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Effects of nitrogen addition on dissolved N_2O and CO_2, dissolved organic matter, and inorganic nitrogen in soil solution under a temperate old-growth forest

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ABSTRACT

Soil solutions at 15 cm and 60 cm depths under a Korean pine and broadleaf mixed forest (>200 years old) at Changbai mountain, northeast China, were sampled using porous ceramic suction cups from July 2006 to October 2007, to study the effects of nitrogen (N) addition on the concentrations of dissolved nitrous oxide (N_2O) and carbon dioxide (CO_2), dissolved organic matter and inorganic N. The actual concentrations of dissolved N_2O and CO_2 in soil solutions could be obtained using a combination of continual three phase equilibrations and gas chromatography. The dissolved CO_2 concentrations in soil solutions at 15 cm and 60 cm depths varied from 4.3 to 15.5 and from 3.5 to 18.3 μg CO_2–C ml^{-1}, respectively, and dissolved N_2O concentrations at both depths varied from 1.8 to 34.9 and from 2.5 to 99.3 ng N_2O–N ml^{-1}, respectively. The addition of N sources such as $(NH_4)_2SO_4$, NH_4Cl and KNO_3 at a rate of 4.5 g N/m^2 each year tended to decrease concentrations of dissolved organic carbon (DOC) in soil solutions at 60 cm depth in 2007, which was contrary to the increase in dissolved CO_2 concentrations under N-fertilized forest plots. However, the N addition did not give an obvious effect on the concentrations of dissolved CO_2 and DOC in soil solutions at 15 cm depth. There was an increase in concentrations of NH_4^+–N, NO_3^-–N and dissolved organic nitrogen (DON) of soil solutions under N-fertilized forest plots. Rainfall after thawing in spring could promote the accumulation of dissolved N_2O in soil solutions at 15 cm and 60 cm depths, particularly at 60 cm depth under the $(NH_4)_2SO_4$ added plots, due to mineralization of DON and nitrification. All the tested properties of soil solutions at 15 cm and 60 cm depths were well correlated. Among these properties, the dissolved N_2O concentrations of soil solutions at both depths were better correlated with the DON concentrations at 60 cm depth, and the dissolved CO_2 concentrations at 15 cm depth with the DOC concentrations at both depths. Hence, both DOC and DON can contribute to the formation of dissolved CO_2 and N_2O in the soil solutions at varying depths under N-fertilized forest plots, respectively. Our observations strongly indicate that N inputs to temperate forest floors can affect the status of N and carbon processes in underlying forest soils.
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1. Introduction

Soil solution chemistry can be considered as a sensitive indicator of biogeochemical processes under forest stands, responding quickly to disturbances or stresses like nitrogen input (e.g. McDowell et al., 2004; Pregitzer et al., 2004; Michel et al., 2006). Due to the limitations of measurement, earlier few studies reported a large range of dissolved N_2O and CO_2 concentrations in soil solutions under various land uses (N_2O: 0.5–9984 ng N ml^{-1}; CO_2: 0.04–53.9 μg C ml^{-1}) (Heincke and Kaupenjohann, 1999; Jacinthe and Groffman, 2001; Sawamoto et al., 2002; Xiong et al., 2006). In addition, they less focused on the concentrations of both dissolved gases in forest soil solutions and the key mechanisms involved in their formation (Heincke and Kaupenjohann, 1999). This shortcoming is not beneficial to studying the sources of N_2O and CO_2 emissions from forest soils *in situ* and how these processes are affected by solution chemistry, especially when N inputs are considered. In addition, earlier studies usually used standard Henry's law constants for analysis of dissolved N_2O and CO_2 concentrations in soil solutions (Davidson and Firestone, 1988; Davidson and Swank, 1990), whereas the constants can be variable with the properties of solutions including salt content and ion intensity. Hence, it is important to study the dynamics of dissolved N_2O and dissolved CO_2 concentrations in soil solutions at various depths under different forest stands, using a suitable measurement method.

Nitrification in upper layers of forest soils and denitrification in deeper soil horizons may contribute to the accumulation of dissolved N_2O in soil solution, especially during thawing in spring (Heincke and Kaupenjohann, 1999; Teepe et al., 2000). The addition of different forms of N to temperate forest floors can usually increase N leaching as

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DON and inorganic N from forest topsoils (e.g. McDowell et al., 1998; Michalzik et al., 2001; Pregitzer et al., 2004), which has been hypothesized to affect the formation of dissolved N_2O in the soil solutions at various depths. Unfortunately, there is limited knowledge about the origin of dissolved N_2O in soil solutions under N-fertilized forest stands. The N inputs from forest topsoils may increase the activities of soil microorganisms and the mineralization of carbon (C) in underlying soils, hence releasing CO_2 into the soil solution. Taken together, the concentrations and interaction of dissolved CO_2, DOC and inorganic N in soil solutions at various depths can improve our understanding of carbon turnover in the underlying soils under N-fertilized forest stands.

Earlier studies usually reported the increase in the export of nitrogen as NO_3^-–N and DON from N-fertilized forest ecosystems, but there were many contrasting results regarding N effects on DON and DOC dynamics in soil solutions in field and laboratory studies (McDowell et al., 1998; Magill and Aber, 2000; McDowell et al., 2004; Pregitzer et al., 2004; Michel et al., 2006). Furthermore, these earlier studies mainly focused on the dynamics of DOC, DON and inorganic N concentrations in forest soil solutions sampled using zero-tension lysimeters rather than using suction cups. At present, there is limited knowledge about the concentrations of dissolved N_2O and CO_2, and their relationships with the concentrations of DOC, DON and inorganic N in soil solutions at various depths under N-fertilized forest stands.

In this study, soil solutions at 15 cm and 60 cm depths under a broadleaf and Korean pine mixed forest at the foot of Changbai mountain, northeast China, were sampled using porous ceramic suction cups from July 2006 to October 2007. The objectives of this present work were to 1) assess whether a combination of multiple phase equilibrations and gas chromatography is suitable for measurement of dissolved CO_2 and N_2O concentrations in soil solutions; and 2) to study the concentrations of dissolved N_2O and CO_2 in soil solutions at various depths after N addition, and their relationships with the concentrations of dissolved organic matter and inorganic N. The results would improve our understanding of N leaching and carbon processes in underlying forest soils due to the increase in atmospheric N inputs.

2. Materials and methods

2.1. Description of forest site and soil properties

Field experiments were located under the Korean pine and broadleaf mixed forest (*Pinus koraiensis* mainly mixed with hardwood trees such as *Tilia amurensis*, *Fraxinus mandsburica* and *Quercus mongolica*, >200 years old, altitude 738 m above sea level) near the National Research Station of Changbai Mountain Forestry Ecology (128°6′E, 42°24′N). The climate is temperate-continental climate, with a long-term cold winter and warm summer. The annual mean temperature is approximately 4.1 °C, and precipitation averages approximately 855 mm at the bottom of the mountain, with more than 80% of rainfall from May to August. The dark brown forest soil belongs to Andosols (Food and Agriculture Organization soil classification). The depth of litters and A-horizons is approximately 3–5 cm and 10 cm, respectively. The main properties of the soils at various depths under such forest stand were reported by Xu et al. (2007). Groundwater table levels under such forest stand were measured at a five-day or ten-day interval each month from May to October in 2006 and 2007, and moisture of litters on the ground was also measured at the end of each month.

2.2. Effects of N addition on soil solution chemistry under forest stand

Sixteen individual plots with 3 m × 3 m each were selected on the flatness under the mixed forest stand. Aqueous solutions of N sources such as $(NH_4)_2SO_4$, NH_4Cl and KNO_3 were respectively sprayed on the ground within four individual plots in four equal monthly doses (July–October) in 2006 and in five equal monthly doses (June–October) in 2007 at a rate of 4.5 g per m^2 each year, corresponding with 5.0 mm rainfall each; tap water was added only to the control. According to the depth of A-horizons and the distribution of tree roots in underlying soil, two sets of porous ceramic suction cups (3.1 cm in diameter and 7 cm in height) were installed at 15 cm and 60 cm depths, respectively, to collect soil solutions of organic layers and beyond root zones (Vandenbruwane et al., 2008). To eliminate the disturbance of soil, soil auger with a diameter of 3.3 cm was used to establish the holes down to 15 cm and 60 cm depths, respectively, and the suction cups connected to PVC tubes were fixed closely inside the holes. The pressure inside each tube within a week at water-filled pore space more than 70% or within 24 h after heavy rainfall was brought to approximately −70 kPa by a portable vacuum/pressure pump (Mityvac4010, Missouri, USA). Over a year from July 2006 to October 2007, soil solutions were sampled via the stopcock attached to each tube to avoid degassing, using 100-ml plastic syringes equipped with a stopcock, and the volume of solutions was measured simultaneously. Considering initial effects of installing the suction cups, the early two collections were discarded. These samples were rapidly transported to the laboratory and stored at 4 °C prior to analysis of solution chemistry. One subsample (50 ml) of some solutions after equilibrating at room temperature for 2 to 3 h was used to measure concentrations of dissolved N_2O and CO_2, as shown below, and then all samples were frozen for analysis of dissolved organic C, total N, NH_4^+–N and NO_3^-–N concentrations.

2.3. Measurement of dissolved N_2O and CO_2 concentrations in soil solution

The combination of multiple phase equilibrations and gas chromatography was used for analysis of dissolved N_2O and CO_2 concentrations in soil solutions in the laboratory. According to Sawamoto et al. (2002), the multiple phase equilibration procedure was adapted and could be connected closely to the injection of a gas chromatograph. Briefly, 50 ml of solutions after equilibrating at room temperature for 2 to 3 h was taken using 100-ml plastic syringes equipped with a stopcock, and another 50 ml of pure N_2 was injected into such syringe via a stopcock. Dinitrogen was used to extract dissolved N_2O and CO_2 from the solutions by shaking with hands for 5 min, and then the headspace gas after standing for another 5 min was completely sampled via a stopcock using another 100-ml syringe for analysis of N_2O and CO_2 concentrations. The equilibration procedure in quintic replicates was done to assess how many times for such phase equilibration would be suitable for calculating actual dissolved N_2O and CO_2 concentrations in the soil solution. Finally, this working approach was used to determine the concentrations of dissolved N_2O and CO_2 in soil solutions under N-fertilized and non-fertilized forest plots.

According to the methods reported by Wang and Wang (2003), concentrations of CO_2 and N_2O in the headspace gases were respectively quantified with a modified gas chromatograph (Agilent 5890, Franklin, USA) equipped with a flame ionization detector (FID) and with an electron capture detector (ECD). Both CO_2 and N_2O were separated by one stainless steel column (2 m length and 2.2 mm i.d.) that was packed with 50–80 mesh Porapack Q, afterwards hydrogen reduced CO_2 to CH_4 in a Nickel catalytic converter at 375 °C, and the CH_4 was detected by FID. The oven was operated at 55 °C, and the FID at 200 °C (ECD at 330 °C for N_2O), respectively, with N_2 as carrier gas at a flow rate of 20 cm^3 min^{-1}. The detector responses were calibrated using certified gas standards, which contained 4.0 ml l^{-1} CO_2 in N_2 and 0.37 μl l^{-1} N_2O in N_2, respectively.

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