



Nitric oxide emissions from soils amended with municipal waste biosolids

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Abstract

Land spreading nitrogen-rich municipal waste biosolids (NO_3^- -N < 256 mg N kg⁻¹ dry weight, NH_3 -N ~ 23,080 mg N kg⁻¹ dry weight, Total Kjeldahl N ~ 41,700 mg N kg⁻¹ dry weight) to human food and non-food chain land is a practice followed throughout the US. This practice may lead to the recovery and utilization of the nitrogen by vegetation, but it may also lead to emissions of biogenic nitric oxide (NO), which may enhance ozone pollution in the lower levels of the troposphere. Recent global estimates of biogenic NO emissions from soils are cited in the literature, which are based on field measurements of NO emissions from various agricultural and non-agricultural fields. However, biogenic emissions of NO from soils amended with biosolids are lacking. Utilizing a state-of-the-art mobile laboratory and a dynamic flow-through chamber system, in-situ concentrations of nitric oxide (NO) were measured during the spring/summer of 1999 and winter/spring of 2000 from an agricultural soil which is routinely amended with municipal waste biosolids. The average NO flux for the late spring/summer time period (10 June 1999–5 August 1999) was $69.4 \pm 34.9 \text{ ng N m}^{-2} \text{ s}^{-1}$. Biosolids were applied during September 1999 and the field site was sampled again during winter/spring 2000 (28 February 2000–9 March 2000), during which the average flux was $3.6 \pm 1.7 \text{ ng N m}^{-2} \text{ s}^{-1}$. The same field site was sampled again in late spring (2–9 June 2000) and the average flux was $64.8 \pm 41.0 \text{ ng N m}^{-2} \text{ s}^{-1}$. An observationally based model, developed as part of this study, found that summer accounted for 60% of the yearly emission while fall, winter and spring accounted for 20%, 4% and 16% respectively. Field experiments were conducted which indicated that the application of biosolids increases the emissions of NO and that techniques to estimate biogenic NO emissions would, on a yearly average, underestimate the NO flux from this field by a factor of 26. Soil temperature and % water filled pore space (%WFPS) were observed to be significant variables for predicting NO emissions, however %WFPS was found to be most significant during high soil temperature conditions. In the range of pH values found at this site (5.8 ± 0.3), pH was not observed to be a significant parameter in predicting NO emissions. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

NO plays an important role in tropospheric photochemistry. Currently, the only known pathway for the production of ozone in the troposphere is the photolysis of NO_2 ($\text{NO}_2 \rightarrow \text{NO} + \text{O}(^3\text{P})$), which further reacts with O_2 to produce ozone (O_3) by the reaction $\text{O}(^3\text{P}) + \text{O}_2 \rightarrow \text{O}_3$. Therefore, increasing NO emissions, in the presence

of hydrocarbons and sunlight, are thought to be the cause of increased regional levels of tropospheric ozone and other photochemical oxidants (Logan, 1983). Yienger and Levy (1995) developed an empirically based model to estimate soil NO_x emissions on a global scale. They have reported that anthropogenic land use is having a significant impact on global soil NO_x emissions and that soil emissions can account for up to 75% of the total NO_x budget depending on location and time of year.

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Although there have been many experiments conducted that have measured NO emissions from various soil types (Johansson and Granat, 1984; Kaplan et al., 1988; Aneja et al., 1995; Williams and Fehsenfeld, 1991; Kim et al., 1994; Roelle et al., 2001), relatively few have included intensively managed agricultural soils, or continued measurements for substantial periods of time (Anderson and Levine, 1987; Sullivan et al., 1996). In the Southeast US, which is NO_x limited, an increase in NO_x emissions is believed to produce a corresponding increase in O₃ levels (Southern Oxidants Study, 1993). O₃ negatively affects human health, as well as ecological systems, such as crop yield. Studies show that prolonged exposure to high ozone levels causes persistent functional changes in the gas exchange region of the lungs. Additionally, ozone plays a critical role in controlling the chemical lifetimes and the reaction products of many atmospheric species (National Research Council, 1991). Gaseous nitric acid (HNO₃), the end product of NO reactions in the atmosphere, combines with either aerosols or water in the atmosphere, and is removed via rain, snow, or other deposition processes, as acidic deposition.

Many current air quality models derive their biogenic NO input data from emissions inventory systems which are often based on a temperature and land use algorithm. While many studies have been conducted on fallow soils, forested soils, grassland, golf course soils, agricultural soils, etc., there are comparatively fewer studies on emissions from biosolid amended soils and therefore no land use type for this category currently exists in the emissions inventory models. In the US alone, more than 6 million dry metric tons of biosolids are generated annually, and it is estimated that 41% of these biosolids are applied to the soil for their nutrient content (US EPA Report, 1999; Peirce and Aneja, 2000). Given that there is a lack of field data from soils amended with biosolids may indicate a possible error in the estimates of NO.

2. Methods and materials

2.1. Biosolids

The biosolids applied in this field study were from a wastewater treatment facility located in North Carolina (NC). The population which this facility serves is approximately 60,000 people and has a flow of wastewater through the plant of approximately $30.3 \times 10^6 \text{ l d}^{-1}$. The wastewater treatment facility consists of: preliminary treatment, primary treatment, secondary treatment, disinfection and sludge treatment and disposal. Preliminary treatment is the part of the process where the wastewater is first screened for large objects via mechanical filters and then enters grit

chambers where sand and inorganic solids settle out. During primary treatment, the heavier solids, which have settled to the bottom, and the lighter solids which have risen to the top, are removed from the tanks by skimming/scraping processes. The wastewater emitted from the primary treatment then enters the secondary treatment. During this step, the liquid is aerated and microbial biomass is introduced to induce nitrification. At the conclusion of the secondary treatment, the liquid is treated with chlorine to remove any infectious organisms and then the water is released to a local stream. The heavier and lighter solids combined together, called biosolids or sludge, are then sent to the treatment part of the facility where they are prepared for land application. After treatment, the biosolids are transported via tanker trucks, to facilities where they are stored until they can be applied to various fields.

2.2. Instrumentation and flux calculation

A Thermo Environmental Instruments Incorporated (TECO) Model 42S, chemiluminescence low level NO analyzer was used in conjunction with a dynamic flow-through chamber to measure NO concentrations (Thermo Environmental Instruments, Inc., 1992). The chamber design, associated mass balance equation and calibration procedures are described in full in Roelle et al. (1999, 2001). All analytical instruments and data collection systems were housed in a mobile laboratory, which was temperature controlled to within the operating range of the instruments.

2.3. Physiographic location and sampling scheme

NO concentration measurements were made on a property which is owned by a municipal water and sewer authority located in central NC. Through a cooperative agreement, a local farmer cultivates the field and the water/sewer authority applies nutrients in the form of biosolids and limes as necessary. The crop grown during our measurement campaign was a small grain including Kenland Red Clover and Hallmark Orchard Grass, which was planted in fall 1998 and amended with biosolids in January 1999 with approximately 80 kg N ha⁻¹. (Note: this year's fertilizer data was not available and these values are from fertilizer records of the preceding year.) The measurements at this field site occurred 10 June 1999–5 August 1999, 28 February 2000–9 March 2000 and 2–9 June 2000. The field site was mowed July 27, limed at the end of August and then received another application of biosolids in mid September (~80 kg N ha⁻¹). Daily averaged NO flux values and the seasonally averaged NO flux calculated for the three measurement periods can be seen in Fig. 1.

The daily sampling scheme consisted of measuring concentrations of NO after the sample exited the

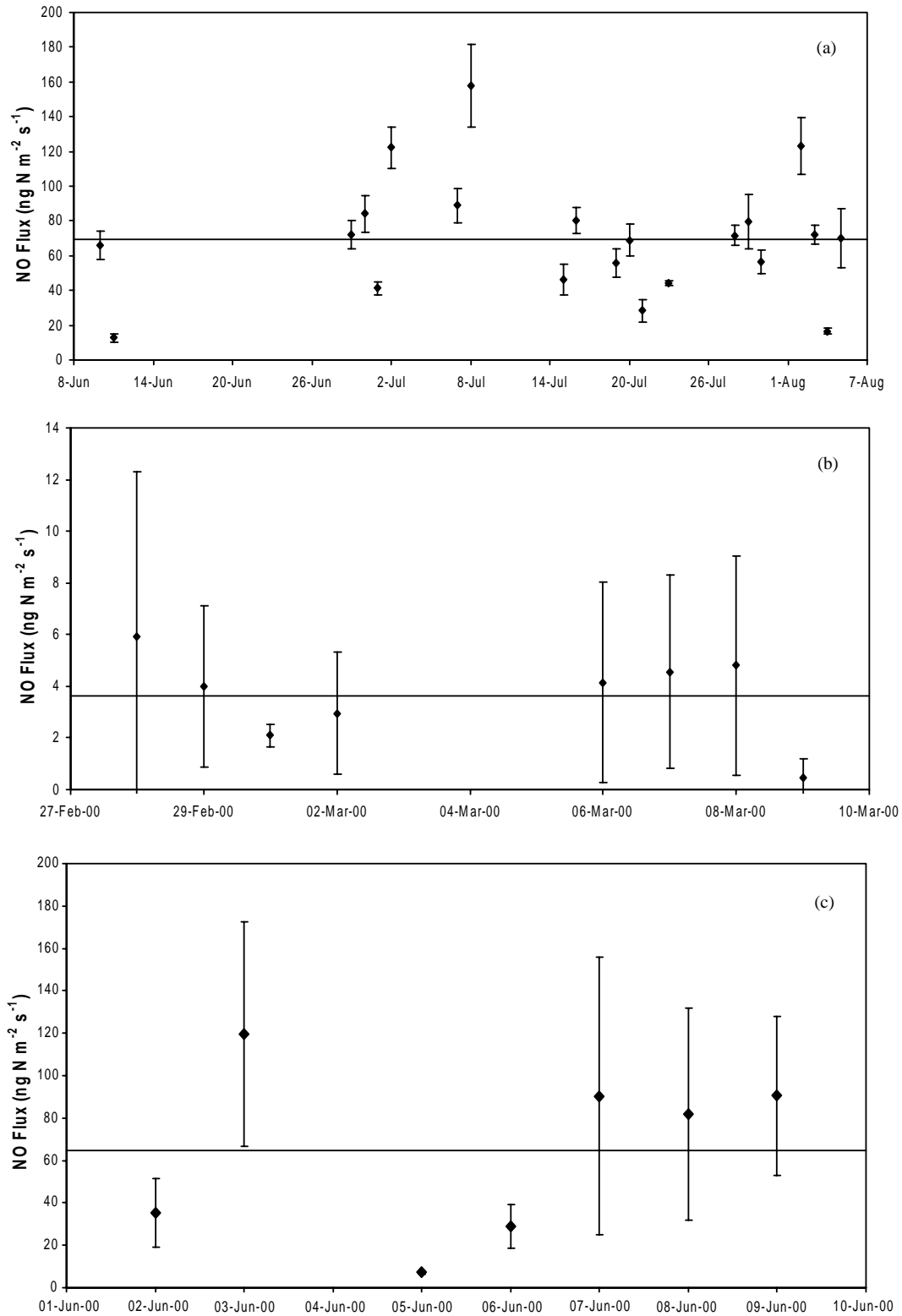


Fig. 1. NO flux versus day of experiment for summer 1999 (a), winter (b) and spring 2000 (c). Vertical bars represent one standard deviation of the NO emissions and the solid horizontal line represents the average emissions for the respective seasons.

Table 1
Characteristics of grab samples of soil at the research site and of the biosolids which are applied to the field^a

Soil, grab sample		Biosolids, grab sample	
Parameter	Quantity	Parameter	Quantity
Ammonia nitrogen (NH ₃ -N)	<28.6 mg N kg ⁻¹ dry weight	Ammonia nitrogen (NH ₃ -N)	23,080 mg N kg ⁻¹ dry weight
Nitrate nitrogen (NO ₃ ⁻ -N)	<5.7 mg N kg ⁻¹ dry weight	Nitrate nitrogen (NO ₃ ⁻ -N)	<256 mg N kg ⁻¹ dry weight
Nitrite nitrogen (NO ₂ ⁻ -N)	<5.7 mg N kg ⁻¹ dry weight	Nitrite nitrogen (NO ₂ ⁻ -N)	<443 mg N kg ⁻¹ dry weight
Total Kjeldahl nitrogen	753 mg N kg ⁻¹ dry weight	Total Kjeldahl nitrogen	41,700 mg N kg ⁻¹ dry weight
pH	5.83	Phosphorus	37,350 mg N kg ⁻¹ dry weight
Soil class	Mineral	Percent solids	3.4%
Bulk density	1.27 g cm ⁻³		
Particle density	2.44 g cm ⁻³		

^a Total Kjeldahl nitrogen = organic N + NH₃-N + NH₄⁺-N.

dynamic flow-through chamber. A daily experiment consisted of placing the chamber on the stainless steel collar, which had been inserted into the soil the previous evening. The chamber was placed on the collar at approximately 8:00 AM and flushed with zero grade air for at least 1 h before data collection began at 9:00 AM. This technique ensured that the concentrations within the chamber reached steady state prior to any data acquisition and allowed for the instruments to undergo their daily calibrations. Daily experiments ended at approximately 5:00 PM and the stainless steel collar was relocated to a random location within a 10 m radius of the mobile laboratory, in preparation for the next days experiment. This procedure allowed a minimum of 16 h for any effect on soil NO flux, due to soil disturbances caused by the insertion of the stainless steel collar, to dissipate.

2.4. Soil analysis

A soil sample was taken from the center of the dynamic flow-through chamber footprint at the end of each measurement period (~ 1 sample per day) and analyzed for soil pH, % water filled pore space (%WFPS) and total Kjeldahl nitrogen (TKN) (organic N + NH₃-N + NH₄⁺-N) by the Duke University Department of Civil and Environmental Engineering, Burlington Research and the NC Biological and Agricultural Engineering Department. Samples were taken with a bucket auger which removed a soil core to a depth of approximately 20 cm. %WFPS is an expression of soil water content and is the percentage of pore spaces in the soil filled with water. The %WFPS is determined using the soil bulk density (the weight of the soil solids per unit volume of total soil) and soil particle density (average density of the soil particles) (Troeh and Thompson, 1993). %WFPS is a convenient expression to describe soil moisture because it accounts for the differing bulk and particle densities of soils and therefore allows for

the comparison of soil moisture from different soil types. The soil bulk density and particle density for the research site were found to be 1.27 g cm⁻³ and 2.44 g cm⁻³, respectively. Characteristics of soil and biosolid grab samples can be found in Table 1.

The mineral soils at this research site are classified in the soil survey as Lignum silty loam, which is described as a moderately well drained, slowly permeable soil on the uplands with slopes of 0–3% (Dunn, 1977). However, soil surveys generally cover broad areas, and a soil texture determination of our specific soil cores within the larger research site identified our soil samples as a sandy loam (Tabachow, 2000). Soil temperature was recorded every minute, and these values were binned and averaged every 15 min using a Campbell Scientific soil temperature probe (accuracy ± 3%) inserted 5 cm into the soil, adjacent to the chamber.

3. Results

3.1. Effects of soil nitrogen content and pH

The data plotted in Fig. 2(a–c) represent daily average NO flux values (0900–1700) versus the daily sampled nitrogen content as TKN, NH₃ and NO₃⁻ respectively. As indicated by the data in Fig. 2a, TKN is not found to be a significant variable in predicting NO emissions. However, Figs. 2b and c reveal that the addition of ammonia or nitrate to the soil produces a corresponding increase in NO emissions. These results are to be expected based on the predominant NO production pathways, namely nitrification and denitrification processes, and the fact that the organic-N must first be converted to inorganic forms prior to nitrification or denitrification processes to occur (Warneck, 1988; Troeh and Thompson, 1993). The fact that NO increases in response to both NH₃ and NO₃⁻ indicates that both nitrifiers and denitrifiers are present in the soil.

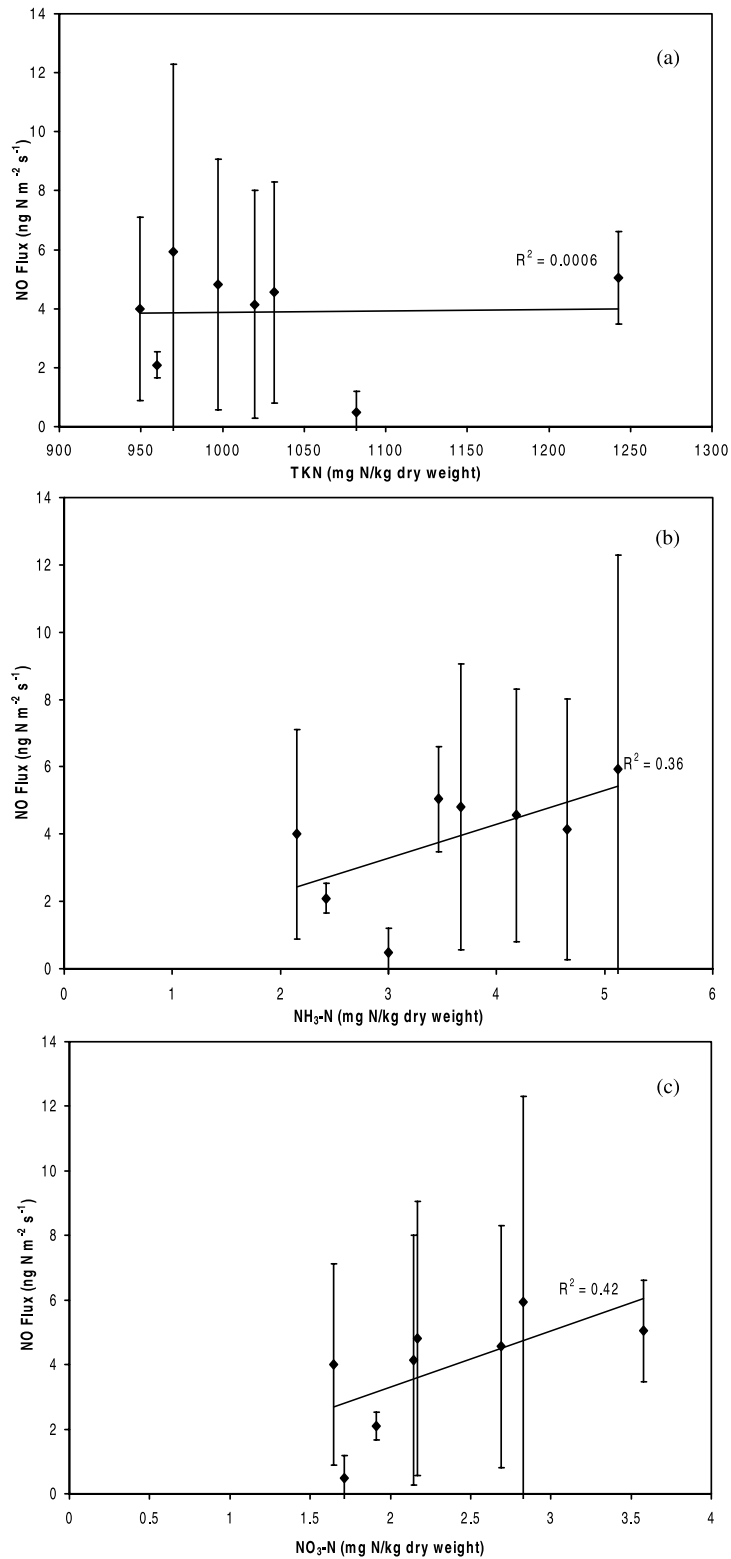


Fig. 2. (a–c). NO Flux (ng N m⁻² s⁻¹) plotted versus total Kjeldahl nitrogen (TKN = organic N + NH₃-N + NH₄⁺-N) (a), NH₃ (b), and NO₃⁻ (c). Vertical bars represent one standard deviation of the NO emissions.

This field site is typically amended with biosolids twice per year and the last application prior to the summer 1999 measurement campaign was in January 1999. In order to examine how biosolid applications immediately affect NO emissions, three experiments were conducted in which 1 served as a control and 2 experiments had biosolids applied to the soil. Previous experiments, both by the NC State University Air Quality Research Group and other researchers have shown that adjacent plots with seemingly homogeneous soils can differ in NO emissions by more than an order of magnitude (Williams and Fehsenfeld, 1991; Valente et al., 1995). In order to make the experimental plot as homogeneous as possible, a depression was dug (13" × 38") large enough to accommodate 2 chambers side-by-side, and deep enough to simulate the depth to which the soil is tilled. Each time an experiment was conducted on this plot, the soil was removed, thoroughly mixed and then returned where it was left undisturbed for 48 h prior to any measurements. On both biosolid amended experiments, the biosolids were collected directly from the purging spigot of the biosolid storage tank outlet which fills the tankers used to transport the biosolids to the field sites. The biosolids were applied to the experimental plots by pouring the biosolids, at an application rate of 80 kg N ha⁻¹, onto the soil surface 30 min prior to any measurements.

Fig. 3a represents the first experiment in which no biosolids were applied to either plot. The purpose of this experiment was to see if mixing the soil had, in fact, produced a fairly homogeneous environment. Although the graph does show a slight divergence between 12:00 and 2:00 PM, in general the NO emissions responded in very similar trends. For example, relative peaks appeared at both plots at approximately the same time (12:00, 2:30, 4:15). Given the fact that adjacent soils can differ by orders of magnitude, and that the largest difference between these two plots never exceeded 45%, we believed that we had created an environment similar enough to examine differences between two side-by-side chambers.

Fig. 3b represents the first of 2 experiments conducted on a plot amended with biosolids. The experiment started similarly to the unamended experiment (Fig. 3a) in that both plots began with fluxes of NO that were within 45% difference of each other. However, unlike Fig. 3a, the amended plot immediately began to increase while the unamended plot maintained a fairly steady profile. From the results of this one experiment, it appears as if the plot amended with biosolids did affect the NO emissions. Fig. 3c is a graph of the second experiment in which one of the plots was amended with biosolids. This experiment began with NO emissions at almost the same exact NO flux value. Similar to Fig. 3a, both plots follow very similar diurnal profiles, with relative minimum and maximum emissions occurring at

approximately the same time throughout the profile. The amended plot, however, appears to respond to the biosolids application with a greater amplitude in emissions than the unamended plot, especially in the late afternoon when the biosolids have had a chance to permeate into the top layer of the soil and come into contact with more of the bacteria which are presumed to be responsible for the production of NO.

Relationships between soil pH and NO flux in field studies have been investigated in the past and have proven difficult to discern due to the fact that pH values remain in a fairly tight range in intensively managed agricultural fields (Roelle et al., 1999). Average pH values for this measurement period were 5.8 ± 0.30 . Statistical analysis of the data measured during this campaign revealed no apparent relationship between NO flux and soil pH. These results are corroborated by the findings of both the Ormeci et al. (1999) and Matson et al. (1997) for similar ranges of pH. Ormeci et al. (1999) reported that NO emissions reach maximum values for near neutral to lower soil pH values. Under more acidic conditions, the chemical decomposition of NO₂ and HONO, otherwise known as chemodenitrification, has been suggested to be responsible for the increased availability of NO in the soil (Galbally, 1989). Controlled laboratory experiments, in which pH was varied while maintaining constant soil temperature and soil moisture resulted in highest NO emissions when soil pH was at its lowest value (4.3) (Ormeci et al., 1999). In an extensive field study conducted in the San Joaquin Valley, CA, Matson et al. (1997) found the highest NO emissions occurring for pH < 4.0. Both studies found NO emissions to be unaffected by changes in pH in the range between 5 and 8.

3.2. Soil temperature and soil water content

An important parameter controlling NO emissions, which has been studied by many investigators, is soil temperature (Williams et al., 1992; Kim et al., 1994; Sullivan et al., 1996; Roelle et al., 1999). Biochemical rates in the soil have been shown to rise exponentially with soil temperature in the range between 15°C and 35°C (Warneck, 1988). The relationship most cited in the literature, and which is also used in the Environmental Protection Agencies' (EPA) Biogenic Emissions Inventory System 2 (BEIS2), is the model proposed by Williams et al. (1992) which is given by:

$$\text{Flux}(\text{ng N m}^{-2} \text{ s}^{-1}) = A \text{Exp}(0.071 T_s), \quad (1)$$

where A is the experimentally derived coefficient associated with land use categories and T_s the soil temperature (°C).

In general, the emission factors (A) will be highest for those crops which receive the greatest application rates of nitrogen fertilizer (i.e., corn, cotton, wheat), as

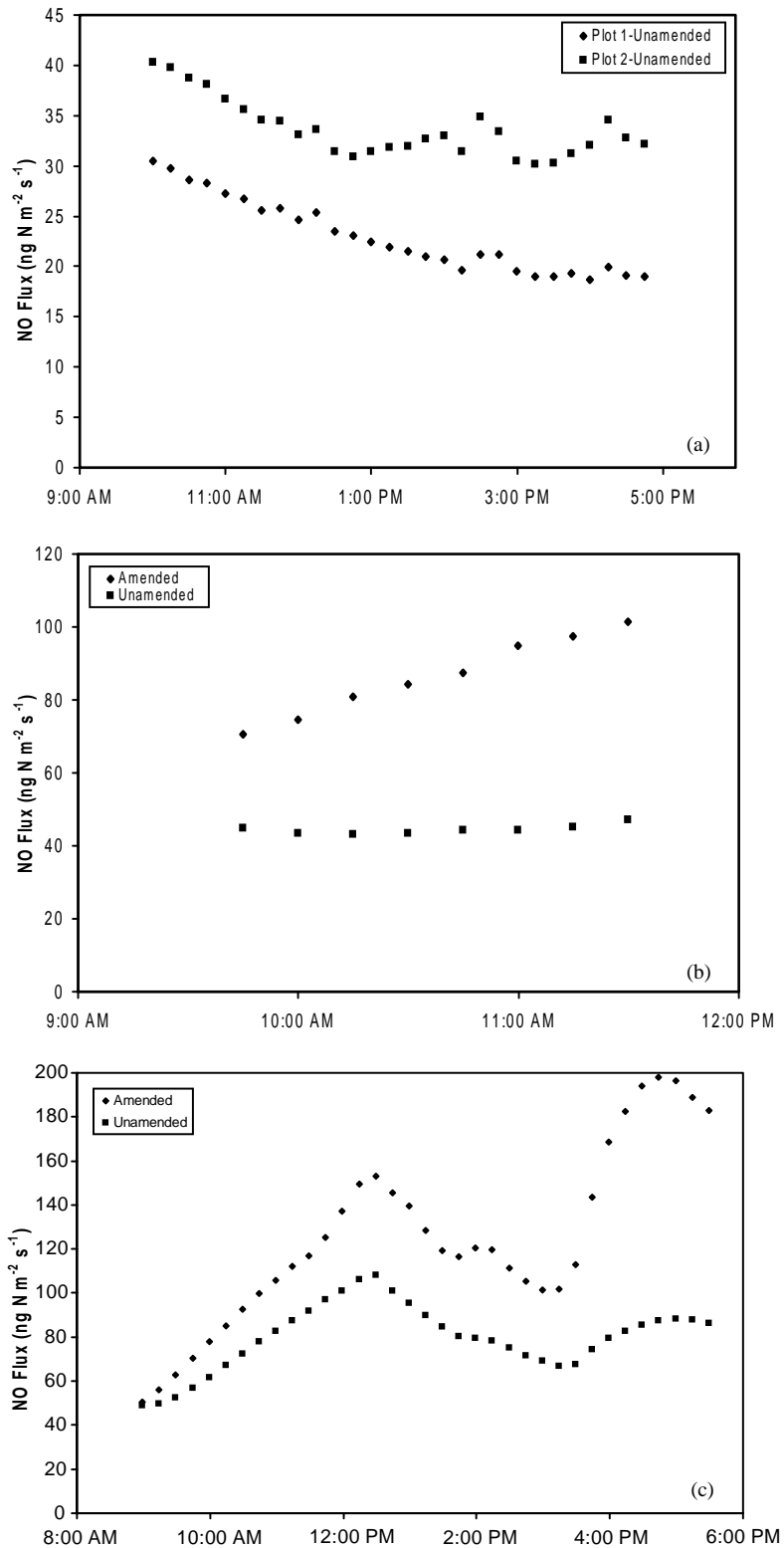


Fig. 3. (a–c). Plots of NO flux versus time of day on a controlled experimental plot. Neither plot was amended in (a), while both (b) and (c) had one plot which was amended at an application rate of 80 kg N ha⁻¹.

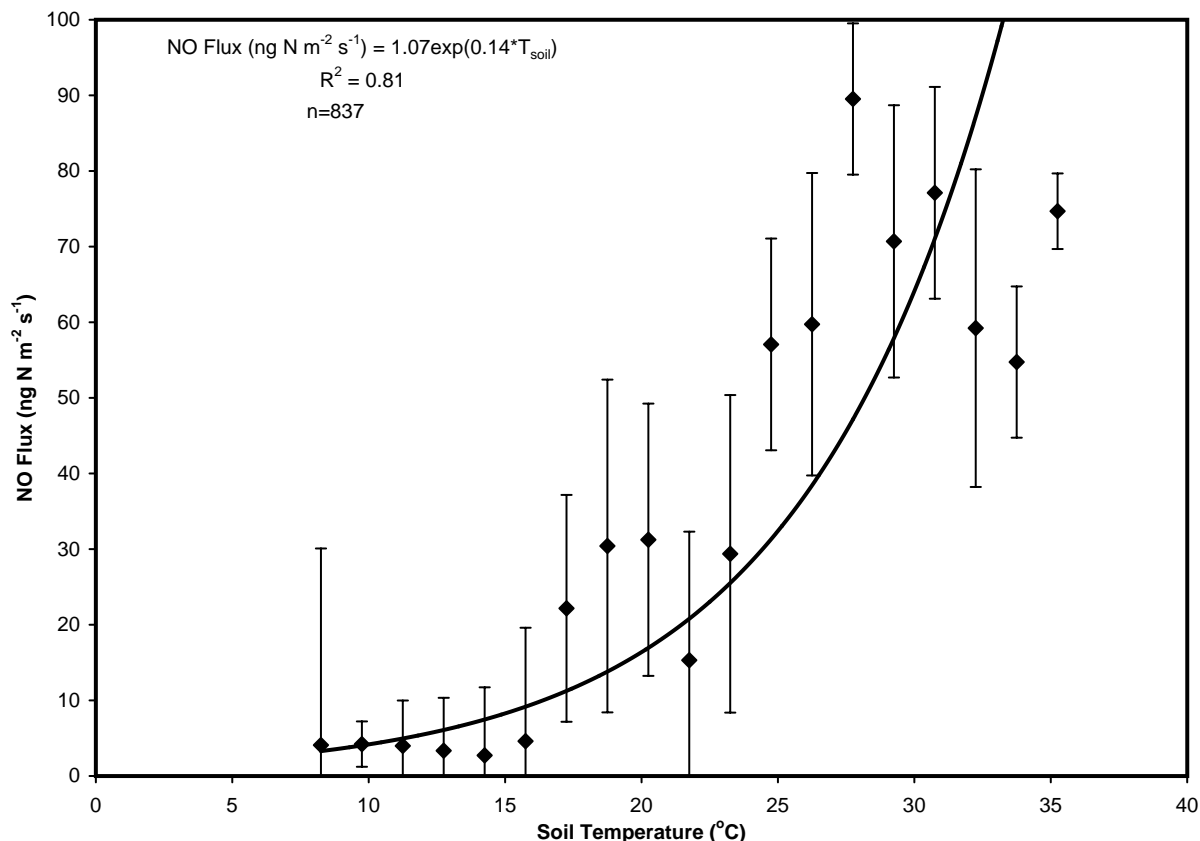


Fig. 4. NO flux plotted versus soil temperature. NO flux has been binned into 1.5°C temperature spans and the average flux for each temperature span is plotted. Vertical bars represent one standard deviation of the NO emissions.

increased N fertilization has been shown to increase NO emissions (Sullivan et al., 1996; Roelle et al., 1999).

In order to examine similar exponential relationships with this dataset, the protocol used by Thornton et al. (1997) was adopted. Soil temperatures were segregated into 1.5°C spans and the corresponding fluxes were averaged to produce one mean NO flux for each temperature span. The results of this procedure are plotted in Fig. 4, with the corresponding regression equation and R^2 values. Due to the strong dependence of NO flux on soil temperature, the highest emissions are found during the summer. Utilizing the regression equation in Fig. 4, and the air-to-soil temperature conversion factors described in Williams et al. (1992), we estimated that summer (June–August) accounts for 60% of the yearly emissions, while fall (September–November), winter (December–February), and spring (March–May) account for 20%, 4%, and 16% respectively. The use of the BEIS2 model would yield a different distribution of NO emissions of 44%, 24%, 10% and 22% for summer, fall, winter and spring, respectively. The distribution of the seasonal emissions

using these two approaches can be seen in Fig. 5, which also shows that the BEIS2 model could underestimate the yearly emissions by a factor of 26.

The relationship between emissions of NO and soil temperature discussed previously is further confounded by the dependence of NO emissions on soil moisture content. Researchers have found that nitrification is optimized for moisture contents between 30% and 65% WFPS (Davidson and Swank, 1986; Linn and Doran, 1984; Parton et al., 1988). Recent laboratory experiments conducted by Ormeci et al. (1999) found that the range for maximum NO emissions occurred between 20% and 45%.

In Fig. 6, NO emissions are binned into values of %WFPS of 25, 35 and 45 and soil temperatures of 15°C, 25°C, 35°C. NO flux follows a general trend of increasing emissions as %WFPS increases within a given soil temperature, except at 25°C. Likewise, within a given %WFPS, NO emissions increase as soil temperatures increase except at 35% WFPS. Soil temperature and %WFPS were both found to be statistically significant parameters for predicting NO

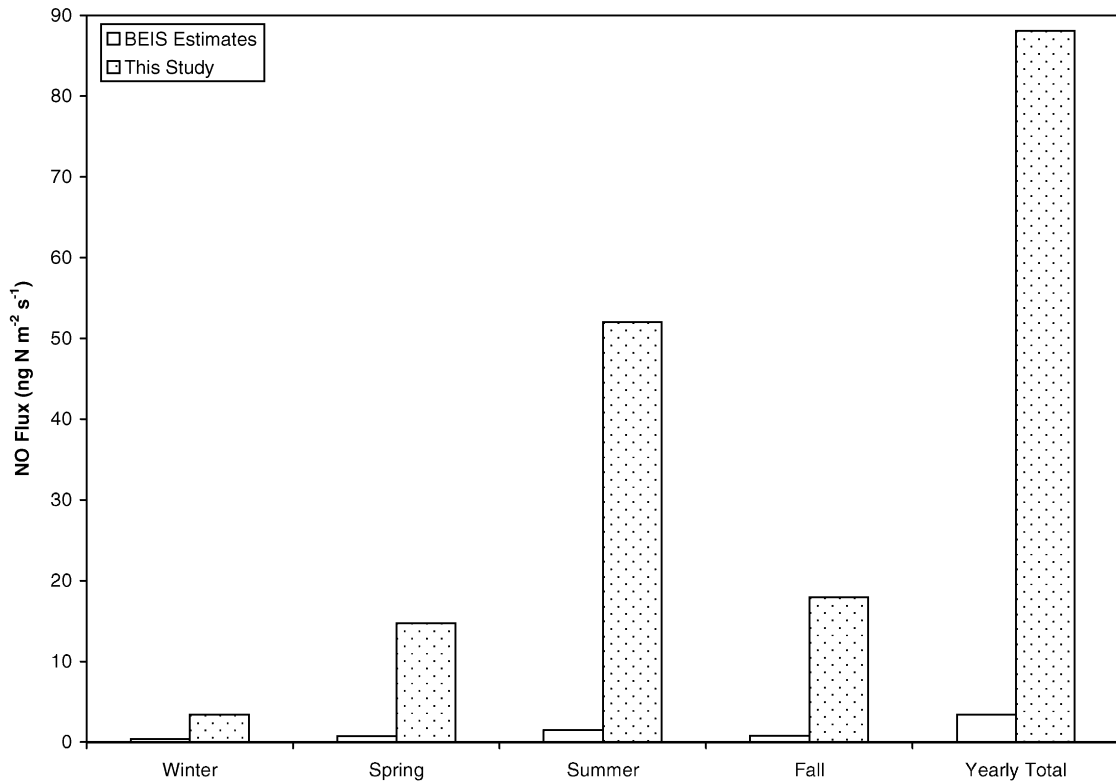


Fig. 5. Estimated NO flux at field site sampled throughout 1999–2000. Clear bar represents estimates using existing BEIS2 model. Stippled bar represents estimated NO flux using algorithm developed during this study.

emissions, however %WFPS was only significant during high ($T = 35^{\circ}\text{C}$; $R^2 = 0.46$) and low ($T = 15^{\circ}\text{C}$; $R^2 = 0.21$) soil temperatures. The highest average NO flux occurred at 45% WFPS and 35°C soil temperature ($119.3 \text{ ng N m}^{-2} \text{ s}^{-1}$), which is expected when considering the temperature dependence alone. However, relatively equivalent NO flux values occurring at 35% WFPS and 25°C ($114.3 \text{ ng N m}^{-2} \text{ s}^{-1}$) seems to indicate that these conditions may also be optimum for maximizing NO production from this biosolid amended field site.

4. Conclusions

NO emissions from fields amended with biosolids were studied during late spring/summer 1999 and winter and late spring 2000, in order to examine what environmental parameters might control these emissions. Soil temperature and %WFPS are both found to be significant parameters for predicting NO emissions, however %WFPS is only significant during high ($T = 35^{\circ}\text{C}$; $R^2 = 0.46$) and low ($T = 15^{\circ}\text{C}$; $R^2 = 0.21$) soil temperatures. When NO was modeled versus TKN and the inorganic components, NO emissions were

found to be dependent only on the inorganic N species. Further, at this field site nitrification and denitrification appeared to be occurring in the soil simultaneously. The average NO emissions from this small grain field were up to a factor of 26 higher than what the EPA's currently used biogenic emissions model would predict for similar crop types. This leads to the conclusion that NO emissions are potentially being underestimated for these biosolid amended soils. It should be noted that the data used to produce this revised NO emissions algorithm was from only one field site during several weeks throughout different seasons of the year. Future work should consist of additional studies from biosolid amended soils to verify the results presented here. The exponential temperature relationship reported here, however has been consistently reported in the literature and therefore provides some basis for this study to be extended throughout the year. It should not be assumed, however that this temperature dependence can be extended to all temperatures, as temperatures outside the range of $15\text{--}35^{\circ}\text{C}$ are often found to alter the oft-cited exponential relationship.

In NC, biosolids are applied to 42,000 acres annually (NCDENR, 1999). This practice serves as an economical means to dispose of the biosolids while saving

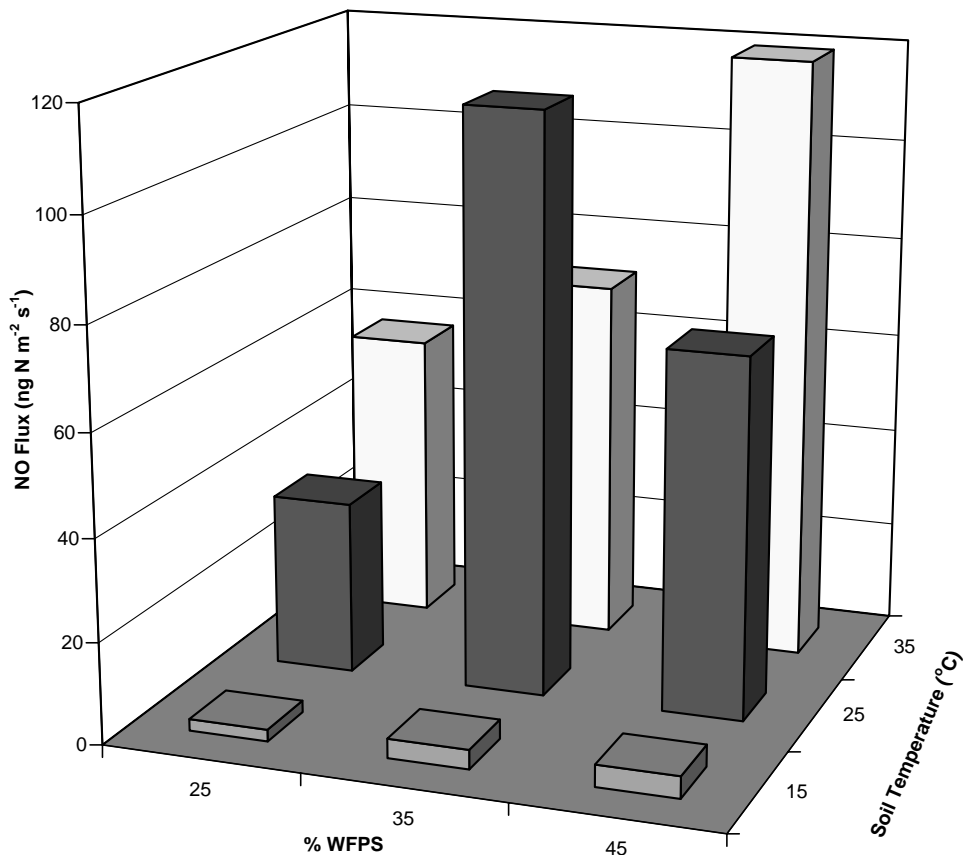


Fig. 6. NO flux plotted versus %WFPS and soil temperature. %WFPS has been binned into values of 25%, 35% and 45% and soil temperature has been binned into values of 15°C, 25°C and 35°C.

farmers the expense of purchasing chemically derived fertilizers. In NC, biosolid amended soils are applied in localized areas and represent a small fraction of total crop land soils (<1%), and therefore will likely represent only a small fraction of the total biogenic NO budget. However, these biosolid amended soils may act as significant sources of localized O₃ production, especially during the hot and stagnant periods of the summer when biogenic NO emissions and photochemical activity are at a maximum. Therefore future work should include a modeling study to examine the localized effects biosolid amended soils have on O₃ production.

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commercial products does not constitute endorsement or recommendation for use.

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