbial processes slow down, reducing the rates of SOM decomposition and microbial respiration. Increasing temperature has been

associated with increasing rates of microbial processes, thus influencing GHG fluxes (Allison, et al., 2010). Soil moisture content and temperature work synergistically to affect soil functions, particularly the rate of decomposition of organic matter, the cycling of C and N through the soil, and GHG flux.

In the United States in 2012, over 86 million tons of municipal solid waste, of which the largest component were organic wastes (64.5 million tons), such as paper and paperboard, yard trimmings and food waste, were recovered through recycling and composting (EPA, 2012). Using these and other residual materials from industrial and waste disposal processes as agricultural soil amendments provides an alternative to disposal in landfills or release into the environment as pollutants. To date, research into residual waste materials (RWM), such as paper fiber, dehydrated food waste, and multisource composts, has focused on their effects on soil quality and crop production (e.g. Long et al., 2016). Because use of these products will also affect the soil biogeochemical processes, and thus GHG production and consumption, there is also a need to investigate effects on GHG flux (Thangarajan et al., 2013; Alvarenga 2015). Measuring GHG fluxes from agricultural soils amended with RWM will help us understand how these amendments influence rates of production and consumption of these gases, as well as their impact on C and N cycling in soil (Thangarajan et al., 2013).

My thesis is composed of two manuscripts that examine the effects of RWM on GHG flux from agricultural soil. The first, “Response of Agricultural Soil Greenhouse Gas Fluxes to Amendment with Residual Waste Materials in Sweet Corn Production,” is a field study that measured GHG fluxes and assessed relationships between GHG

fluxes and soil properties. The second, “Response of Agricultural Soil to Amendment with Residual Waste Materials: Effects of Temperature and Moisture on Greenhouse Gas Flux,” examines the effect of moisture and temperature on GHG flux and changes to soil properties in an amended agricultural soil using microcosms.

**MANUSCRIPT – I: RESPONSE OF AGRICULTURAL SOIL GREENHOUSE
GAS FLUXES TO AMENDMENT WITH RESIDUAL WASTE MATERIALS
IN SWEET CORN PRODUCTION**

In preparation for submission to HortScience

Ashley Waggoner, Rebecca Long and Jose A. Amador

Laboratory of Soil Ecology and Microbiology, University of Rhode Island, Kingston, RI 02881

Abstract

In the United States, 64.5 million tons of organic materials – consisting of paper and paperboard, yard trimmings, and food wastes – were recovered through recycling and composting in 2012. The use of these and other residual waste materials (RWM) as agricultural soil amendments may offer a solution to the problem of reduced soil organic matter in agricultural soil. RWM can affect soil physical, chemical and biological soil properties, and add C, N and other nutrients to the soil. These changes can affect soil biogeochemical processes and may alter the flux of greenhouse gases (GHG), such as CO₂, CH₄, and N₂O. We quantified the magnitude of GHG flux from soil plots amended with five different RWMs, paper fiber with chicken manure (PF), dehydrated food waste (DFW), yard waste compost (YW), biosolids and yard waste compost (BIO), multisource compost (MC), or a mineral fertilizer (MF) used to grow corn over the course of a growing season. We used plots planted to buckwheat to simulate a fallow field as a control (CTL). We also examined correlations between soil properties and GHG fluxes. Amending the soil with RWMs changed the magnitude and timing of CO₂ flux. Dehydrated food waste, PF, and CTL reached maximum CO₂ flux in June, and DFW and PF had significantly higher CO₂ flux than CTL. In contrast, BIO, MC, MF, and YW did not reach maximum CO₂ flux until August, with values that were not significantly different from CTL. Effects of RWM and MF on CH₄ and N₂O fluxes were variable, and fluctuated between net consumption and net production for most amendments. Carbon dioxide for CTL and CH₄ for MC fluxes were significantly correlated with active C. Nitrous oxide for PF was significantly correlated with moisture content. GHG fluxes in plots amended with

RWM generally were not different from control plots over the course of a growing season. Our results suggest that, with few exceptions, use of these RWMs as soil amendments in sweet corn production may not cause substantial increases in GHG fluxes from soil.

Introduction

There is an increasing need to reduce greenhouse gas (GHG) emissions from anthropogenic sources, including agriculture, which is responsible for 14% of anthropogenic GHG emissions (IPCC, 2014). Agricultural soils are one of the largest anthropogenic sources of N₂O emissions (Ball et al., 2014; Duxbury, 1994). They are also sources of CO₂, and can be sources or sinks of CH₄ depending on management practices. While atmospheric levels of N₂O and CH₄ are much lower than CO₂, increases in the concentration of N₂O and CH₄ are of particular concern because their global warming potential is 298 and 25 times higher, respectively, than CO₂ (IPCC, 2007).

Land use changes have led to increased release of soil-sequestered carbon (C) (soil organic matter (SOM)) through increased decomposition and associated GHG emissions (IPCC, 2014; Lal and Bruce, 1999). In addition to serving as a sink for atmospheric C, SOM plays an essential role in soil processes, including moisture retention, nutrient supply, and maintaining soil structure. Conventional agricultural practices such as tillage and removal of plant biomass during harvest lower SOM, a problem that may be remedied by amending the soil with organic materials. In the United States, organic materials make up the largest component of municipal solid wastes, of which 64.5 million tons was recovered in 2012 through recycling and composting of paper and paperboard (51%), yard trimmings (22%) and food wastes (2%) (EPA, 2012). Using these and other residual materials from industrial and waste disposal processes as agricultural soil amendments provides an alternative to disposal in landfills or release into the environment as pollutants (Long et al., 2016).

Novel residual waste materials (RWM), such as paper fiber, gelatin waste, and dehydrated food waste, have been investigated for use as agricultural soil amendments (Long et al., 2016). They can add C, N and other nutrients to the soil, and can affect physical, chemical and biological soil properties. Changes in available C and nutrients and in soil properties alter biogeochemical processes in the soil, and can affect the flux of C and N-containing greenhouse gases (GHG), including CO₂, CH₄, and N₂O (Thangarajan et al., 2013). Variability in the composition of RWMs affects the magnitude of changes to nutrient availability, organic matter, and pH in soil (Long et al., 2016; Mondini et al., 2007; Mukherjee et al., 2014). Thus, differences in RWM composition and effects on soil properties may also affect the magnitude and direction of GHG flux from RWM-amended soils. Research on the use of RWM in agriculture has mainly focused on their impact on soil properties and crop production, while their effects on the flux of GHGs from agricultural soils have received little attention (Johnson et al., 2007).

In a companion study, Long et al. (2016) examined the use of novel RWMs as soil amendments and their effects on soil properties and crop production. Soil amendment with yardwaste compost and biosolids/yardwaste co-compost resulted in significantly higher levels of SOM. Positive effects on soil properties such as increases in soil pH, and availability of inorganic N were observed for other RWMs. These changes can impact C and N cycling as well as the flux of GHG from soil. To examine the effects of novel RWM amendments on GHG flux, we measured net production of CO₂, CH₄, and N₂O in plots used to grow sweet corn during the 2014 growing season of the Long et al. (2016) study. Our objectives were to (i) quantify effects of RWMs on the

magnitude and direction of GHG flux from amended soil plots over the course of a growing season and (ii) examine correlations between soil properties and GHG flux.

Materials and Methods

Study design

Our measurements were carried out in the context of a study conducted to examine the effects of novel RWMs as soil amendments on soil properties and crop production during the 2013 and 2014 growing seasons at the University of Rhode Island's Greene H. Gardner Crop Science Field laboratory in Kingston, RI (Long et al., 2016). We measured GHG flux from amended soil planted with sweet corn (*Zea mays* cvs. Applause and Montauk) during the 2014 growing season.

The experiment employed a randomized block design of seven 4.6 m × 4.6 m plots with four replications. Only six of the plots of each replication were used in our study. We established an additional four 4.6 m × 4.6 m plots in which we grew and maintained a summer cover crop of buckwheat (*Fagopyrum esculentum*) to simulate a fallow agricultural field. These plots served as a control (CTL) (Fig. 1.1). In July 2014, the buckwheat was mowed and tilled into the soil, and re-sown for the remainder of the growing season. Prior to the start of GHG flux measurements in May of 2014, the plots were amended with paper fiber with chicken manure (PF), dehydrated food waste (DFW), yard waste compost (YW), biosolids and yard waste co-compost (BIO), or multisource compost (MC) at a rate of 10 Mg C/ha over a two-year period, or mineral fertilizer (MF) at a rate of 112 kg N/ha/yr. Additional information on management practices can be found in Long et al. (2016).

GHG flux measurements

The flux of CO₂, CH₄, and N₂O was measured monthly in 2014. Samples were collected on June 2/3, June 30/July 1, August 4/5, August 30/31, and October 13, between 9 am and 12 pm, using the static chamber method (Flessa et al., 1995). On each sampling date, a cylindrical plastic chamber (27-cm dia., 20-cm high), fitted at the top with a rubber septum, was placed at random within the center interrow of each plot, and pushed ~2.5 cm into the soil. Gas samples from the chamber were obtained immediately after placement and at 15 and 30 min after placement using a 20-mL gas-tight syringe. The syringe was pumped repeatedly to mix the gases in the headspace prior to sample collection. Gas samples were transferred to 20-mL evacuated glass vials, and analyzed using a Shimadzu GC-2014 Greenhouse Gas Analyzer (Shimadzu Corp., Kyoto, Japan) fitted with a flame ionization detector for CH₄ and CO₂ (post-methanizer) and an electron capture detector for N₂O.

The best-fit linear regression was applied to the concentrations from the three collected samples for each plot. The mass of each GHG within the chamber, n (mol), was calculated using the Ideal Gas Law, $n=RT/PV$, where n =mol GHG per mol air, P =atmospheric pressure (atm), T =chamber air temperature (K), R =universal gas constant (0.0821 L atm/mol K), and V =volume (L). The rate of GHG production per unit area was calculated using the slope of the best-fit line, volume of air in the chamber and cross-sectional area.

Soil properties

Soil samples were collected monthly, during the same time period as GHG flux sampling, and analyzed for moisture content, organic matter, pH, electrical

conductivity (EC), nitrate (NO_3^-), and ammonium (NH_4^+) as described in Long et al (2016). Additionally, we analyzed the soil samples for active C using the potassium permanganate method of Weil et al. (2003). Active C was determined colorimetrically by measuring absorbance at 550 nm with a Biotek Powerwave 340 Microplate Scanning Spectrometer (Biotek Instruments, Inc., Winooski, VT). Pre-amendment soil properties are shown in Table 1.1.

Data on soil temperature and soil moisture content were collected from a U.S. Climate Reference Network (USCRN) station less than 500 m from the experimental plots (Fig. 1.2).

Residual waste materials

Six soil amendment treatments were applied by hand and incorporated by disc harrow in late May 2014 (Long et al., 2016). The amendments were applied in the condition they were delivered. All amendments (except MF) were applied at a rate sufficient to supply 10 Mg organic C/ha over the 2013 and 2014 growing seasons. The MF was applied to provide 112 kg N/ha/yr. Amendments were:

1. Biosolids/yardwaste co-compost (BIO), consisting of ground leaves, brush and municipal biosolids composted by in-vessel horizontal agitated bin.
2. Dehydrated food waste (DFW) from a New York restaurant. It was ground, dehydrated, and aerobically incubated for 18 h to reduce bulk.
3. Multisource compost (MC) made from leaf and wood chips as bulking agents, mixed with farm and zoo animal manure and bedding, fish scraps, seashells, seaweed, coffee grinds, wood ash and food scraps and composted in Rhode Island using active windrows.

4. Mineral fertilizer (MF) (20N-8.7P-16.6K) with 3.5% ammonium-N, 5.5% nitrate-N and 11% urea N.
5. Paper fiber/chicken manure blend (7:1) (PF) using paper fiber from dewatered primary sludge from recycled paper processing. To reduce its high C:N ratio (>57:1), the paper fiber was blended with a composted chicken manure product at a rate of 7 parts paper to 1 part chicken manure.
6. Yard waste compost (YW) produced in Rhode Island using leaves composted in active windrows.

Amendment properties from the study site are shown in Table 1.1.

Statistical analyses

A two-way analysis of variance followed by the Holm-Sidak test was used to identify statistically significant differences at $p < 0.05$ for GHG flux among months for an amendment, and among amendments within a month. A Pearson product moment correlation coefficient (R) was used to evaluate the relationship between GHG flux and soil properties. Statistical analyses were performed using SigmaPlot 11.0 (Systat Software Inc., 2008).

Results

Carbon dioxide

A significant interaction ($p = <0.001$) was found between the amendments and months. Mean CO₂ fluxes from DFW and PF were significantly higher ($p = <0.001$) than CTL. Significant differences were also found in mean CO₂ flux between all months (except July and August, and July and September) in the order June > August > July > September > October.

The timing and magnitude of maximum CO₂ flux among the amendments followed a general trend (Fig. 1.3). The CTL, DFW, and PF treatments reached maximum CO₂ flux in June (with DFW and PF over three times greater than CTL), remaining relatively constant (CTL) or declining steadily (DFW and PF) for the remainder of the season. In contrast, CO₂ flux in plots amended with MF, MC, BIO, and YW increased monthly until reaching a maximum in August, and declined for the remainder of the season. Maximum CO₂ flux values over the growing season followed the order: DFW > PF > YW > BIO > MC > MF > CTL.

Dehydrated food waste and PF also had the two highest fluxes observed during a single month during the study, and were the only amendments with a CO₂ flux significantly higher than CTL in June (Table 1.2). Between July and September, most amendments had a CO₂ flux higher than CTL, but no significant differences from CTL were observed. By the end of the growing season in October, most treatments had lower flux values than CTL with no significant differences.

We found a significant correlation between CO₂ flux and soil active carbon for the CTL treatment ($R = 0.94$) (Table 1.3). No other significant correlations were found between CO₂ flux and soil properties.

Methane

Amendment with MF and PF resulted in net CH₄ consumption (negative flux) throughout the growing season (Fig. 1.3). All other amendments and the CTL had periods of net production (positive flux) and net consumption (Fig. 1.3). Net CH₄ production was observed for CTL and DFW in June; for DFW and YW in July; for MC in August and September; and for BIO and YW in October. There was no clear temporal pattern in the magnitude or direction of methane flux for any of the treatments.

Plots amended with MC had the highest net CH₄ production over the growing season in September, with flux values decreasing in the order: MC > YW > PF > MF > BIO > CTL (DFW had no net CH₄ flux during this sampling period). Amendment of plots with MF resulted in the highest net consumption over the growing season in October, with values following the order: MF > PF > DFW > MC > CTL > YW > BIO. There were no significant differences in CH₄ flux between amended plots and the CTL, or between months for any of the treatments. There was also no significant interaction between amendment and month (Table 1.2).

There were significant correlations between CH₄ flux and soil properties that varied by amendment. Methane flux was positively correlated with EC for the CTL treatment ($R = 0.94$), with temperature for DFW, and with soil active C in MC (Table 1.3).

Nitrous oxide

The highest positive N₂O flux (June) and the highest negative N₂O flux (October) were observed for the CTL treatment (Fig. 1.3). Net N₂O production was observed for nearly all amendments in June, with values ranging between 0.016 and 0.768 mg N₂O-N/m²/d, following the order: CTL > DFW > BIO > PF > MC > MF > YW. The exception was YW, which had a net consumption. Flux values in other months fluctuated between net consumption and net production for all treatments, with no clear temporal pattern. No significant interaction was found between amendments and months. No significant differences from the CTL were observed.

There was a significant negative correlation between N₂O flux and pH for BIO, and a positive correlation between N₂O flux and moisture content for PF (Table 1.3).

Discussion

Carbon dioxide

Studies of CO₂ flux from corn cultivation in Minnesota reported a 3-year average between 1,019 and 1,033 mg/m²/d (Johnson et al., 2010). A study on the effects of soil amendments in Ohio found CO₂ fluxes of 1,000 to 10,000 mg/m²/d (Mukherjee et al., 2014). Possinger and Amador (2015) reported flux values of 800 to 3100 mg CO₂-C/m²/d in a study at a nearby location growing sweet corn. The CO₂ flux values we observed were lower than those reported in these studies.

Differences in the physical and chemical properties of the amendments may explain the significantly higher CO₂ flux for DFW and PF relative to CTL in June. Moisture content (especially in the PF treatment) and levels of easily decomposable substrates in non-composted amendments (such as DFW and PF), can lead to increased microbial biomass and CO₂ flux in soil (Mondini et al., 2007, Li et al., 2013; Mukherjee et al., 2014; Zhu et al., 2015). Paterson et al. (2011) suggested that increased CO₂ flux could be from microorganisms present, primarily in composted amendments. In contrast, Saison et al. (2006) found increased CO₂ flux to be the result of increased substrate availability for the microbial community already present in soil, with the addition of amendments inducing a response from that community. Additionally, composting of amendments could explain the absence of significant differences in CO₂ flux between the composted amendments (BIO, MC, YW) and the CTL treatment. Composting results in the decomposition of labile C, resulting in an amendment enriched with more recalcitrant C, which reduces the response of CO₂ flux when these amendments are added to soil.

The occurrence of CO₂ flux maxima in August for BIO, MC, MF, and YW-amended plots can be explained by warmer temperatures at the end of the summer or plant growth processes (Fig. 1.2). The effects of temperature on soil CO₂ flux have been studied extensively, with warmer temperatures leading to increased CO₂ flux (Allison et al., 2010; Navaries et al., 2015; Schaufler et al., 2010; Smith et al., 2003). In a study of organic amendments, temperature and moisture effects on GHG, Li et al. (2013) found that rising temperature resulted in increased CO₂ flux. Since our measurement of CO₂ flux was for total soil respiration, which included CO₂ from microorganisms and plant roots, it is also possible our increased flux in August was due to increased CO₂ flux from the rhizosphere due to the plant growth processes (Raich, 2005). In contrast, we would not see such an increase in the CO₂ flux from DFW and PF plots due to the already increased flux caused by the addition of the easily decomposed C in these non-composted materials being consumed shortly after application as indicated by CO₂ flux maxima in June. This would leave the more recalcitrant C in the soil, reducing the response of flux to increased temperature and covering up the increased flux from plant roots in August. Additionally, we would not see this pattern of flux in the control, due to the different growth patterns of buckwheat to corn.

Application of amendments leads to changes in edaphic properties, including pH, electrical conductivity, and availability of inorganic N (Long et al., 2016), which affect soil microorganisms, C and N cycling, and thus would be expected to affect the flux of GHGs (Thangarajan et al., 2013; Alvarenga et al., 2015; Annabi et al., 2007). Although changes in soil properties were observed in response to RWM amendments

in the companion study by Long et al. (2016), soil properties were generally not correlated with CO₂ flux (Table 1.3). This was surprising, given the well-established relationship between CO₂ flux and temperature and moisture content (Linn and Doran, 1984; Smith et al., 2003; Li et al., 2013; Davidson et al., 1998). It is possible that temperature and moisture content did not vary over a sufficiently large range (Fig. 1.2) for correlations to be significant.

The significant positive correlation between CO₂ and soil active C in CTL could be explained by differences in sampling. Root structures can affect the chemical, physical and biological properties of the soil and have differing levels of CO₂ respiration, leading to altered GHG flux from different systems (Larionova et al., 2006; Skiba et al., 1998). The amended samples were taken from between the rows of corn, where some roots were included but the shoots were excluded, while in CTL the chambers were placed over a portion of shoots and roots, causing CO₂ values to be affected by root respiration and plant CO₂ uptake.

Methane

Our results generally showed net CH₄ consumption for all treatments, with no significant differences relative to CTL or between months. Instances in which net production was observed never had a flux greater than 0.2 mg CH₄-C/m²/d (Fig. 1.3). The CH₄ flux values from our CTL and amended plots are within the range of -18.6 and 28.7 mg CH₄-C/m²/d reported for studies of organic and mineral fertilizer-amended agricultural soils (Lessard et al, 1997; Mapanda et al., 2011). Avizinis (2012) reported fluxes between -3.6 and 0.2 mg CH₄-C/m²/d from undisturbed soils at a similar site, within the range of those found in our study.

We found that the magnitude and direction of methane flux was highly variable, regardless of amendment type. Methane production (methanogenesis) and oxidation are microbial soil processes, both of which take place in agricultural soils (Duxbury, 1994; Segers, 1998; Johnson et al., 2007). Agricultural soils can be sinks, sources or neutral for CH₄ flux depending on the growing season, soil treatments, moisture content, and N levels (Duxbury, 1994; Johnson et al., 2007; Mapanda et al., 2011). The magnitude and direction of methane flux values show a high degree of spatial and temporal variability (Conrad, 1996; Lessard et al., 1997; Smith et al., 2000). The variable nature of CH₄ flux is related to the conditions and substrates required for methanogenesis and CH₄ oxidation. Net CH₄ production is a result of higher methanogenesis than CH₄ oxidation. Methanogenic archaea require anoxic conditions (found in microsites in aerobic soils) as well as substrates (acetate, CO₂, and H₂) that are produced by fermentation. Even when conditions necessary for methanogenesis are met, methanotrophic bacteria present in agricultural soils can oxidize CH₄ produced in the soil and CH₄ present in the atmosphere, resulting in net CH₄ consumption if the rate of consumption offsets CH₄ production.

We found significant positive correlations between CH₄ flux and EC for CTL, CH₄ flux and temperature for DFW, and CH₄ flux and active C for MC (Table 1.3). The correlation between CH₄ flux and EC for the CTL could result from the buckwheat in the CTL plots. When buckwheat is growing at its fastest, N is quick to mineralize, which would have increased the amount of NH₄⁺ present, creating competition with CH₄ for methane monooxygenase and resulting in lower net consumption of CH₄ (Bjorkman, 2009). Increased salinity can also result in reduced

nitrification (Adviento-Borbe et al., 2006), which would also lead to increased levels of NH_4^+ . This may have taken place in the CTL treatment, but not the amended soils, because the amended plots were growing corn, which affects the levels of NH_4^+ and changes in EC differently than buckwheat.

A positive correlation between CH_4 flux and temperature has been shown previously, with rising temperatures leading to increased aerobic microbial activity (Conrad, 1996). This reduces O_2 availability, creating anoxic microsites and promoting fermentation processes, which produce substrates for methanogenesis. The correlation between active C and CH_4 flux may represent a similar mechanism to that involving increased temperature, with higher levels of labile C causing increased microbial oxygen consumption (Garcia-Marco et al., 2014). In addition, these relationships may involve reduced CH_4 oxidation from increased levels of NH_4^+ in soil, which inhibits methane oxidation (Conrad, 1996; Thangarajan et al., 2013).

The absence of significant correlation between CH_4 flux and most soil properties is likely explained by the variable nature of CH_4 flux from agricultural soils, which is driven by the complex series of conditions needed for methanogenesis.

Methanogenesis generally takes place in anoxic microsites in agricultural soils, which are established under certain moisture regimes. Additionally, the presence of NH_4^+ can interfere with CH_4 oxidation, introducing another confounding variable into the relationship between CH_4 flux and soil properties.

Nitrous oxide

Nitrous oxide flux in our study ranged between -0.7 and 0.6 $\text{mg N}_2\text{O-N/m}^2/\text{d}$ – except for CTL in June (0.8 $\text{mg N}_2\text{O-N/m}^2/\text{d}$). This is generally lower than reported by

others. For example, N₂O fluxes as high as 0.7 mg N₂O-N/m²/d (Skiba et al, 1998; Odlare et al., 2012) and 0.8 mg N₂O-N/m²/d (Mapanda et al., 2011) have been reported for agricultural and semi-natural soils in Scotland, agricultural soil amended with two types of biogas residues in a lab experiment, and soil amended with composted manure or mineral fertilizer growing corn in Zimbabwe, respectively. Values of N₂O flux from corn plots in conditions similar to ours ranged between -0.04 to 10.84 mg N₂O-N/m²/d (Molodovskaya et al., 2010) and 5.1 to 19.9 mg N₂O-N/m²/d (Amador and Avizini, 2013).

Like CH₄, N₂O flux shows high spatial and temporal variability that are dependent on soil conditions (Cayula et al., 2010; Flessa et al., 1995; Johnson et al., 2007; Loecke and Robertson, 2009). Nitrous oxide is produced primarily via nitrification and denitrification, largely controlled by O₂ diffusion in soil, and subject to the influence of many factors, including soil properties and the physical and chemical properties of amendments (Garcia-Marco et al., 2014; Mukherjee et al., 2014).

Differing N₂O fluxes could be due to differing amounts and types of N introduced by the amendments (Table 1.1). Since the organic amendments were added based on total C inputs to soil, the amount and type of N added by each amendment were different. Higher N₂O fluxes could also be the result of the physical and chemical properties of the amendments, which affect O₂ diffusion and rates of nitrification and denitrification (Zhu et al., 2015). Denitrification is a heterotrophic process in which C and N cycling are coupled. The addition of organic C and its composition (labile or recalcitrant) could affect N₂O flux by providing organic C for denitrification and/or

increasing microbial respiration, which would lead to lower O₂ availability, promoting denitrification (Garcia-Marco et al., 2014; Mukherjee et al., 2014).

Because NH₄⁺ and NO₃⁻ are substrates, respectively, for nitrification and denitrification, we expected to find a correlation between soil inorganic N and N₂O flux, but no significant correlations were found (Table 1.3). Others have also reported on the absence of a relationship between inorganic N and N₂O flux. For example, Adviento-Borbe et al. (2006) found that N₂O flux was more likely related to soil N turnover than the size of the inorganic N pool. However, we observed a significant, positive correlation between moisture content and N₂O flux for PF. Nitrous oxide can result from incomplete denitrification due to the presence of O₂ (Sylvia et al., 2005). While increased soil moisture content results in decreased O₂ diffusion and anoxic microsites where denitrification can take place, some O₂ is still present in the soil. Since PF was 1.02 g moisture/g amendment when it was added to the plots, incorporating the amendment into the soil may have created conditions leading to incomplete denitrification.

There was also a significant negative correlation between pH and N₂O flux from BIO. Decreases in soil pH can be caused by nitrification, when H⁺ are released as NH₄⁺ is oxidized to NO₃⁻, which reduces the pH of the soil. Additionally, decreased soil pH and the ratio of N₂O:N₂ produced from denitrification are correlated, with a higher ratio of N₂O:N₂ resulting from acidic conditions (Amos et al., 2005; Thangarajan et al., 2013; McMillan et al., 2016). This correlation could be the result of nitrification causing decreased soil pH resulting in higher N₂O:N₂ ratio in the BIO amended plots.

Conclusions

The use of RWM as soil amendments affected the magnitude, direction and timing of GHG fluxes from an agricultural field planted with corn. Maximum CO₂ flux for the amendments that were not composted (CTL, DFW and PF) – with the highest amount of readily degradable C – was highest in June, whereas amendments that were composted – and therefore had the lowest amount of readily degradable C – did not reach maximum CO₂ flux until August. Net flux of CH₄ and N₂O fluctuated between production and consumption, with no observable temporal pattern for any of the treatments tested. The GHG fluxes in our study were within the range of those reported by others in the same or similar soils and conditions, suggesting that, with few exceptions, use of these RWMs as soil amendments may not cause substantial increases in GHG fluxes. In comparison to keeping a field sown with a cover crop of buckwheat instead of in sweet corn production, there were also few significant differences.

The overall lack of significant correlation between GHG flux and soil properties, especially for CH₄ and N₂O flux, is likely due to complex events involved in production/consumption of these gases and the narrow range of soil properties, like moisture content and temperature. The impact of uncontrolled variables common to field studies, such as precipitation, temperature and unaccounted nutrient additions, could also contribute to the lack of correlation, but even with these variables, the GHG fluxes we observed were not concerning. We suggest using a microcosm-scale experiment to further evaluate the effects of environmental variables on the flux of GHG in soils amended with RWM.

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Figures and Tables

Table 1.1. Characteristics of pre-amended soil and of amendments applied in 2014. Values are means (n=3-4). After Long et al. (2016).

Amendment	NH ₄ ⁺ mg/kg	NO ₃ ⁻ mg/kg	C %	N %	C:N	Water Cont. %	Org. Matter %	EC mS	pH	Active C g C/kg
Pre-amendment soil [§]	1.4	2.7	-	-	-	18	6	-	-	0.47
Biosolids/yardwaste co-compost	47.6	4009	33	3	10.3	45	54	7.0	7.9	55.98
Dehydrated food waste	36.1	205	49	3	14.4	3	63	10.1	5.5	98.70
Multisource compost	431	392	15	2	9.2	40	19	5.3	7.1	49.73
Mineral fertilizer	-	-	-	-	-	0	0	345	4.6	50.26
Paper fiber/chicken manure (7:1 ratio)	331	1226	40	1	33.9	102	79	9.5	6.4/7.0*	62.03*
Yardwaste compost	201	2.23	20	2	13.0	78	34	1.8	6.7	52.82

[§] Mean value based on samples of all plots used to grow sweet corn pre-amendment (n = 28).

* Paper fiber and chicken manure were measured separately

Table 1.2. Significant differences ($p < 0.05$) for CO₂ flux over the growing season for each amendment[§] and among amendments within a month relative to control*. Methane and N₂O flux values are not shown because no significant differences were observed.

Gas	Month	CTL	BIO	DFW	MC	MF	PF	YW
CO ₂	June	A	AB	A*	A	A	A*	A
	July	A	AB	B	A	A	B	AB
	Aug	A	A	B	A	A	B	B
	Sept	A	A	BC	A	A	B	AB
	Oct	A	B	C	A	A	B	A

[§] Same letter indicates no significant difference for each amendment over the growing season

* Indicates significant difference among amendments within a month relative to control

Table 1.3. Pearson product moment correlation coefficients of relationships between GHG flux and soil properties. Bold values indicate significant correlation ($p < 0.05$).

Gas flux	Variable	Amendment							
		All	CTL	BIO	DFW	MC	MF	PF	YW
CO ₂	Soil active C	-0.18	0.94	0.40	-0.82	-0.29	0.38	-0.77	0.29
	Temperature	0.49	0.66	0.58	0.87	0.72	0.46	0.81	0.15
	Electrical conductivity	0.17	0.82	0.28	0.83	0.67	0.00	0.79	0.09
	Inorganic N	0.03	0.53	0.33	-0.07	0.68	0.02	0.63	0.10
	pH	-0.20	0.29	-0.01	-0.15	-0.17	-0.03	-0.30	0.34
	Moisture content	0.14	0.51	-0.12	-0.07	0.40	0.37	-0.11	0.21
CH ₄	Soil active C	0.12	0.87	0.01	-0.63	0.92	0.43	0.47	0.49
	Temperature	0.03	0.31	-0.84	0.90	-0.38	0.51	0.31	-0.26
	Electrical conductivity	-0.15	0.94	-0.78	0.80	-0.44	0.05	0.14	-0.05
	Inorganic N	-0.18	0.77	-0.83	0.07	-0.48	0.14	0.16	0.10
	pH	0.14	0.30	0.60	-0.23	0.75	-0.39	0.16	-0.74
	Moisture content	-0.08	0.72	0.20	-0.20	-0.12	-0.14	0.01	-0.31
N ₂ O	Soil active C	-0.04	0.36	0.42	-0.68	0.68	-0.03	-0.61	0.11
	Temperature	0.15	0.50	0.31	0.60	-0.01	-0.82	0.12	-0.21
	Electrical conductivity	0.21	0.58	0.44	0.64	0.00	-0.52	0.51	0.10
	Inorganic N	0.01	0.49	0.36	0.17	-0.12	-0.61	0.48	0.26
	pH	-0.04	-0.04	-0.89	-0.45	0.03	0.80	0.24	-0.16
	Moisture content	0.01	-0.14	-0.85	0.09	-0.86	0.24	0.92	0.45

* Air temperature within the flux chamber.

	CTL				CTL			CTL				CTL	
BUFFER STRIP													
DFW	MC	N/A	YW	BIO	PF	MF	YW	BIO	MF	MC	N/A	PF	DFW
BIO	YW	PF	DFW	MC	MF	N/A	DFW	N/A	BIO	PF	YW	MF	MC

Figure 1.1. Diagram of treatment layout. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost; N/A = not applicable to this study. Not to scale.

Figure 1.2. Daily average soil moisture and soil temperature at 10 cm during the 2014 growing season as reported by the U.S. Climate Reference Network station at the Green H. Gardiner Crop Science Field Laboratory in Kingston, RI. Vertical dashed lines indicate dates when flux measurements and soil sampling took place. <http://www.ncdc.noaa.gov/crn/>

Figure 1.3. Mean ($n=4$) flux of CO₂, CH₄ and N₂O in control and amended plots during the 2014 growing season. Bars represent one standard deviation of the mean. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost.

**MANUSCRIPT – II: RESPONSE OF AGRICULTURAL SOIL TO
AMENDMENT WITH RESIDUAL WASTE MATERIALS: EFFECTS OF
TEMPERATURE AND MOISTURE ON GREENHOUSE GAS FLUX**

In preparation for submission to Journal of Environmental Quality

Ashley Waggoner, Rebecca Long and Jose A. Amador

Laboratory of Soil Ecology and Microbiology, University of Rhode Island, Kingston, RI 02881

Abstract

The conversion of forests to agricultural lands, followed by long-term agricultural use can lead to lower levels of soil organic matter (SOM). The loss of SOM in agricultural soils can be counteracted by amendment with residual waste materials (RWM) like biosolids and composts. These materials add C and N to the soil, affecting physical, chemical, and biological properties and processes, including production of C and N-containing greenhouse gases (GHGs): CO₂, CH₄ and N₂O. This, coupled with spatial and temporal variations in temperature and moisture, control the flux of C and N-containing GHGs from soil. We used microcosms to investigate the magnitude and direction of GHG flux as a function of soil temperature and moisture in an agricultural soil amended with RWM. Soil was amended with 10 Mg total C ha⁻¹ in the form of paper fiber with chicken manure (PF), dehydrated food waste (DFW), yard waste compost (YW), biosolids and yard waste compost (BIO), multisource compost (MC), or 112 kg N ha⁻¹ of mineral fertilizer (MF). Un-amended soil was used as a control (CTL). Soil moisture was adjusted to permanent wilting point, field capacity or saturation. Microcosms were incubated at 10, 15, 20, or 25°C and gas concentrations determined over 14 days. The highest CO₂ flux was observed at 25°C and field capacity, with CO₂ production following the order DFW>MC>CTL>PF>MF>BIO>YW. When compared to the CTL, CO₂ flux from DFW was higher at most moisture-temperature combinations, while MF and YW were always lowest. CO₂ flux was not correlated with levels of active C in the amendments. Net CH₄ flux was not affected by moisture differences among treatments, but varied in response to temperature and amendment. Nitrous oxide production was most

responsive to moisture, regardless of temperature and amendment type. Most amendments did not result in increased GHG flux relative to CTL, and in some instances resulted in decreased flux for CH₄ and N₂O at moisture contents and temperatures likely to be encountered in the field. The exception was DFW, which produced the largest flux of CO₂ at 20°C and field capacity, of N₂O at 20°C and saturation, and the second largest flux of CH₄ at 20°C and saturation. Relative to the CTL, most amendments did not increase global warming potential from GHG emissions, except under saturated conditions, where increased N₂O production was realized. In general, RWM – except for DFW – do not cause increased GHG fluxes of concern.

Introduction

Soil organic matter (SOM) is essential for microbial soil processes involved in production and consumption of C and N-containing greenhouse gases (GHGs), including CO₂, CH₄ and N₂O (Sylvia et al., 2005; Smith et al., 2003). The conversion of forests and grasslands to agriculture and long-term agricultural practices, such as tillage, has led to lower levels of SOM in agricultural soils (Balesdent et al., 2000). The growing need to lower the amount of organic waste materials deposited into landfills and released into the environment as pollutants has led to the evaluation of alternative uses for residual waste materials (RWM), such as biosolids, biochar, composts, and other residuals from industrial and waste disposal processes (Long et al., 2016; Cayuela et al., 2010; Lal and Bruce, 1999; Li et al., 2013; Mondini et al., 2007; Thangarajan et al., 2013; Zhu and Bruun, 2014). One alternative is the use of organic RWM as soil amendments to counteract the loss of SOM and/or as a source of nutrients in agricultural soils (Long et al., 2016; Huang et al., 2004; Johnson et al., 2007; Li et al., 2013; Thangarajan et al., 2013, Zhu and Bruun, 2014).

Amendment of soil with RWM adds C and N-containing substrates, which directly and indirectly affect physical, chemical, and biological properties and processes, and ultimately GHG flux. In a review, Thangarajan et al. (2013) explain how the use of organic amendments in soil leads to alteration of soil properties, and subsequently methanogenesis, nitrification, denitrification, and priming effects. Additionally, organic amendments can affect GHG flux through the direct emission of GHG from the amendments, and indirectly by affecting the soil biogeochemical processes that create these GHG.

Most biogeochemical processes in soil are strongly impacted by fluctuations in temperature and moisture (Allison et al., 2010; Conant et al., 2011; Singh and Gupta, 1977; Linn and Doran, 1984; Schaufler et al., 2010). Soil moisture controls aeration because water directly competes with air for pore space and slows down O₂ diffusion (Schaufler et al., 2010). When water filled pore space (WFPS) is low, there is more opportunity for gas exchange between the soil and the atmosphere, allowing for greater diffusion of O₂ into the soil and release of CO₂ from the soil. Although greater diffusion of O₂ allows for increased O₂ consumption by microorganisms, low WFPS can cause microbial osmotic stress and restrict the diffusion of substrates, affecting the rate of microorganism utilization and decomposition of those substrates (Schaufler et al., 2010). Alternatively, when WFPS is too high, O₂ diffusion is restricted, leading to anoxic conditions, and decreased rates of aerobic microbial activity. Linn and Doran (1984) found that at ~60% WFPS microbial respiration is at a maximum, and there is generally adequate O₂ diffusion for aerobic microbial activity and sufficient moisture for substrate diffusion, producing suitable environmental conditions for aerobic microbial activity.

Microbial physiology and enzyme activity are temperature-sensitive (Allison et al., 2010), such that changes in soil temperature affect the rate of C mineralization, fermentation, methanogenesis, nitrification and denitrification. At low temperatures, microbial processes generally experience reduced rates, which increase with rising temperatures. This increase is faster when rising from lower temperatures, and becomes more gradual at higher temperatures (Kirschbaum, 1995). Optimal temperature is dependent on the process, soil, and possibly the physiological

adaptation of the microorganisms in soil from different locations (Fang and Moncrieff, 2001). Soil moisture also affects the amount of energy needed to warm the soil, further confounding the effects of soil moisture and temperature.

Through alterations to the soil environment and substrate availability, the combination of temperature and moisture amplify effects on the rate of SOM decomposition and C and N biogeochemical processes, and thus GHG production and consumption. Increased CO₂ production can result from increasing soil moisture content and temperature, which leads to increases in the rate of microbial activity and O₂ consumption, while restricting O₂ diffusion. This can lead to the establishment of anaerobic microsites that support microbial production of CH₄ and its precursors, as well as nitrification and denitrification, which produce N₂O.

Most studies of RWM have focused on the impact of traditional amendments, like green composts or manures, on crop yields and soil properties (Johnson et al., 2007). In contrast, there is little information about the effects of novel RWM like paper fiber, dehydrated food waste, yardwaste and biosolids co-compost and multisource composts. Because of their potential effects on soil properties and biogeochemical processes, the use of novel RWM as agricultural amendments can affect the flux of CO₂, CH₄ and N₂O from agricultural soils (Thangarajan et al, 2013). Agricultural soils are already a concern when it comes to GHG fluxes due to the release of previously stored carbon as CO₂ and CH₄, as well as N₂O production from fertilizer. Due to the higher global warming potential of CH₄ and N₂O fluxes (25 and 298 times that of CO₂, respectively) (IPCC, 2007), potential increases in the flux of these gases are of particular concern. Therefore, it is important to understand the impact of the use of

RWM as soil amendments on GHG fluxes before advocating their use in agricultural production.

In a previous field study (Waggoner et al, 2016), we examined the effects of novel RWM as soil amendments on GHG flux in soil used to grow sweet corn. Soil was amended with paper fiber with chicken manure (PF), dehydrated food waste (DFW), yard waste compost (YW), biosolids and yard waste compost (BIO), multisource compost (MC), or mineral fertilizer (MF). Plots amended with DFW or PF had maximum CO₂ flux in June, while maximum CO₂ flux was observed from composted amendments (BIO, MC, YW) and MF in August. We generally found net CH₄ consumption during the study, and although there was also net CH₄ production, fluxes were never significantly different from the control. Nitrous oxide flux was variable between months and amendments, with no detectable temporal patterns. The lack of control on environmental variables in the field study made it difficult to identify the factors controlling GHG fluxes, leading to our lab experiment. In the present study, we used microcosms with soil moisture adjusted to permanent wilting point, field capacity, and saturation and incubated at temperatures of 10, 15, 20, and 25°C – a range of conditions likely to be encountered in the field – to better understand the role of temperature and moisture in controlling GHG fluxes from soil amended with RWM. Our objectives are to discern (i) how GHG fluxes in amended soils responded to changes in temperature and moisture, and (ii) the role of active C in the amendments, and changes in soil pH, electrical conductivity, and inorganic N on GHG flux. Based on results from the field study, we expected higher GHG flux from microcosms amended with DFW. Additionally, we hypothesized that the level of

active C in the amendments would affect the rate of CO₂ flux, with amendments having less active C producing a higher CO₂ flux later in the incubation.

Materials and Methods

Microcosms

We used microcosms to test the combined effects of temperature and moisture on GHG flux and soil properties after amendment with RWM or a mineral fertilizer. Microcosms consisted of 0.95-L glass Mason jars filled with 150 g dry weight soil and fitted with rubber septa in the lids for gas sampling. The soil was mapped as an Enfield silt loam. It was collected in October 2014 from the top 20 cm of a field planted with buckwheat (*Fagopyrum esculentum*) as a cover crop during the 2014 growing season. It was stored at 4°C before being sieved to pass a 2-mm-mesh sieve and air-dried.

We tested six different amendments: paper fiber mixed with chicken manure (7:1 ratio) (PF), dehydrated food waste (DFW), yard waste compost (YW), biosolids and yard waste co-compost (BIO), multisource compost (MC), and a mineral fertilizer (20N-8.7P-16.6K) (MF). Unamended soil served as control (CTL). The RWM were added at a field equivalent rate of 10 Mg C ha⁻¹, whereas the mineral fertilizer was added at a field-equivalent rate of 112 kg N ha⁻¹.

To account for the flush of CO₂ expected when dry soil is rewetted, we incubated the microcosms at room temperature with lids removed for 2-3 days after adding deionized (DI) water but before adding the amendments (Kim et al., 2012). The soil water content was adjusted by adding DI water to achieve a final volumetric soil moisture content of 10%, 25%, or 44%, corresponding to the permanent wilting point, field capacity, and saturation, respectively. The bulk density was adjusted to 1.0 g cm⁻³

by tapping the jar on the lab bench, and the microcosms incubated in the dark at 10, 15, 20, or 25°C for 14 days. Each combination of moisture, temperature and amendment was replicated three times.

Gas sampling and analysis

The concentrations of CO₂, CH₄, and N₂O in the headspace of the microcosms were determined for gas samples collected every 2 days over 14 days. We used a 20-mL gas-tight syringe that was pumped repeatedly to mix the gases in the headspace prior to sample collection. Gas samples were transferred immediately to a 20-mL evacuated glass vial, and the samples were analyzed using a Shimadzu GC-2014 Greenhouse Gas Analyzer (Shimadzu Corp., Kyoto, Japan) fitted with a flame ionization detector (for CH₄ and CO₂ (post-methanizer)) and an electron capture detector (for N₂O). After gas sampling, the lid from each microcosm was removed, the headspace flushed with humidified compressed air, and the lid replaced.

Ambient concentration of each gas in air sampled from the lab was determined and subtracted from the concentration of the gas from the headspace of the microcosms. The resulting concentration, along with headspace volume (0.9 L), the cross-sectional area (0.0064 m²) of the microcosm, and the incubation time were used to calculate flux.

Soil sampling and analysis

50-g samples of soil were removed from the microcosms on Day 0, immediately after addition of the amendments, and on Day 14 of the experiment. Samples were analyzed for pH, electrical conductivity (EC), NH₄⁺ and NO₃⁻. Soil pH and EC were

determined using a 1:2 soil/water ratio with a Denver Instruments Ultrabasic UB-10 Benchtop pH meter (Denver Instruments, Bohemia, NY) and a Fisher Scientific Dual-Display conductivity meter (Thermo Fisher Scientific Inc., Waltham, MA), respectively. After extraction with 2N KCl (Gugino et al., 2009), the concentration of NH_4^+ and NO_3^- in soil was determined colorimetrically using a microplate method (Doane and Horwath, 2003; Weatherburn, 1967) with a Biotek Powerwave 340 Microplate Scanning Spectrometer (Biotek Instruments, Inc., Winooski, VT). Soil moisture content was measured gravimetrically.

Prior to the experiment, the organic matter content of soil was determined by loss-on-ignition at 550°C for 5 hours. As a measure of labile C, the active C content of the soil was determined colorimetrically by the potassium permanganate method of Weil et al. (2003) using a Biotek Powerwave 340 Microplate Scanning Spectrometer to measure absorbance at 550 nm. Total C and N content were measured by flash combustion and chromatography with a Costech Instruments ECS 4010 elemental combustion system CHNS-O (Costech Analytical Technologies Inc., Valencia, CA). Characteristics of the unamended soil are found in Table 2.1.

Characterization of residual waste materials

Total C and N content of the RWM was determined at the University of Rhode Island Graduate School of Oceanography (Narragansett, RI) using an EA1108 CHN Analyzer (CE Instruments, Inc., Wigan, Ireland). Levels of NH_4^+ and NO_3^- , pH (1:5 amendment/water ratio) and active C content were determined as described above. The paper fiber and chicken manure were tested separately, and values for the mixture

mathematically calculated at a 7:1 ratio. Characteristics of the individual RWM are found in Table 2.1.

The RWM were also tested separately for their capacity to produce GHG. Residual waste material (0.5 g dry weight) was mixed with 5 mL DI water and placed in 38-mL glass serum bottle. The bottle was capped with a rubber septum, crimp sealed with an aluminum collar, and incubated at 25°C. Gas samples were collected once per day for 3 days and analyzed as described above. The bottles were flushed with humidified compressed air and resealed after each sample was collected. Each amendment was replicated three times.

Statistical analyses

Statistical analyses were performed using SigmaPlot 11.0 (Systat Software Inc., 2008). A three-way ANOVA using the Holm-Sidak test was used to identify statistically significant ($p < 0.05$) differences for the effect of temperature, moisture, and soil amendment relative to the control for GHG flux, changes in pH, and changes in EC. Pearson product moment correlation coefficients were used to evaluate the relationship among soil properties and gas flux.

Results and Discussion

Carbon dioxide

Time course of CO₂ flux. Although we controlled for the flush of gases that result from rewetting soil (Kim et al., 2012), the maximum CO₂ flux (CO₂ max) over the time course of the sampling period always occurred on the first day of sampling, or shortly thereafter (data not shown). This was similar to reports from Fang and Moncrieff (2001) on incubation studies of small soil samples. This timing suggests that maximum flux could be partially related to disturbance of the soil during microcosm setup, as this happened in all samples, including CTL. The higher flux could result from a priming effect, caused when a flush of more readily decomposable nutrients, such as RWM, is added to the soil, increasing activity from previously dormant microorganisms (Fontaine et al., 2003).

Maximum CO₂ flux. Because increasing temperatures lead to increased microbial activity and higher rates of decomposition (Allison et al., 2010; Conant et al., 2011; Garcia-Marco et al., 2014), we expected that warmer temperatures would result in an increased CO₂ flux. We found that temperature increased CO₂ max for all amendments and moisture contents, similar to results reported by Amos (2005) and Schaufler et al. (2010). We observed the highest CO₂ max from DFW (81.13 mg CO₂-C/m²/d) at 25°C and field capacity (Fig. 2.1). Under these conditions, CO₂ max followed the order: DFW > MC > CTL > PF > MF > BIO > YW, with values for DFW and MC significantly higher, and PF, MF, BIO, and YW significantly lower, than CTL (Table 2.2). The remaining amendments (except YW) also reached CO₂ max at field capacity with BIO, CTL, and MF at 20°C and PF and MC at 25°C. Yardwaste compost

achieved CO₂ max at saturation and 25°C, although the highest CO₂ flux for YW at all other temperatures was at field capacity.

Cumulative CO₂ production. To further understand how CO₂ flux is affected by RWM, we determined the cumulative amount of CO₂ produced during the 14-d incubation period. The effect of temperature and moisture observed on CO₂ max persisted, with the highest cumulative CO₂ production observed at 20°C (BIO and DFW) and 25°C (CTL, MC, MF, PF and YW) (Fig. 2.2).

In addition to the effect of temperature on CO₂ production, we also observed significant interactions ($p = <0.001$) between temperature, moisture and amendment. The highest cumulative production of CO₂ was observed at field capacity for all amendments except CTL and YW (Fig. 2.2). Saturated soil had higher CO₂ production than soil at permanent wilting point, and both generally had lower CO₂ production than soil at field capacity. Linn and Doran (1984), Fang and Moncrieff (2001), and Schaufler et al. (2010) had comparable findings, with very dry or very wet conditions resulting in reduced CO₂ flux. The reduced levels of CO₂ production in samples at permanent wilting point are due to decreased moisture content preventing biological activity (Linn and Doran, 1984). Conversely, increasing water content leads to increased CO₂ production from increased microbial activity, which is variable depending on soil properties (Linn and Doran, 1984; Schaufler, 2010). Increased water filled pore space results in reduced O₂ availability and diffusion, explaining the reduced production of CO₂ between field capacity and saturated soils (Linn and Doran, 1984; Schaufler et al, 2010).

For cumulative CO₂ production, CTL often had higher CO₂ flux relative to most amendments, although the differences were not significant (Table 2.3). Amendment with DFW and PF often led to increased CO₂ production relative to the CTL, possibly a result of addition of easily decomposable organic C and N substrates from DFW and PF, which would not have been present in the composted (BIO, MC, and YW) amendments or MF (Annabi et al., 2007). In contrast, YW and MF led to decreased CO₂ production relative to CTL. This could be due to toxicity or other negative effects on the microbial community from the YW, or to C immobilization due to the lack of additional C from MF. Biosolids, PF, and MC did not cause significant changes to CO₂ when compared to CTL, suggesting that they did not alter the soil environment enough to significantly affect microbial processes.

Amendment CO₂ production. Since these were organic amendments – except for the mineral fertilizer – we expected them to host microbial communities capable of producing GHG. We examined the possibility that some of the GHG flux from our amended soils could be produced directly by the amendments. We found that all amendments, except the DI water that was used as a control, produced CO₂ (Fig. 2.3). Paper fiber had the highest production, with amendments following the order: PF > BIO > YW > DFW > MC > MF > DI. While the amendments alone did produce GHG, this contributed to less than 0.002% of the cumulative production from the microcosms at 25°C. Thus, the flux of CO₂ from amended soil was a result of the interactions caused by the amendments, soil, moisture, and temperature (Paterson et al., 2011; Saison et al., 2006) and not produced by the amendments themselves.

Relationship of CO₂ production to active C. The cumulative amount of CO₂ from soil amended with DFW was always significantly higher than CTL (Table 2.3). We hypothesized that this was due to the level of active C in the amendment, since the amendments were applied based on equal total C content. We tested the level of active C in the amendments, expecting that amendments with higher levels of active C would have higher GHG production, specifically CO₂, than amendments with lower levels of active C (Table 2.1). Specifically, we expected DFW and PF to have the highest levels of active C and highest GHG flux based on their composition and minimal processing, since these amendments were not composted, which results in decomposition and removal of the most active C. In contrast, we expected that composted RWM (BIO, MC, and YW) would have the lowest GHG fluxes, since active C content would be lower from microbial decomposition during composting.

The active C content of the amendments was not statistically different. The amount of active C added by the amendments was also minor compared to the level of active C already present in the soil (~63 mg active C/microcosm). When the amount of active C added by the amendments was compared to cumulative CO₂ production, we found that CO₂ production was not directly related to the amendment active C levels. The differences may be better explained by the differences seen through management of moisture and temperature.

We note that the method used to determine the active C – oxidation with potassium permanganate – was developed to test active C in soil. Since the C composition of the amendments is different from soil, this method may not best represent the biologically available C in the amendments. Meijide et al. (2007) suggest

using the C:N ratio in combination with the dissolved organic carbon in order to predict the effect of amendments on C and N in soil. This could be a better approach to characterize the C content of amendments and their relationship to GHG flux, although Long et al. (2016) reported that the C:N ratio of the amendments did not provide reliable information in regard to N mineralization from these amendments.

Relationship of CO₂ production to changes in EC, pH and inorganic N. Changes in salinity and pH of the soil can affect soil microorganisms and the related decomposition and mineralization processes (Wichern et al., 2006). We found that changes in pH were primarily affected by temperature, with moisture having a minimal effect, while changes in EC were variable between moisture content and temperature for all amendments (data not shown). Increasing moisture content generally amplified the effect on EC, possibly a result of increased water content releasing more ions into the soil solution.

We observed a significant negative relationship between changes in soil pH and cumulative CO₂ flux, with decreased soil pH associated with increased CO₂ production (Table 2.4, Fig. 2.5). Additionally, there was a relationship with soil moisture and temperature (data not shown) evident among samples at 25°C and field capacity. This could suggest that microbial activity, improved by increased temperature and moisture, produced higher levels of CO₂, leading to decreased pH from increased processes such as decomposition of organic matter and nitrification. The varied extent of decrease in pH among the amendments (at 25°C all resulted in net decrease) could be the result of differences in buffering capacity of the amendments. Although organic amendments have been used to counteract the effects

of salinization (Wichern et al, 2006), or could potentially increase salinity, of which there are potential detrimental effects to microorganisms (Rietz and Haynes, 2003); nevertheless, there was no significant relationship between changes in EC and cumulative CO₂ production (Table 2.4, Fig. 2.4).

Although the C and N cycle are tightly coupled, suggesting there may be a relationship between changes in inorganic N and cumulative CO₂ production, no linear relationship was found (Table 2.4).

Comparison to field study. In the field study (Waggoner et al., 2016), we rarely saw significant differences between the amended soils and the control, except in June, when DFW and PF were both significantly higher than the control and on succeeding sampling dates. While this trend of significantly higher CO₂ flux continued for DFW, we did not always see the same significant differences with PF in the microcosm incubations.

Methane

Time course of CH₄ flux. We observed fluctuations between net production (a result of a higher rate of methanogenesis than CH₄ oxidation) and net consumption (higher CH₄ oxidation than methanogenesis) in CH₄ during incubation (data not shown). Temperature affected the magnitude and timing of the fluctuations. At 25°C, there was minimal fluctuation from MC, MF, PF, and YW, while DFW and CTL began the incubation with net production at sampling day 2, but generally resulted in net consumption after sampling day 6. In contrast, at 10°C, all samples (except MF) began with net production, with subsequently lower values until day 8, when values increased again. Both magnitude and timing of fluctuation between net production and

net consumption remained variable at 15 and 20°C. These variations in the timing of the fluctuation could be a result of the need to establish anaerobic microsites and produce substrates needed for methanogenesis, with the initial production a result of the incubation after rewetting the soil, but prior to amendment, which we used as a counter measure for the increased flux of CO₂ that comes from rewetting soil.

Maximum CH₄ flux. Maximum flux of CH₄ (CH₄ max) responded with significant differences to changes in temperature and amendment only at saturation (Fig. 2.1), which was similar to findings from Garcia-Marco et al. (2014), who reported response of CH₄ flux mainly to soil temperature, with only a small response to moisture. In general, CH₄ max was positive (net production), with increased net production at higher temperatures. We observed the highest CH₄ max from all amendments at 25°C and saturation following the order DFW > MC > MF > PF > BIO > YW > CTL, but only YW, BIO, and MC were significantly higher than CTL (Fig. 2.1, Table 2.2). This could be a result of decreased CH₄ oxidation from the increased temperature. Increased moisture could also restrict the CH₄ in the headspace from diffusing into the soil for oxidation.

Cumulative CH₄ production. Cumulative CH₄ production followed a trend of decreased net production/increased net consumption with increasing temperature (Fig. 2.2). Interestingly, at 20°C and saturation, BIO, MC, PF, and YW had net consumption, while CTL (0.03 mg CH₄-C/m²/d) and DFW (0.02 mg CH₄-C/m²/d) had the highest net production at permanent wilting point and saturation, respectively. At 15°C, CTL had net consumption, whereas most other amendments had net production (Fig. 2.2).

Saturated soil generally produced the highest net production/lowest net consumption of CH_4 . This is likely due to increased methanogenesis, a result of reduced gas diffusion leading to the establishment of anoxic conditions and production of substrates, as well as from decreased methane oxidation. Decreased oxidation could also result from reduced gas diffusion, leaving methanotrophs unable to access the CH_4 or O_2 necessary for CH_4 oxidation (Segers, 1998). In contrast, lower moisture allows for greater gas diffusion and increased methane oxidation (Smith et al., 2003). Additionally, because of the need for an anaerobic environment and substrates created under anoxic conditions for methanogenesis, lower moisture content also leads to less CH_4 production (Smith et al, 2003; Segers, 1998).

Amendment CH_4 production. When we examined the amendments separately for their capacity to produce or consume CH_4 , all amendments resulted in net CH_4 consumption following the order $\text{DFW} > \text{DI} > \text{PF} > \text{YW} > \text{BIO} > \text{MF} > \text{MC}$ (Fig. 2.3). While this suggests that the amendments, once incorporated into the soil, could promote increased methane oxidation or reduced production, CH_4 consumption from the amendments accounted for a minute fraction of the net change in CH_4 levels at 25°C , as was the case for CO_2

Relationship of CH_4 production to change in N. Methane consumption is sensitive to N fertilization because there is direct competition for the binding site on methane monooxygenase between CH_4 and NH_3 due to their similarity in size and shape (Sylvia et al., 2005; Schauffler et al, 2010). Because of this and the addition of N compounds in the amendments, we expected to find a relationship between change in either inorganic N and CH_4 or NH_4^+ and CH_4 . No relationship was found for change in

inorganic N and CH₄, but there was a significant positive correlation between increase in NH₄⁺ and increased CH₄ (Table 2.4, Fig. 2.6 and 2.7). This is likely due to the increase in NH₄⁺ restricting CH₄ oxidation through competition for methane monooxygenase (Sylvia et al, 2005).

Comparison to field study. The microcosms incubated at warmer temperatures generally resulted in net CH₄ consumption from the amended soils. When we altered the moisture and temperature of the microcosms, we observed significant interaction between moisture, temperature and amendments, with significant differences between the amended soils and CTL. Methane flux in the field study (Waggoner et al., 2016) was variable, generally resulting net CH₄ consumption, regardless of amendment type, with no significant differences between the amended soils and the CTL.

Nitrous oxide

Time course of N₂O flux. We generally observed the highest N₂O fluxes (N₂O max) from saturated treatments, while fluxes from treatments at permanent wilting point and field capacity were minimal. The N₂O flux in saturated treatments were always highest on the first day of sampling for all amendments and CTL at 20 and 25°C (data not shown). However, at 10°C (BIO, MC, MF, and YW) and 15°C (MF and YW) the highest flux was observed on the second day of sampling. The differences in the time course for the maximum flux could be the result of incomplete denitrification caused by high O₂ levels, and a function of how quickly the available O₂ in the pore space was consumed. The O₂ is consumed more rapidly at warmer temperatures, producing N₂O max earlier. Since O₂ diffuses slowly at high WFPS, denitrification would have resulted in a higher ratio of N₂:N₂O. Linn and Doran (1984)

reported similar findings, with maximum N₂O flux via denitrification occurring shortly after rainfall, which increases WFPS, creating conditions for denitrification.

Maximum N₂O flux. Unlike CO₂ and CH₄ flux, N₂O flux did not have significant interactions between temperature and moisture or amendment, although there was a significant interaction between moisture and amendment ($p = 0.004$). The maximum N₂O flux (N₂O max) was observed in saturated soil (Fig. 2.1). This is in line with findings of a positive relationship between moisture and N₂O (Schaufler et al., 2010), but contrary to results showing that N₂O flux increased with temperature (Garcia-Marco et al, 2014; Schaufler et al., 2010). While many amendments produced N₂O max higher or lower than CTL, significant differences only existed at saturation, when temperature was not considered (data not shown).

Cumulative N₂O production. The cumulative amount of N₂O produced also responded to moisture content, with no detectable pattern in response to temperature (Fig. 2.2). The cumulative N₂O produced from amended samples was only significant when the value was greater than CTL at a specific moisture and temperature (Table 2.3). No trend between amendment, moisture content and temperature was found. The exception was DFW, which had generally higher production than CTL, and increased with temperature up to 20°C under saturation.

Amendment N₂O production. The amendments by themselves had net N₂O production (Fig. 2.3). The highest N₂O production was from YW (0.02 µg N₂O-N/g amendment) with the rest following the order YW > BIO > MC > MF > PF > DFW > DI. Again, N₂O production from the amendments was a minor portion of the N₂O flux from the microcosms.

Relationship of N₂O production to changes in inorganic N and pH. Most N₂O produced in soil comes from nitrification and denitrification by microorganisms. We found a negative relationship between change in inorganic N and cumulative N₂O production (Fig. 2.7). This suggests that when the overall change in inorganic N is negative, N₂O net flux is increased, suggesting that nitrification and denitrification are resulting in N₂O production.

To further study the role of inorganic N in N₂O production, we also examined the relationship between change in NH₄⁺ and NO₃⁻, respectively, with cumulative N₂O production at saturation (data not shown). We found a positive significant correlation between change in NH₄⁺ and N₂O, and a negative significant relationship between change in NO₃⁻ and N₂O. In the saturated samples, we observed a general increase in NH₄⁺ from samples at 20°C (CTL, DFW, MC, YW) and 25°C (all), while NO₃⁻ decreased at all temperatures, except DFW and MF at 10°C, where it increased. An increase in NH₄⁺ suggests increased N mineralization and/or release from cation exchange sites, creating a larger pool of inorganic N. Increased saturation could assist in this process through the diffusion of substrates or other cations in the soil solution.

The decrease in NO₃⁻ with simultaneous N₂O production could indicate incomplete denitrification caused by presence of O₂ (Sylvia et al., 2005). Nitrification is another possible source of N₂O production, which as an aerobic process would have also been likely in the beginning of the incubation when more O₂ was diffused throughout the soil.

Because nitrification can produce N₂O and release acidity into the soil, and decreased soil pH can affect the ratio of N₂O:N₂ produced from denitrification, we

examined the relationship between change in pH and cumulative N₂O flux (Fig. 2.5) (Amos et al., 2005; Thangarajan et al., 2013; McMillan et al., 2016). No relationship was observed (Table 2.4).

Comparison to field study. In the microcosms, we primarily observed net production of N₂O, while in the field, measurements of N₂O varied between net production and net consumption.

Global warming potential

GHG emissions are commonly converted to their CO₂ equivalent in order to understand the environmental and atmospheric impacts, or global warming potential (GWP), of increased production. The fluxes of CH₄ and N₂O from soil to the atmosphere are smaller than CO₂, but these gases are more powerful: CH₄ and N₂O have 25 and 298 times, respectively, the global warming potential of CO₂ (IPCC, 2007). Thus, small changes to CH₄ and N₂O fluxes can have greater consequences than increased CO₂. When we converted the cumulative fluxes of CH₄ and N₂O to their CO₂ equivalent (Fig. 2.8), soils at permanent wilting point and field capacity did not contribute substantially to the GWP from CH₄ or N₂O. Only at saturation was there an increased GWP from CH₄ or N₂O. This was particularly true to DFW at 20°C, especially at saturation. Caution is needed with the use of DFW in fields that experience highly saturated conditions.

Conclusions

Carbon dioxide flux was the most affected by moisture, temperature and amendment interactions, although, only DFW had consistent significantly higher CO₂ production than CTL. Biosolids, MC, and YW generally had CO₂ production lower than the CTL, suggesting the amendments may provide beneficial qualities for future C sequestration without increasing CO₂ emission substantially. Dehydrated food waste and PF often resulted in CO₂ production higher than the CTL; but using active C levels to predict increased CO₂ flux did not help to discriminate between the non-composted and composted amendments.

Methane flux was variable, responding to temperature and amendment with minimal effect from moisture. Amendment with RWM generally resulted in lower net CH₄ production/net consumption, while the CTL resulted in lower net CH₄ consumption/net production. Nitrous oxide production was only observed at substantial levels under saturation, and was rarely significantly higher than the CTL. Dehydrated food waste was the exception, with the highest CO₂, N₂O, and CH₄ production under most temperature and moisture conditions, and often significantly higher than the CTL.

Our results suggest that use of RWM materials as agricultural soil amendments will have different effects on GHG flux. Greenhouse gas flux responses are affected by moisture and temperature, but the effect may not be major. To further our understanding of how the use of RWM as soil amendments alters soil properties and GHG flux, it would be important to examine different agricultural management and crop production practices using long term studies.

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Figures and Tables

Table 2.1. Characteristics of soil and amendments used in the microcosm experiment. Values are means (n=3-4). After Long et al (2016).

	NH_4^+	NO_3^-	C	N	C:N	Water Content	Organic Matter	Active C	pH
Material	mg/kg	mg/kg	%	%		%	g/g	g/kg	
Pre-amendment soil	-	-	22	2	15.1	1*	5	0.42	5.0
Biosolids/yardwaste co-compost	48	4009	33	3	10.3	45	54	55.98	7.9
Dehydrated food waste	36	205	49	3	14.4	3	63	98.7	5.5
Multisource compost	431	392	15	2	9.2	40	19	49.73	7.1
Mineral fertilizer	-	-	-	-	-	0.0	0.0	50.26	4.6
Paper fiber/chicken manure (7:1 ratio)	331	1226	40	1	33.9	102	79	62.03 [§]	6.4/7.0 [§]
Yardwaste compost	201	2.23	20	2	13.0	78	34	52.82	6.7

* Water content of air-dried soil

[§] pH for paper fiber and chicken manure measured separately

Table 2.2. Order of least square means (n=3) for maximum GHG flux from lowest to highest by gas, temperature and moisture content. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost; PWP = permanent wilting point; FC = field capacity; and SAT = saturation. *Denotes significant difference from CTL when significant interaction between amendment, temperature and moisture content is present.

Gas	Temp (°C)	Moisture	Maximum GHG Flux mg/m ² /d produced						
			Lowest to Highest						
CO ₂	10	PWP	YW*	MF	BIO*	MC*	CTL	PF	DFW
		FC	YW*	MF*	MC	BIO*	PF*	DFW*	CTL
		SAT	YW	MF	MC	BIO	CTL	DFW*	PF
	15	PWP	MF	PF*	YW*	BIO	MC	CTL	DFW*
		FC	MF	YW	PF	BIO	MC	CTL	DFW
		SAT	MF	YW*	BIO*	PF	MC*	CTL	DFW
	20	PWP	MC*	MF*	YW	BIO	PF	CTL	DFW*
		FC	YW*	MC*	PF*	MF	BIO*	CTL	DFW*
		SAT	MF*	YW*	CTL	BIO*	MC*	PF*	DFW*
	25	PWP	BIO	YW*	MF	CTL	PF	MC	DFW
		FC	YW*	BIO*	MF*	PF*	CTL	MC*	DFW*
		SAT	MF	BIO	PF	YW*	CTL	MC	DFW*
CH ₄	10	PWP	MF	YW	DFW	BIO	PF	CTL	MC
		FC	MF	YW	PF	MC	BIO	DFW	CTL
		SAT	MF	YW	BIO	MC	CTL	DFW	PF
	15	PWP	CTL	MF	YW	BIO	DFW	MC	PF
		FC	MF	CTL	YW	MC	BIO	PF	DFW
		SAT	MF	MC	BIO	DFW*	PF*	YW	CTL
	20	PWP	YW	MC	PF	MF	BIO	CTL	DFW
		FC	YW	PF	MC	MF	BIO	CTL	DFW
		SAT	YW*	MC*	PF*	BIO*	MF*	DFW*	CTL
	25	PWP	BIO	MC	YW	MF	DFW	CTL	PF
		FC	CTL	MC	BIO	MF	YW	PF	DFW
		SAT	CTL	YW*	BIO*	PF	MF	MC*	DFW
N ₂ O	10	PWP	YW	DFW	BIO	MC	MF	PF	CTL
		FC	MF	CTL	PF	DFW	MC	BIO	YW
		SAT	YW	MF	MC	BIO	DFW	CTL	PF
	15	PWP	BIO	DFW	YW	PF	CTL	MF	MC
		FC	BIO	MC	PF	YW	CTL	MF	DFW
		SAT	CTL	PF	MF	MC	BIO	YW	DFW
	20	PWP	DFW	CTL	BIO	PF	MF	MC	YW
		FC	CTL	BIO	PF	MF	MC	YW	DFW
		SAT	BIO	YW	MC	PF	MF	CTL	DFW
	25	PWP	BIO	MC	YW	MF	DFW	CTL	PF
		FC	CTL	MC	BIO	MF	YW	PF	DFW
		SAT	CTL	YW	BIO	PF	MF	MC	DFW

Table 2.3. Order of least square means (n=3) for cumulative amount of GHG produced from lowest to highest by gas, temperature and moisture content. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost; PWP = permanent wilting point; FC = field capacity; and SAT = saturation. *Denotes significant difference from CTL when significant interaction between amendment, temperature and moisture content is present.

Gas	Temp (°C)	Moisture	Cumulative Gas Flux (mg/m ² /d) produced						
			Lowest to Highest						
CO ₂	10	PWP	YW*	MF*	BIO*	MC	CTL	PF*	DFW*
		FC	YW*	MF*	MC*	BIO*	PF*	CTL	DFW*
		SAT	YW	MF*	BIO	MC	CTL	PF	DFW*
	15	PWP	MF*	PF*	YW*	BIO*	MC*	CTL	DFW*
		FC	MF	YW	BIO	PF	CTL	MC*	DFW*
		SAT	MF*	BIO*	YW*	CTL	PF*	MC	DFW*
	20	PWP	MF	MC*	YW	BIO	CTL	PF	DFW*
		FC	YW*	MC	MF*	BIO*	CTL	PF*	DFW*
		SAT	MF*	MC*	CTL	BIO*	PF*	YW*	DFW*
	25	PWP	MF*	YW*	BIO*	MC	CTL	PF	DFW
		FC	MF*	BIO*	YW*	MC*	CTL	PF*	DFW*
		SAT	MF*	BIO*	PF	MC	YW*	CTL	DFW*
CH ₄	10	PWP	YW	MF	BIO	DFW	PF*	MC*	CTL
		FC	MF	YW	DFW	PF	MC	BIO	CTL
		SAT	YW*	MF*	BIO*	PF*	MC*	DFW*	CTL
	15	PWP	CTL	MF	YW*	DFW	BIO	PF	MC
		FC	CTL	MF	MC	YW	PF	DFW	BIO
		SAT	CTL	BIO	MF*	MC	PF	DFW*	YW
	20	PWP	MC*	PF	YW	CTL	MF	BIO*	DFW*
		FC	YW*	PF*	MC*	CTL	MF*	BIO	DFW*
		SAT	PF	YW*	MC*	CTL	MF	DFW	BIO*
	25	PWP	MF	MC	PF	YW	BIO	CTL	DFW*
		FC	PF	MF*	YW*	BIO*	MC*	CTL	DFW*
		SAT	PF	MF	BIO	MC	YW	DFW*	CTL
N ₂ O	10	PWP	DFW	MC	CTL	MF	BIO	YW	PF
		FC	CTL	MF	DFW	PF	MC	BIO	YW
		SAT	YW	CTL	MC	DFW	MF	BIO	PF
	15	PWP	BIO	YW	CTL	DFW	MF	PF	MC
		FC	BIO	MC	CTL	YW	PF	MF	DFW
		SAT	CTL	PF	MC	BIO	MF	YW	DFW
	20	PWP	DFW	CTL	MF	BIO	PF	MC	YW
		FC	CTL	BIO	MF	PF	MC	YW	DFW
		SAT	BIO	YW	PF	MC	MF	CTL	DFW
	25	PWP	MC	BIO	YW	MF	CTL	DFW	PF
		FC	MC	BIO	CTL	MF	YW	PF	DFW
		SAT	CTL	YW	BIO	PF	MF	MC	DFW

Table 2.4 Pearson product moment correlation coefficients of relationships between GHG flux and soil properties. Bold values indicate significant correlation ($p < 0.05$).

Gas Flux	Variable	All	CTL	BIO	DFW	MC	MF	PF	YW
CO ₂	pH	-0.48	-0.58	-0.54	-0.70	-0.48	-0.5	-0.27	-0.64
	EC	0.29	0.40	0.46	0.44	0.13	0.39	0.21	0.02
	Inorg N	0.06	0.05	0.05	0.40	-0.01	-0.06	-0.02	-0.31
CH ₄	pH	0.26	0.28	0.87	0.12	0.57	0.09	0.19	0.42
	EC	0.09	0.68	-0.46	0.50	-0.53	0.13	-0.39	-0.68
	Inorg N	-0.001	-0.5	-0.05	0.41	-0.16	-0.45	0.24	0.33
	NH ₄	0.22	0.3	-0.44	0.41	0.23	0.12	0.11	0.08
N ₂ O	pH	0.0004	0.14	0.33	-0.12	0.15	0.06	0.06	0.12
	EC	0.04	0.24	-0.44	0.42	-0.53	-0.29	-0.25	-0.73
	Inorg N	-0.45	-0.62	-0.68	-0.53	-0.81	-0.70	-0.68	-0.64
	NO ₃	-0.46	-0.55	-0.78	-0.62	-0.88	-0.83	-0.62	-0.7
	NH ₄	0.37	0.45	0.64	0.72	0.91	0.44	0.43	0.33

Figure 2.1. Least square mean (n=3) for maximum GHG flux from each amendment as a function of moisture and temperature. Bars represent standard error of mean. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost.

Figure 2.2. Least square mean (n=3) for cumulative amount of GHG produced from amended soils as a function of temperature and moisture. Bars represent standard error of mean. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost.

Figure 2.3. Mean (n=3) GHG flux from 0.5g of soil amendments with 5mL deionized water, or deionized water alone (used as a control), measured over 3 days. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost.

Figure 2.4. Relationship between change in EC and cumulative GHG production/consumption for 14 day sampling period in amended soils. Bars represent standard error of GHG mean. Line is linear regression of all treatments. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost.

Figure 2.5. Relationship between change in pH and cumulative GHG production/consumption for 14 day sampling period in amended soils. Bars represent standard error of GHG mean. Line is linear regression of all treatments. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost.

Figure 2.6. Relationship between mean (n=3) change in ammonium over 14 days and mean (n=3) methane flux over 14 days by amendment. Bars represent standard error of GHG mean. Line is linear regression of all treatments. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost.

Figure 2.7. Relationship between change in inorganic N content and cumulative GHG flux for 14 day sampling period in amended soils. Bars represent standard error of GHG mean. Line is linear regression of all treatments. CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost.

Figure 2.8. Cumulative GHG emissions as CO₂ equivalents (N₂O and CH₄ at 298 and 25 times CO₂, respectively). CTL = control; BIO = biosolids and yardwaste co-compost; DFW = dehydrated food waste; MC = multisource compost; MF = mineral fertilizer; PF = paper fiber with chicken manure; YW = yardwaste compost. *Methane production was not large enough to show on graph.*

CONCLUSIONS

The use of RWM as soil amendments affected the magnitude, direction and timing of GHG flux, and fluxes of CO₂, CH₄, and N₂O responded to differences in temperature and moisture. In the microcosms, we were able to replicate the increased magnitude of CO₂ flux from the amended soils in the field study. The levels of CO₂ flux in the microcosms were smaller than the field study due to the lack of soil horizons in the microcosms. In the field, biogeochemical processes, moisture and temperature are affected variably throughout the soil horizons, which is important to consider when making management decisions based on microcosm results.

In both the field and microcosm studies, DFW resulted in significantly higher flux values than the CTL for CO₂, CH₄, and N₂O, suggesting that it could cause a problem for future use as an agricultural soil amendment. However, this analysis did not take into account GHG emissions during the production and composting procedures prior to application. Therefore, we would be better served using a life cycle analysis of all amendments, before making the recommendation for use based solely on GHG production. It is also important to note that, although DFW was one of the highest GHG producers in our study, it was still below reported ranges from other studies. We were not able to accept the hypothesis that the differences in GHG flux from the amended soils could be a result of the differing levels of readily degradable organic C (active C) in the amendments. Examination of the active C composition of the amendments and its effects on GHG flux would be an interesting direction for future research.

In the field, we saw inconsistent levels of CH₄ and N₂O flux produced, likely due to the high variability and complexity in production/consumption of these gases, and/or the narrow range of soil properties, such as water content and temperature. Variability is common to field studies, and led to our microcosm-scale experiment to further evaluate the effects of moisture and temperature. We often found similar or reduced CH₄ fluxes from the amended soils relative to the CTL. These amendments may be useful in promoting C sequestration through reduced GHG flux, and would be another direction for future research.

We found less variability in the flux of N₂O from the amended soil when temperature and moisture were controlled. While the amendments led to changes in soil properties and N₂O flux, the flux was generally more associated with changes in moisture content than with the amendment.

Because management practices are not mimicked throughout agriculture, future research into the effects of RWM soil amendments on GHG flux should include different application rates, management regimens and crop production systems. This is particularly true of systems where there would be increased moisture or temperatures, such as tropical and subtropical environments or in paddy rice production.

This field study only examined short-term responses to the addition of these amendments. If these amendments are to be used as a long-term solution for waste reuse, a long-term field study on the effects to soil properties and GHG flux should be performed. This would allow for examining the effects of reapplication, as well as providing an opportunity to gather information about the offseason and residual effects of application.

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