A Rapid Method for Measuring Feces Ammonia-Nitrogen and Carbon Dioxide-Carbon Emissions and Decomposition Rate Constants

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A Rapid Method for Measuring Feces Ammonia-Nitrogen and Carbon Dioxide-Carbon Emissions and Decomposition Rate Constants

Jiyul Chang, David E. Clay,* Sharon A. Clay, Alexander J. Smart, and Michelle K. Ohrtman

ABSTRACT

A rapid approach is needed for determining the effectiveness of precision conservation on soil health as evaluated using CO₂ and NH₃ emissions. This study demonstrated an approach for calculating CO₂–C and NH₃–N emissions and associated rate constants when feces were applied to bare soil or soil + vegetation. In addition, point CO₂–C emission measurements were compared with near continuous measurements. The CO₂–C emissions were measured at 2 h intervals over 20 d, whereas ammonia volatilization was measured three times daily for 7 d. Total CO₂–C emissions over 20 d were 5% lower [186 g CO₂–C (m² × 20 d) −1] than point measurement collected at 1100 h every day [197 g CO₂–C (m² × 20 d) −1], and about 10% lower than if collected every 2 d [206 g CO₂–C (m² × 20 d) −1]. A Fast Fourier transformation (FFT) showed that temperature and NH₃–N and CO₂–C emissions followed diurnal cycles and that they were in-phase with each other. Over 7 d, 20% of feces NH₃–N was volatilized and that this loss was similar when feces were applied over vegetation or mixed into the soil. Feces additions increased the amplitude of the CO₂–C diurnal cycle, and the fecal-C first-order rate degradation constants were higher when mixed with soil [0.0109 ± 0.0043 g(g×d) −1, p = 0.1] than applied over vegetation [0.00454 ± 0.00336 g(g×d) −1, p = 0.1].

Core Ideas

• Carbon storage and ammonia volatilization from feces can be quantified using techniques described in this article.
• Carbon dioxide and NH₃ emission follow diurnal cycles and it is difficult to accurately predict CO₂ loss and ammonia volatilization based on point measurements.
• Conducting rapid assessments that produce definitive findings helps build trust between scientists and on-farm producer collaborators.

Collaborative projects between farmers/ranchers and scientists can be very rewarding, as well as produce lasting positive impacts on the environment (Smart et al., 2015). However, success requires the development of trust between the farmer and the scientists, and ability to use short-term field experiments to produce results that can be communicated to the farming community in a timely manner. In projects addressing soil health, this may involve conducting demonstration or targeted experiments focused on one or two questions. This project is focused on the question, what is the fate of the C and N in cattle feces?

Carbon and N budgets are based on accurate measurements of the C and N additions and losses. Additions represent the C or N that is added through photosynthesis or fertilizer or manure applications, whereas losses represent leaching, erosion, and gaseous emissions. Research has shown that management, soils, and climatic conditions interact to influence both additions and losses in ecological systems. To accurately measure nutrient additions and losses, sampling approaches must be tested and modified for each unique problem (Clay et al., 1996, 2006; Chang et al., 2016b).

Three basic approaches have been used to determine CO₂ emissions in grassland systems (Fynn et al., 2009). The first approach measures CO₂–C or NH₃ emission in the laboratory (Murwira et al., 1990; Van Kessel et al., 2000; Kyvsgaard et al., 2000; Powell et al., 2006; Ayadi et al., 2015). Laboratory experiment are most useful for measuring mineralization potential (Franzluebbers et al., 2000; Van Kessel et al., 2000), evaluating responses mechanisms (Adu and Oades, 1978), or determining the impact of a specific treatment on many factors including biological activity (Clay et al., 1990). However, the removal of the samples from the field or drying and grinding the samples can change the soils physical and biological characteristics (De Nobili et al., 2006).

In the second approach, soil organic carbon (SOC) losses are determined by difference. In this approach, changes in SOC and net aboveground and belowground productivity are measured at the beginning and completion of an experiment (Schuman et al., 1999; Franzluebbers et al., 2000; Tate et al., 2003; Chang et al., 2004, 2016b; Clay et al., 2005, 2006, 2015; Derner et al., 2006; Derner and Schuman, 2007; Smart et al., 2010a, 2010b; Dunn et
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Compounds can be mineralized into CO$_2$ or integrated into the SOC (Clay et al., 2005, 2006, 2012, 2015). In this experiment, the importance of feces-C was not determined. Based on the definition of forage digestibility (Minson, 1998; Laubach et al., 2013). To calculate fecal C and N additions required, based on the high costs many of these continuous measurement experiments are not replicated. For example, Cao et al. (2004) measured CO$_2$–C emissions every 2 h using closed chambers at two experimental sites with different temperatures, rainfall, vegetative surface coverage, and grazing intensities. Based on these data, total emissions were estimated at 5560 kg CO$_2$–C (ha × year)$^{-1}$ in a lightly grazed (2.55 sheep ha$^{-1}$) and 4170 kg CO$_2$–C (ha × year)$^{-1}$ in a heavily grazed (5.35 sheep ha$^{-1}$) system. However, due to high costs many of these continuous measurement experiments are not replicated. For example, Cao et al. (2004) measured CO$_2$–C emissions every 2 h using closed chambers at two experimental sites with different temperatures, rainfall, vegetative surface coverage, and grazing intensities. Based on these data, total emissions were estimated at 5560 kg CO$_2$–C (ha × year)$^{-1}$ in a lightly grazed (2.55 sheep ha$^{-1}$) and 4170 kg CO$_2$–C (ha × year)$^{-1}$ in a heavily grazed (5.35 sheep ha$^{-1}$) system. In this experiment, the importance of feces-C was not determined. If a protocol could be developed, this basic approach may be suitable for on-farm studies. Because little fertilizer is applied to rangeland systems, long-term productivity and plant and soil health assessments may require estimates of N losses through denitrification, leaching, or volatilization (Clay et al., 1990, 1996; Smart et al., 2013; Chang et al., 2016a). However, many rangeland studies do not measure C and N cycling in the field which can result in large errors in C footprint or regional assessments (Ryden et al., 1987). For example, little information is available about the fate of feces in rangeland systems. For cattle, feces contain almost all of the organic C and about half of the excrated N. The rest of the N is contained in urine, which is composed of between 60 and 80% urea. Nitrogen losses from urine can be as high as 50% (Petersen et al., 1998; Laubach et al., 2013). To calculate fecal C and N additions and losses, the amount of fecal C and N added to the system is required. Based on the definition of forage digestibility (Minson, 2012), feces can be estimated using the following equation:

$$\text{Feces} = \text{Consumed forage} \times \left[1 - \frac{\text{digestibility (g kg}^{-1}\text{)}}{1000}\right] \quad [1]$$

Following deposition, the ammonia can be volatilized, nitrified, and used by the surrounding plants, whereas the C-containing compounds can be mineralized into CO$_2$ or integrated into the SOC (Clay et al., 2005, 2006, 2012, 2015). The above discussion highlights the importance and potential impacts in working with farmers and ranchers in collaborative projects. However, maintaining these collaborations requires active communication and the timely reporting of findings to the farmer collaborators. In addition, many of experimental approaches designed for long-term projects may not be suitable for on-farm studies. This study demonstrated a short-term approach for calculating total NH$_3$–N and CO$_2$–C emissions and associated rate constants when feces were applied to bare soil or soil + vegetation. In addition, total CO$_2$–C emissions were compared with point measurements at a specific time. Due to the limited number of chambers that can be physically connected to a single analyzer, it was not feasible for experiments to contain true replications. We overcame this hurdle by repeating the experiment in four different environments.

**MATERIALS AND METHODS**

**Carbon Dioxide–Carbon Emissions and Ammonia Volatilization**

The experimental design was a randomized complete block. Each blocks represented 20 d experiments that were initiated on 12 June, 2 July, 26 July, and 19 August in 2013. Each block was conducted at a new site, where fresh feces were applied. During each experiment, CO$_2$–C emissions were measured every 2 h, and in a linked experiment, ammonia volatilization was measured three times daily for 7 d starting on the first day of each CO$_2$ study. In this study, near continuous CO$_2$ emissions over 20 d were compared with point measurements collected at 1100 h every day, every second day, and every third day. These point measurements were contained within in the continuous data set. Each block contained each of the following treatments:

1. Lightly mixed soil,
2. Vegetation that was clipped to 2 cm,
3. Lightly mixed soil plus suspended fecal material,
4. Simulating trampling that lightly mixes the fecal material with the surface soil,
5. Fresh fecal material that was suspended above the vegetation, and
6. Fresh fecal material applied over clipped vegetation.

The experiment contained two types of controls. The first control was that feces were not applied to the soil (Treatment 1) or the soil plus vegetation (Treatment 2), whereas in the second control, the feces were physically separated from the soil (Treatment 3) or the soil plus vegetation (Treatment 5). The treatments were selected to allow for CO$_2$–C and NH$_3$–N emissions from the soil and feces to be calculated by difference. In Treatments 2, 5, and 6 the vegetation was moved to a height of 2 cm prior to the start of each replication. This height was selected to simulate very heavy grazing intensity (90% of aboveground biomass; Hart, 2001), and to prevent vegetation interference with the CO$_2$ automated sampling system. In Treatments 4 the feces were lightly mixed into the surface 7.5 cm with a trowel to simulate cattle trampling. For Treatments 3 and 5, fresh fecal materials were deposited on 14-cm diam. plastic plates that were placed on a platform suspended 2.5 cm above the soil. The plates did not interfere with automated CO$_2$
measurements. Treatments 1, 2, 3, and 5 were used to examine CO₂–C emissions from soil, vegetation, and the fecal materials. At the beginning of each block (experiment), composite soil samples consisting of eight soil cores from the 0- to 7.5-cm depth were collected from the area where the chambers were installed. These samples were not located within the areas occupied by the chambers. At the completion of each block, four soil cores from the 0- to 7.5-cm soil depth were collected from each treatment. The samples were analyzed for bulk density, ammonium N, nitrate N, total N, and total C (Clay et al., 2015).

**Site Characteristics**

This experiment was conducted on a Barnes clay loam (fine-loamy, mixed, frigid Udic Haploboroll), that was located near Brookings, SD. The coordinates of the site were 44°20'26" N, −96°48'28" W. The slope was between 0 and 2%. The climatic conditions were characterized by cold winters and hot summers, a growing season from April to October, a frost-free period that ranges from 120 to 160 d, and an average annual temperature of 6.5°C (Chang et al., 2016b). According to the Köppen classification it is characterized as Dfa. The soil texture in the surface 7.5 cm was a clay loam with a pH (water) of 7.0 and a bulk density of 1.29 g cm –3. In addition, following combustion (1000°C) and analysis, the soil was found to contain 5.3 g N kg soil –1 and 44.1 g C kg soil –1 (Clay et al., 2015). In the study area, the pasture botanical composition was 5% smooth bromegrass (Bromus inermis L.), 20% Kentucky bluegrass (Poa pratensis L.), 70% quackgrass [Elytrigia repens (L.) Desv. ex Nevski], and 5% birdsfoot trefoil (Lotus corniculatus L.). Prior to the study the site had been managed similarity for at least 5 yr.

**Climatic Conditions**

Precipitation from 1 Jan. to 31 Dec. 2013 was approximately 64 cm, which was similar to the long-term rainfall average of 62 cm. Rainfall in June, July, and August was 14.9, 9.2, and 3.9 cm, respectively, and the average volumetric soil moisture contents ([beginning + final]/2) were 0.38, 0.31, 0.33, and 0.22 g water cm –3 for the 12 June, 2 July, 26 July, and 19 August experiments, respectively. These moisture contents were measured with a commercial sensor. The average air temperatures during each experimental replication were 21.2, 23.5, 17.8, and 23.6°C for the 12 June, 2 July, 26 July, and 19 August experiments, respectively.

**Fecal Collection and Characterization**

Fecal materials were collected from four adult cows grazing a pasture when the experiments were initiated in June 2013. As standard in the region, the livestock diets were augmented with an appropriate feed supplement containing Ca, P, Na, Cl, Mg, K, Cu, Se, Zn, and Vitamins A, D3, and E. Based on forage analysis, the grazed forage had a digestibility of between 600 and 700 g kg –1 and it contained 180 g crude protein kg –1, 530 g neutral detergent fiber (NDF) kg –1, 290 g acid detergent fiber (ADF) kg –1, and 91 g ash kg –1. The fecal materials were collected in a bucket before it reached the soil. After collection, the materials were mixed, stored in sealed containers, and cooled to 5°C. The average fecal pH and moisture content (MC) were 7.5 and 85% [MC = 100×(wet-dry)/wet weight], respectively. The same fecal material was used in all experimental blocks. Dried fecal material contained 18.2 g total N kg –1 and 38.5 g total C kg –1, which was determined on a ratio mass spectrometer after combustion at 1000°C. The δ¹³C value was −28.62 %o, which indicated that the excreted materials were primarily derived from C₃ plants (Kim et al., 2008). Inorganic N was extracted from fresh fecal materials with 1 M KCl and analyzed on a spectrometer to determine fecal NH₄–N, which averaged 370 mg NH₄–N (kg dry fecal material) –1 (Kim et al., 2008).

Quantifying Carbon Dioxide-Carbon and Ammonia-Nitrogen Emissions

In the CO₂–C emission experiment, one fecal pile (500 g wet weight equivalent to 75.4 g dry material or 29 g C) was placed in the center of a 314 cm² chamber. This deposition rate was equivalent to 15.9 kg wet fecal material m –2 (2.4 kg dry fecal material m –2 or 920 g C m –2). The feces size was selected to ensure that CO₂–C that was derived from soil, plants, and feces could be accurately measured. The CO₂–C gas flux from each treatment was measured every 2 h over 20 d by an 8100A Automated Soil CO₂ Flux System (LI-COR, Lincoln, NE) that was connected to six gas chambers. Soil surface temperatures were measured continuously with thermocouples.

In the ammonia volatilization experiment, the fecal deposition rate was 1.72 kg dry fecal m –2 which contained 636 mg NH₄–N m –2. The fecal material and soil were open to the atmosphere between collection periods and covered to make a closed gas sampling chamber when gas samples were collected. The collection chambers had width, length, and height dimensions of 22 by 30 by 21 cm with an effective air volume (total volume – pump volume) of 11.9 L. The NH₄–N gas was captured three times a day (700, 1400, 1900 h) for 7 d using an electric pump placed above the soil within the chamber to push air at the rate of 57.6 L h –1 for 20 min through a glass bottle containing 20 mL of boric acid (0.32 M H₃BO₃). The total amount of trapped NH₃ gas was determined by titration with 0.0025 M H₂SO₄ (Clay et al., 1990). The sampling protocols were selected based on the expected air temperatures (Clay et al., 1990). The NH₃–N trapping efficiency was calculated to be 69.5±11.9% by placing a known amount of NH₃ on an impervious surface, followed by NH₃ collection and analysis as described above. The efficiency was calculated with the equation, %trapped = 100×[applied NH₃–trapped NH₃]/applied NH₃. The efficiency value was used to correct the measured NH₃–N losses.

The percentage of the NH₃ loss from feces after 7 d was calculated using the equation, %feces N loss = [{(treated-control)/[(total mg NH₄–N m –2 added)]}]. For example, [100×(483 mg NH₄–N/m² – 364 mg NH₄–N/m²)/370 mg NH₄–N/kg feces×1.72 kg feces/m²] = 18.7%]. In this equation, the control is the ammonia loss in Treatments 1 and 2, and the treatments are the losses in Treatments 3 through 6.

Determining Carbon Dioxide and Ammonia Cycles and Phase Shift

The FFT of the air temperatures, NH₃–N, and CO₂–C emissions were used to convert the temporal data to the frequency domain (Chang et al., 2016a). This analysis was conducted using Microsoft Excel using a method reported by Klingenberg (2005). This analysis is used to determine patterns and phase shifts in temporal data sets (Fig. 1). The FFT analysis can be used to identify
the different cycles that occur within the data set, and if two data sets are in or not in phase with each other. For example, it can be used to determine the temperature phase shift with increasing soil depth. Over longer periods of time, this approach can be used to separate daily and seasonal cycles from each other (Thoning et al., 1989). Figure 1 shows that the FFT analysis could be used to characterize the phase shift in the CO₂–C. Two data sets consist of an original data set and one that was offset 6 h. Both data sets had near identical frequency distributions, however analysis of phase angle showed that the two cycles were offset 6 h.

A FFT of the CO₂–C and temperature data showed that the temperature and CO₂–C cycle phase shift was 19 h. Chang et al. (2016a) had similar results. It is important to point out that not all biological systems follow identical patterns and phase shifts. For example, Clay et al. (1990) reported that soil water and soil temperature both followed diurnal cycles, however they were 12 h out of phase with each other, and that the amplitude of the diurnal cycle was reduced by covering the soil with residue.

The FFT analysis of NH₃ volatilization and CO₂–C emissions was based on 21 NH₃ volatilization measurements over 7 d and 240 CO₂–C measurements over 20 d, respectively. Because the FFT analysis requires equal time between the samples, the observed relationship between temperature and measured NH₃ volatilization values were used to populate the data set. The amplitudes and phase shifts of the dominant frequency were determined using the equation,

$$y(t) = A \cos \left( \frac{2\pi t}{T} - \phi \right)$$

where $T$ is the interval, $y(t)$ is the gas concentration at time $t$, $A$ is amplitude of the cosine curve, $\phi$ is phase angle of the cosine curve, and $c$ is the frequency of wave cycles (Carr, 1995; Chang et al., 2016b). The amplitude ($A$) represents the height of CO₂–C 24 h emission peak, whereas the phase angle or shift represents the peak offset. The phase angle was the minimum value in the diurnal cycle, whereas the shift + 1200 h was the maximum value. In this experiment, $T$ is 1 (a day in 24 h period) and $c$ is 1 (a complete cycle).

The total amount of CO₂–C and NH₃ emissions after 7 and 20 d were calculated. Based on these values, the CO₂–C or NH₃–N emissions from the soil, feces, and vegetation were determined based on following calculations:

a. Soil CO₂–C or NH₃–N emissions = Treatment 1,
b. Soil + grass CO₂–C or NH₃–N emissions = Treatment 2,
c. Vegetation CO₂–C or NH₃–N emissions = Treatment 2 – Treatment 1,
d. Suspended feces CO₂–C or NH₃–N emissions over soil = Treatment 3 – Treatment 1,
e. Soil-mixed feces CO₂–C or NH₃–N emissions over soil = Treatment 4 – Treatment 1,
f. Suspended feces CO₂–C or NH₃–N emissions over vegetation = Treatment 5 – Treatment 2,
g. Feces CO₂ or NH₃–N emissions applied over vegetation = Treatment 6 – Treatment 2.

The statistical analysis of the cosine amplitudes and phase shifts, as well as CO₂–C and NH₃–N emissions were conducted in PROC GLM in SAS (SAS Institute, 2008). In this analysis, blocks were random and the treatments were fixed. The $p$ value for calculated confidence intervals was $p = 0.10$. Correlation coefficients between the measured parameters were calculated.

![Fig. 1. Original and data shifted 6 h to the right in the top chart. Fourier transformation in bottom charts. These data indicate that the dominant frequency in the temporal data was one cycle per day. However, additional analysis showed that in a data set that was shifted 6 h, the calculated phase shift accounted for this shift.](image)

![Fig. 2. The relationship between time and natural log (ln) fecal C remaining. In this chart the slope is the first order rate constant and has the units $g (g \times day)^{-1}$. The open circles are the grass + fecal material and the filled circles are the soil + fecal material treatment.](image)
Determining Feces-Carbon First-Order Mineralization Rate Constants

The fecal-C first-order rate constants were the absolute value of the slope between the time in days \((x)\) and the natural log of the fecal C remaining \([\text{fecal C at time zero} - \text{fecal-C CO}_2–C\) emissions] at 0, 7, and 20 d (Mamani-Pati et al., 2010; Chang et al., 2016a). The first-order rate constants for soil-mixed feces and feces applied over vegetation for each block were computed (Fig. 2). These rate constants were used to estimate the amount of fecal-C that remained using the equation, \(\text{fecal remaining} = \text{fecal initial} \times \exp\left(-kt\right)\), where \(k\) is the rate constant. Twenty day area adjusted \(\text{CO}_2–C\) emissions were calculated for the treatments where feces were lightly mixed into the soil or applied over vegetation. For the feces that was lightly mixed with the soil, the 20 d area-corrected \(\text{CO}_2–C\) emissions were calculated by combining \(\text{CO}_2–C\) emissions from the soil (Treatment 1) and the soil + mixed feces (Treatment 4). The \(\text{CO}_2–C\) losses from bare soil (Treatment 1) were calculated by combining the losses from Days 1 through 7 with Days 8 through 20. For example, kg C loss ha\(^{-1}\) in bare soil treatment (Treatment 1) was equal to \(7 \times 3.05 \text{ g (m}^2\times\text{d})^{-1} + 13 \times 3.54 \text{ g (m}^2\times\text{d})^{-1} 	imes 10,000 \text{ m}^2\text{ha}^{-1} \times \frac{1000 \text{ g}}{1 \text{ kg}} = 674 \text{ kg CO}_2–C\) ha\(^{-1}\). The \(\text{CO}_2\) from areas where the feces was lightly mixed with the soil was based on an estimated fecal deposition. This value was based on a forage digestibility value of 560 g kg\(^{-1}\), a livestock consumption rate of 1460 kg biomass (ha × year)\(^{-1}\) which resulted in an emission of \(\text{CO}_2\) from feces \([7.53 \text{ g CO}_2–C (m}^2\times\text{d})^{-1}\) treatment. Differences in the \(\text{CO}_2\) emissions between the mixed soil and vegetation treatments were attributed to several factors including plant respiration and/or that the plant stimulated soil organic matter mineralization (Phillips et al., 2010).

Similar fecal-C \(\text{CO}_2–C\) emissions were observed for the first 7 d when they were suspended over soil \([9.6 \text{ g C (m}^2\times\text{d})^{-1}\) or vegetation \([10.4 \text{ g C (m}^2\times\text{d})^{-1}\). When the feces were applied and partially mixed into the soil, \(\text{CO}_2\) emissions (Treatment 4 – Treatment 1) increased 59% when compared with the mixed soil without feces. This increase is attributed to the fecal materials stimulating heterotrophic respiration.

RESULTS AND DISCUSSION

Carbon Dioxide Emission

Air temperatures and \(\text{CO}_2–C\) emissions followed a diurnal cycle that had maximum values between 1500 and 1800 h of the day and minimum values between 300 and 600 h of the day (Table 1, Fig. 3). Similar \(\text{CO}_2–C\) emissions and soil temperatures phase shifts were attributed to the impact of temperature on microbial activity and that \(\text{CO}_2\) solubility decreases with increasing temperature (Chang et al., 2016a). During the first 7 d, \(\text{CO}_2–C\) emissions were almost 50% less in the lightly mixed soil \([3.05 \text{ g CO}_2–C (m}^2\times\text{d})^{-1}\] than the clipped vegetation \([7.53 \text{ g CO}_2–C (m}^2\times\text{d})^{-1}\) treatment. Differences in the \(\text{CO}_2\) emissions between the mixed soil and vegetation treatments were attributed to the apparent stimulations of plant respiration and/or that the plant stimulated soil organic matter mineralization (Phillips et al., 2010).

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Table 1. The influence of soil, vegetation, suspended (sus.) fecal, and feces applied over vegetation or where the soil is lightly mixed to simulating cattle traffic on the amplitude (amp, A) and phase shift (\(\Delta\phi\)) of the diurnal cycle of CO2 loss [g CO2–C (m2 × d)] from cow fecal materials. Relative loss is the difference between CO2–C loss Treatments 3, 4, 5, and 6 and the appropriate controls (Treatments 1 and 2). The phase shift plus 12 h represents the time of maximum temperature. The amplitude represents the height of the diurnal cycle.

| Treatment no. | Treatments | 1–7 d | 8–20 d |
|---------------|------------|-------|--------|
|               |            | Amp.  | Phase shift | CO2–C loss | Relative loss | Amp.  | Phase shift | CO2–C loss | Relative loss |
|               |            | g m2  | hour       | g (m2 × day)\(^{-1}\) | g (m2 × day)\(^{-1}\) | g m2  | hour       | g (m2 × day)\(^{-1}\) | g (m2 × day)\(^{-1}\) |
| 1             | Lightly mixed soil | 0.053c\(†\) | 3.19 | 3.05e | 0.060b | 3.71 | 3.54d |
| 2             | Vegetation  | 0.055c | 3.47 | 7.53d | 0.080c | 3.43 | 9.11c |
| 3             | Lightly mixed soil + suspended feces | 0.483a | 4.09 | 12.6c | 0.320a | 3.97 | 9.02c | 5.48a |
| 4             | Lightly mixed soil with feces | 0.429b | 4.29 | 16.7ab | 0.137b | 4.11 | 10.3bc | 6.79a |
| 5             | Veg. + suspended feces | 0.509a | 4.25 | 18.0a | 0.342a | 4.19 | 14.4a | 5.25a |
| 6             | Veg. + feces | 0.342b | 4.35 | 16.1b | 0.089b | 4.27 | 11.0b | 1.89b |
| p             | <0.0001 | 0.133 | <0.0001 | 0.071 | 0.004 | 0.247 | <0.0001 | 0.03 |
| LSD(0.1)      | 0.085 | ns\(‡\) | 1.36 | 3.19 | 0.109 | ns | 1.38 | 0.03 |

Air temp. 7.17 4.07 7.54 4.08

\(†\) The same letters within a column are not significantly different \((p = 0.10)\).
\(‡\) ns: nonsignificant.
between the average temperatures and first-order rate constants is similar to the findings of Clay et al. (2010, 2012), and is attributed to soil temperature diurnal variability (Fig. 2). When the feces were applied over vegetation, the feces-C mineralization rate constants were not correlated to either soil water or air temperature. These results were attributed to the feces not being mixed into the soil. To assess the repeatability of the measurement system, CO2–C emissions of feces suspended over bare soil and vegetation were compared. For this time period, the CO2–C emission rates were similar and the difference between these two treatments represented 4.3% of the total CO2–C emitted.

Comparison Between Near Continuous and Point Carbon Dioxide-Carbon Emissions Measurements

In this experiment, gas samples are collected and analyzed on near continuous basis. However, to reduce the cost associated gas sample collection and analysis, Parkin and Venterea (2010) recommend that the samples be collected at a time that corresponds to the average temperature and where possible these points should be as close together as possible. Based on these recommendations, numerous studies have been conducted where point greenhouse gas emissions are measured at regular time intervals over the study. For example, Hamido et al. (2016) measured CO2–C emissions weekly from 1200 to 1400 h, whereas Nykanen et al. (1995) did not identify when the samples were collected. Generally, total emissions are determined by using linear interpolation across sampling times.

Based on the FFT, the peak temperatures occurred at about 1600 h (1200 h + 400 h phase sift). Based on the measured temperatures in Fig. 3, the average temperature occurred at 1019 ± 0.93 h. A comparison between the CO2–C emissions at 1100 h and near continuous measurement showed that the two measurements were highly correlated ($r^2 = 0.99^{**}$), however they predicted different emissions. Point samples when collected daily at 1100 h, every 2 d, and every 3 d, when averaged across blocks and the four treatments (1, 2, 4, and 6) had emissions of 196, 206, and 200 g CO2–C (m² × 20 d)⁻¹, respectively. In all cases, these values were 5 to 10% greater than the near continuous measurement of 186 g CO2 (m² × 20 d)⁻¹. In addition, sampling every 2 d had different results than sampling every day, and delaying sample collection from 1100 to 1300 h in the soil + manure treatment between Days 1 and 2 (Fig. 1) would have increased emission 62% [from 7.53 to 12.2 g CO2–C (m² × hour)⁻¹]. This assessment suggests that point measurement can be used to provide qualitative emissions. However, if the samples are not collected at the average temperature, they may not be accurate.

Ammonia-Nitrogen Volatilization from Cow Fecal Materials

In northern Great Plains rangeland systems, the primary sources of N are atmospheric deposition, N₂ fixation by legumes, and feces-N and urine depositions from animals. Because little N fertilizer is applied to these systems, their long-term productivity relies on minimizing N losses (Vlassak et al., 1973; Reeder and Schuman, 2002; Köchy and Wilson, 2001; Fornara and Tilman, 2012; Keuter et al., 2014).

Ammonia loss from the feces followed a diurnal cycle with peak values occurring at 1400 h (Fig. 2). These results are in agreement with Sherlock and Goh (1985) and Clay et al. (1990) who reported that NH₃ peaks matched temperature peaks. This diurnal cycle was attributed to the temperature dependence of microbial activity and decreasing NH₃ solubility with increasing temperature. Decreased NH₃ volatilization when mixed with the soil was expected, and even though volatilization was numerically lower when mixed, it was not significant (Table 2).

The total amount of volatilized NH₃–N in the non-feces treatments (Treatments 1 and 2) for the first 7 d was 0.36 g NH₃–N m⁻². When feces was applied (Treatments, 3, 4, 5, and 6), the total loss over 7 d was 0.49 g NH₃–N m⁻² (Table 2). Based on the difference between the treated and untreated soil, approximately 20% of the fecal NH₄–N was volatilized. These values are higher than the 3.9% loss reported by Fischer et al. (2016). Differences between Fischer et al. (2016) and our results, are attributed to Fischer et al. (2016) making a comparison with total N, whereas we only considered NH₃–N in the feces. Laubach et al. (2013) used a micrometeorotical technique to measure NH₃ volatilization above a small paddock containing both feces and urine patches. In Laubach et al. (2013), NH₃ volatilization was measured at 5 m heights above the soil surface.
Volume with and without feces may explain why previous studies have
area corrected 20 d CO2–C emissions from bare soil (676 kg
tration (Conant and Paustian, 2002; Yuan and Hou, 2015). The
feces C ranged between 33 and 71 kg C ha–1.
emission 7.6%. The 90% confidence interval for mineralized
amount of feces-C emitted was 25 kg CO-C and the 90% con-
idence interval for the mineralized feces-C was between 6.3
based on temporal and spatial variability, they reported that
11.6% of the dung N was volatilized. However, they did not
provide treatments where NH3 from soil, feces, and urine could be
separated and they did not report the efficiency of the col-
lection system. Laubach et al. (2013) value of 11.6% was much
higher than the 3.9% reported by Fischer et al. (2016). Similarly,
Lee et al. (2011) in a laboratory study had slightly lower NH3
volatilization which ranged from 1 to 13%. In our study, 20% 
NH3–N volatilization loss is similar to the losses reported for 
urea (Clay et al., 1990) and simulated urine (Sherlock and Goh, 1985) and lower than the losses reported for surface-applied
manure (Stevens and Laughlin, 1997; Lee et al., 2011; Hristov et
al., 2011).

Calculating the Potential Impact of Feces on Whole Paddock Carbon Dioxide Emissions

Area corrected CO2–C emissions for the lightly mixed soil and for the lightly mixed soil plus feces were 674 and 727 kg 
CO2–C ha–1, respectively. These calculations suggest that
52 kg feces-C ha–1, or 19% of the applied feces-C was respired 
over 20 d, and that the feces deposition increased total CO2–C 
emission 7.6%. The 90% confidence interval for mineralized 
feces-C ranged between 33 and 71 kg C ha–1.

When the feces were deposited over the clipped vegetation, 
slightly different results were obtained. In the clipped vegeta-
tion, 9.3% of the feces-C was emitted and CO2–C emissions 
increased from 1711 kg CO2–C ha–1 in area without feces to
1736 kg CO2–C ha–1 in areas with feces. By difference, the
amount of feces-C emitted was 25 kg CO-C and the 90% confi-
dence interval for the mineralized feces-C was between 6.3
and 39 kg C ha–1. The small differences between the grassland 
with and without feces may explain why previous studies have 
reported that grazing can produce a mixed impact on C seques-
tration (Conant and Paustian, 2002; Yuan and Hou, 2015). The
area corrected 20 d CO2–C emissions from bare soil (676 kg 
CO2–C ha–1) were much lower than areas with only vegetation 
(1711 kg CO2–C ha–1).

SUMMARY

In the northern Great Plains, farmers and ranchers are inter-
ested in conducting research on techniques to increase their soil 
C levels. This paper demonstrated an approach to assess precision
conservation treatments at targeted locations. In addition, the 
research compared total CO2–C emissions over 20 d using near
continuous measurements with point measurements collected 
at 1100 h every day, every 2 d, and every 3 d. This comparison 
showed that the two methods were highly correlated, however
point measurements over estimated total emissions. These find-
ings suggest that targeted point sampling for greenhouse gases 
can contain substantial uncertainty.

The temporal data was converted to the frequency domain 
using the FFT. This analysis confirmed that temperature, NH 
volatilization, and CO2–C emissions followed a diurnal cycle 
and that differences in the phases were not detected. If the mea-
surements would have been collected over a several years, FFT 
could have been used to separate the seasonal and diurnal cycles.

In situ measurements of CO2 emissions showed that manage-
ment can influence CO2–C emissions and that mixing feces 
with soils increased CO2 emissions. The first-order fecal-C 
mineralization rate constants and 90% confidence intervals for 
the feces mixed with soil and for the feces applied over vegeta-
tion were 0.0109 ± 0.0043 g (g×d) –1 and 0.00454 ± 0.00336 g 
(g×d) –1, respectively. The rate constants and digestibility values 
were used to calculate area corrected CO2–C emissions. The area 
corrected 20-d CO2–C emissions for the simulated trampled 
soil and for the feces that was simulated to be trampled into the 
soil were 674 and 726 kg CO2–C ha–1, respectively. These values 
indicate that in bare soil, there was a 7% difference between the soil and soil plus feces treatment, and that of the 270 kg of feces-C 
added, the 90% confidence interval for mineralized feces-C 
ranged between 33 and 71 kg C ha–1. In range systems, highly 
trampled bare soil is often found near shade and food and water
sources. In the vegetation treatment, there was a 1.4% difference 
between the vegetation (1711 kg CO2–C ha–1) and vegetation 
plus feces (1736 kg CO2–C ha–1). These calculations show that 
accurate accounting requires the measurement or estimation of the 
feces deposition rate. Once the locations and amounts are 
determined, techniques discussed in this paper can be used to 
calculate NH3–N and CO2–C emissions.

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Table 2. Total NH3–N loss (g NH3–N m–2) over 7 d and per-
centage of loss relative to the controls (Treatments 1 and 2) and 
initial amount of NH3 contained in the feces.

| Treatments                              | NH3–N loss  | NH3–N loss |
|-----------------------------------------|-------------|------------|
| Lightly mixed soil                      | 355b†       |            |
| Vegetation                              | 364b        |            |
| Trampled soil + suspended feces         | 483a        | 19.4       |
| Soil trampled with feces                | 475a        | 18.1       |
| Veg. + suspended feces                  | 506a        | 23.0       |
| Vegetation + feces                      | 488a        | 20.9       |
| p                                       | 0.005       | ns†        |
| LSD(0.1)                                | 0.072       |            |

† The same letters in parentheses that represent they are not significantly different at the 10% level.
‡ ns: nonsignificant.
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