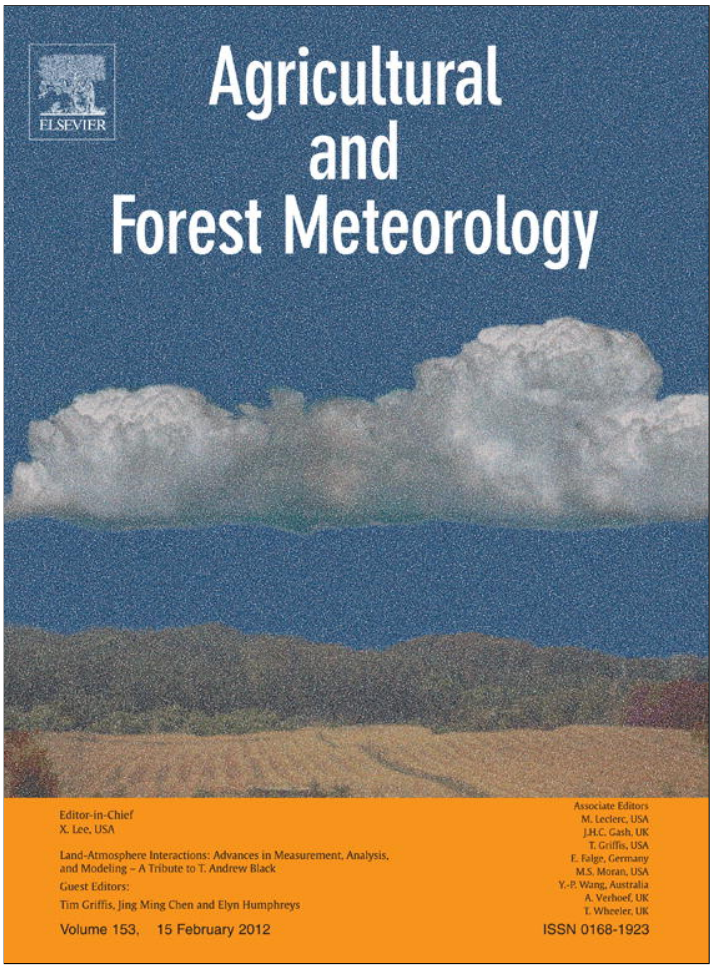

Agricultural and Forest Meteorology 153 (2012) 144–153

Contents lists available at SciVerse ScienceDirect

Agricultural and Forest Meteorology

journal homepage: www.elsevier.com/locate/agrformet

Interannual, seasonal, and diel variability in the carbon isotope composition of respiration in a C_3/C_4 agricultural ecosystem

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ARTICLE INFO

Article history:
Received 14 December 2010
Received in revised form 8 September 2011
Accepted 21 September 2011

Keywords:
Stable isotopes
Heterotrophic respiration
Autotrophic respiration
Automated chambers
Flux partitioning
Eddy covariance
Flux gradient
Tunable diode laser spectroscopy
Aboveground plant respiration

ABSTRACT

The stable carbon isotope ratio, $^{13}CO_2/^{12}CO_2$, is a valuable tracer for studying the processes controlling the autotrophic (F_{Ra}) and heterotrophic (F_{Rh}) contributions to ecosystem respiration (F_R) and the influence of photosynthesis on F_R. There is increasing interest in quantifying the temporal variability of the carbon isotope composition of ecosystem respiration (δ_R) because it contains information about the sources contributing to respiration and is an important parameter used for partitioning net ecosystem CO_2 exchange using stable isotope methods. In this study, eddy covariance, flux gradient, automated chambers, and stable carbon isotope techniques were used to quantify and improve our understanding of the temporal variability in F_R and δ_R in a C_3/C_4 agricultural ecosystem. Six years (2004–2009) of isotope flux-gradient measurements indicated that δ_R had a very consistent annual pattern during both C_3 (soybean) and C_4 (corn) growing seasons due to significant contributions from F_{Ra}, which was strongly influenced by the isotope composition of the recent photosynthate. However, in the spring, δ_R exhibited a C_3 signal regardless of the crop grown in the previous season. One hypothesis for this anomaly is that at these low soil temperatures microbial activity relied predominantly on C_3 substrates. Automated chamber measurements of soil respiration (F_{R_s}) and its isotope composition (δ_{R_s}) were initiated in the early corn growing season of 2009 to help interpret the variability in δ_R. These measurements showed good agreement with EC measurements of F_R (within 0.5 μmol m^2 s^{-1}) and isotope flux gradient measurements of δ_R (within 2‰) at nighttime for near-bare soil conditions (LAI < 0.1). At peak growth, nighttime δ_R above the corn canopy was consistently 1–6‰ more enriched than δ_{R_s}. The relatively enriched signal above the canopy indicates that δ_R was strongly influenced by aboveground plant respiration ($F_{R,ag}$), which accounted for about 40% of F_R. The automated chamber data and analyses also revealed a strong diel pattern in δ_{R_s}. In the early growth period, δ_{R_s} showed a sharp morning enrichment of up to 4‰ followed by a gradual depletion throughout the afternoon and evening. Daytime enrichment in δ_{R_s} was most pronounced during dry conditions and was not observed when the upper soil was near saturation. We provide anecdotal evidence that the diel variability during early growth may have been influenced by turbulence (advection/non-diffusive transport), which reduced the kinetic fractionation effect. At peak growth, there is evidence that the sheltering effect of the corn plants diminished the influence of turbulence on the chamber measurement of δ_{R_s}. Further research is needed to evaluate and separate the contributions of biotic and abiotic (advection and non-steady state effects) influences on chamber δ_{R_s} observations.

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1. Introduction

The release of CO_2 from the soil and living biomass, referred to as ecosystem respiration (F_R), is the major pathway for carbon loss in terrestrial ecosystems (Rustad et al., 2000; Ryan and Law, 2005; Herbst et al., 2008). Consequently, much attention has been aimed at how changes in climate and land use impact the autotrophic (F_{Ra}) and heterotrophic (F_{Rh}) source components of F_R (Hanson et al., 2000; Baldocchi et al., 2006; Sakata et al., 2007). Understanding the physical and biological controls on F_{Ra} and F_{Rh} is a necessary step for the development and validation of land surface schemes that forecast F_R.

The stable isotopologue $^{13}CO_2$ can be used to help trace the flow of carbon through an ecosystem and improve our understanding of ecosystem function (Hanson et al., 2000; Ehleringer et al., 2002; Wingate et al., 2010). A number of environmental factors have been shown to cause F_R and its isotope composition (δ_R) to vary. Differences in the carbon isotope ratios of the substrates of F_{Ra} and

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0168-1923/$ – see front matter. Published by Elsevier B.V.
doi:10.1016/j.agrformet.2011.09.018

F_{Rh} can have a strong influence on δ_R, which can be used to determine source changes in F_R (Högberg and Ekblad, 1996; Griffis et al., 2005; Baggs, 2006). In C_3/C_4 agricultural ecosystems, differences in the photosynthetic discrimination of C_3 (i.e. *Glycine max*) and C_4 (i.e. *Zea mays*) plants produce distinct isotope signatures (Farquhar, 1983; Farquhar et al., 1989), which can be used to evaluate the relative contributions of F_{Ra} and F_{Rh} (Rochette et al., 1999; Drewitt et al., 2009). In agricultural systems, the step change from a C_3 to a C_4 crop can result in dramatic seasonal changes in δ_R (Griffis et al., 2005).

In natural ecosystems, temporal changes in δ_R can be important, but more subtle (Ponton et al., 2006). Bowling et al. (2001) demonstrated that variability in forest δ_R could be linked to recent (2–4 day lag) changes in atmospheric vapor pressure deficit and its effect on plant stomatal response and photosynthetic discrimination. Similar evidence has been reported for other forest sites (Ekblad and Högberg, 2001; Högberg et al., 2007). Unger et al. (2010) utilized δ_{R_s} measurements to demonstrate that the F_{R_s} pulse observed immediately after soil rewetting ("Birch" effect) was from microbial sources in an evergreen oak woodland. Also, Marron et al. (2009) found that in a beech forest diel variability in δ_{R_s} was influenced by changes in the relative contribution of litter decomposition and root respiration.

Post-photosynthetic fractionation, associated with either dark respiration (Duranceau et al., 1999; Klumpp et al., 2005; Wingate, 2008) or the mobilization of newly assimilated organic matter in the plant (Hobbie and Werner, 2004; Gessler et al., 2008; Kodama et al., 2008; Wingate et al., 2010; Barbour et al., 2011a), has been shown to cause the carbon isotope composition of above and below ground respiration to differ from the isotope composition of newly assimilated CO_2 by up to several per mil. Post-photosynthetic fractionation, therefore, can lead to uncertainty in the values of above and below ground δ_{Ra} which can affect the partitioning of F_R.

Over the last several years there has been increased interest in using chambers to measure the isotope composition of respiration from ecosystem components (soil, roots, leaves) to better understand the variations described above under field conditions (Kayler et al., 2010; Wingate et al., 2010; Midwood and Millard, 2011). Under steady state and with diffusive gas transport, chamber measurements of δ_{R_s} should be equal to the biological source within the soil (Cerling et al., 1991; Susfalk et al., 2002; Millard et al., 2008; Risk and Kellman, 2008). Recent studies, however, have demonstrated that chamber (dynamic and static) isotope measurements are often adversely influenced by turbulence and non-steady-state (NSS) conditions, bringing into question the interpretation and value of such data (Risk and Kellman, 2008; Wingate, 2008; Kayler et al., 2010; Midwood and Millard, 2011). For instance, dry porous soils are highly susceptible to advection and mixing of atmospheric CO_2 into the soil profile, which can introduce errors of several per mil in δ_{R_s} (Millard et al., 2008; Midwood and Millard, 2011). Further, Kayler et al. (2010) found that advection caused δ_{R_s} to vary by up to 1‰ in a Douglas-fir stand.

In this study, eddy covariance, flux gradient, and automated soil chamber systems were combined with tunable diode laser (TDL) spectroscopy to quantify variability in δ_R at the interannual (2004–2009), seasonal, and diel time scales in a C_3/C_4 agricultural ecosystem. Specifically, in this study we: (1) determine the main factors influencing seasonal and interannual variation in δ_R, (2) utilize eddy covariance, flux gradient, and soil chamber data to estimate the contribution of aboveground plant respiration to the total respiration of the ecosystem, and (3) examine the biotic and abiotic factors influencing diel variability in the carbon isotope composition of soil respiration during a corn growing season.

2. Methodology

2.1. Study site

Field research was conducted at the University of Minnesota Rosemount Research and Outreach Center (RROC), located 25 km south of St. Paul, MN. Micrometeorological and stable isotope measurements were made in a 17 ha homogeneous agricultural field for the 2004–2009 field seasons with chamber measurements added in 2009. The soil is a Waukegan silt loam with an average bulk density of 1.14 g cm^{-3} and a relatively high organic carbon content (2.6%) that is underlain with a thick layer (>20 m) of coarse sand and gravel deposited by glacial outwash (Griffis et al., 2004). The upper soil (0–40 cm) at RROC is well mixed from tillage and the carbon isotope composition of the soil organic matter (δ_{SOM}) in this layer is about −18‰. At lower depths, δ_{SOM} is more enriched ranging from −15 to −13‰ (Griffis et al., 2005). The field site has been in agricultural production for 125 years with pre-settlement vegetation consisting of upland dry prarie. Prior to the 2002 growing season, the field was in corn production for four consecutive years. Since 2002, the field has been in an annual corn/soybean rotation.

2.2. Ecosystem scale observations

2.2.1. Micrometeorological measurements

Eddy covariance (EC) was used to measure net CO_2 exchange (F_N) over the course of the corn and soybean growing seasons (Baker and Griffis, 2005). The EC system consisted of an infrared gas analyzer (LI7500, LiCor, Lincoln, NE) and a three dimensional sonic anemometer (CSAT3, Campbell Scientific Inc., Logan, UT). These instruments were mounted on a boom that was adjusted according to changes in the height of the crop (Baker and Griffis, 2005).

For the 2004 through 2009 growing seasons, soil temperature was measured at depths of 5 and 10 cm using Type E thermocouples (chromel – constantan) and surface temperature was measured with an infrared thermometer (Model IRTS-P, Apogee Instruments, Logan, UT). A TDR100 (Campbell Scientific Inc., Logan, UT) was used to measure half-hourly soil water content at the 10 cm depth, although only data from the 2005, 2006, 2007, and 2009 growing seasons were available at the time of analysis. Other micrometeorological variables, including wind speed and net radiation, were measured on a nearby tower approximately 3 m above the soil surface. Unless otherwise stated, all measurements for this study were averaged on a half-hourly time period.

2.2.2. Carbon isotope composition of ecosystem respiration

The mixing ratios of $^{12}CO_2$ and $^{13}CO_2$ were measured using the TDL spectroscopy technique (Bowling et al., 2003; Griffis et al., 2004). Mixing ratios were measured at two sampling inlets mounted on a tower. Each inlet consisted of a heated Swagelok inline filter and a brass critical flow orifice that controlled the flow rate at 0.260 L min^{-1} (Griffis et al., 2007). The two sample inlets were positioned at approximately 1 m (z_1) and 2.5 m (z_2) above the roughness sublayer, respectively, and were adjusted throughout the growing season to maintain a constant distance above the height of the canopy. Air sampled at the inlets was pulled to an instrument trailer at the edge of the field site using Synflex tubing (Synflex Type 1300, Aurora, OH, USA) (Griffis et al., 2007). Inside the instrument trailer, the gradient sample lines were buffered using stainless steel mixing volumes to damp out the influence of turbulent fluctuations. The sample lines were then connected to a custom made manifold which controlled air flow into a tunable diode laser (TGA100A, Campbell Scientific, Logan, UT, USA). Each inlet was measured every 2 min for 45 s per sample, with the first 7 s of each sample omitted for pressure equilibration (Bowling et al., 2003; Griffis et al., 2004). The TDL was calibrated every 5 min

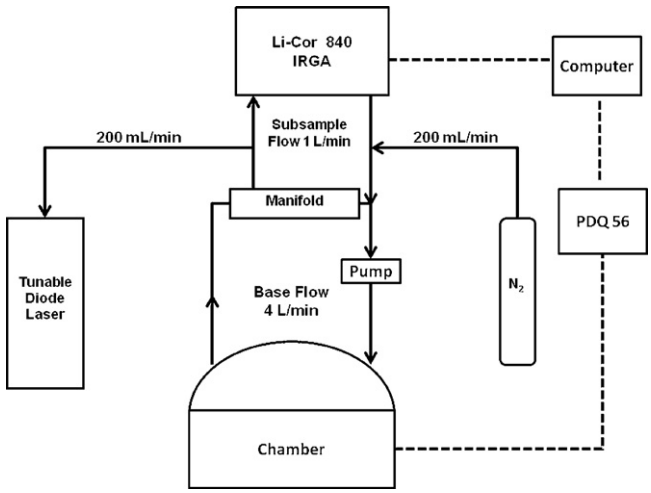

Fig. 1. Schematic of the automated chamber system with TDL for carbon isotope analysis of CO_2 efflux. The dashed lines indicate electrical connections.

with two gases of known $^{12}CO_2$ and $^{13}CO_2$ concentration that are traceable to the NOAA Earth System Research Laboratory (ESRL). Calibration gases were measured for 30 s, with data omission and averaging similar to sample line measurements (Griffis et al., 2007).

The isotope flux ratio method was used to estimate δ_R and was expressed in delta notation using (Griffis et al., 2004):

$$\delta_R = \left(\frac{F_R^{13}/F_R^{12}}{R_{VPBD}} - 1 \right) \times 1000 \quad (1)$$

For this study, the average δ_R value was calculated by plotting nightly (22:00–04:00 h) F_R^{13} values against F_R^{12} values. A geometric Type II regression was used to derive δ_R from the flux ratio plot. Linear fits with a coefficient of determination (r^2) less than 0.9995 were rejected from the analysis to limit uncertainty in the δ_R value. To isolate the carbon isotope composition of ecosystem respiration, only periods of photosynthetic inactivity were used. We limited the data analysis to well mixed ($u_* > 0.1\,\mathrm{m\,s^{-1}}$), nighttime conditions for the 2004 through 2009 growing seasons. Using these criteria, uncertainty in δ_R using the flux ratio method was typically ±3.5‰ in the spring and ±1.7‰ in the growing season (Griffis et al., 2005).

2.2.3. Carbon isotope composition of soil respiration

A closed, non-steady state chamber system was deployed at the RROC research field during the summer of 2009 to quantify variability in F_{R_s} and δ_{R_s}. The chamber system used for this study was designed by the Biometeorology and Soil Physics Group at the University of British Columbia (Gaumont-Guay et al., 2006). For this study, only two chambers were utilized. Each chamber dome was made of clear acrylic and had a headspace volume of 0.06 m^3.

Synflex tubing (Synflex Type 1300, Aurora, OH, USA) carried air from the sample inlet to a custom made manifold that controlled sampling to a Li-Cor 840 IRGA (Li-Cor Inc., Lincoln, NE, USA). After air from the sample line was analyzed by the IRGA, a diaphragm pump (Model NMP850KNDCB, KNF Neuberger Inc., Trenton, New Jersey, USA) returned the air back to the chamber headspace in a separate Synflex tube (return line) (Fig. 1). The base flow rate to and from the chambers was set at 4 L min^{-1}. Base flow from the chamber sample line was subsampled at 1 L min^{-1} through the IRGA and returned to the chamber return line. All chambers were equipped with a small internal fan to ensure a well mixed headspace. Chamber measurements were performed every 15 min with a sample duration time of 300 s. The chambers were mechanically opened when not being sampled, allowing the soil inside the collar to be exposed to the atmosphere.

The chambers were positioned 6 m apart and 20 m from the north edge of the research field and placed within the same corn row. No vegetation was allowed to grow within the chambers. Chambers were equipped with a metal skirt placed around a rubber seal made of ethylene propylene diene terpolymer (EPDM) to minimize infiltration of ambient air due to wind gusts. Chambers were also equipped with a "pig-tail" pressure vent tube to eliminate a pressure gradient between the inside and outside of the chamber. The "pig-tail" shape of the pressure vent tube was designed to dampen the Venturi effect which can occur in windy conditions (Conen and Smith, 1998; Bain et al., 2005). Covers made of reflective insulating material (Reflectix, Markleville, IN, USA) and mylar tape were placed on the chambers to reduce the increase of air temperature within the chamber headspace during sampling (Bavin et al., 2009). With the reflective covers in place, chamber air temperature typically increased 0.6 °C during a single sampling event.

The δ_{R_s} signal was measured by incorporating a tunable diode laser (TDL) into the automated chamber system. A subsample from the chamber sample line brought air to the TDL at $1.48 \times 10^{-4}\,\mathrm{mol\,s^{-1}}$ (200 mL min^{-1}). Sample air was dried by a Nafion dryer (PD-Series, Perma Pure, Toms River, NJ, USA) before analysis by the TDL. Although total CO_2 concentration was measured continuously during chamber sampling by the Li-Cor 840 IRGA, measurement of $^{13}CO_2$ and $^{12}CO_2$ by the TDL was not continuous. Instead, the TDL sampled chamber air for 3 discrete, 10-s intervals during the 300-s chamber closure. The three intervals were combined and a best fit linear regression was used to calculate $F_{R_s}^{13}$ and $F_{R_s}^{12}$ from the chambers. Flux-gradient measurements of $^{13}CO_2$ and $^{12}CO_2$ concentrations above the corn canopy were conducted by the TDL when it was not sampling chamber air. A typical chamber measurement of $^{13}CO_2$, $^{12}CO_2$, and total CO_2 concentrations is shown in Fig. 2.

Soil respiration (F_{R_s}) was calculated using:

$$F_{Rs}^{x} = \frac{P\,V\,d^{x}CO_2/dt}{A\,T_a\,R} \quad (2)$$

whose superscript x indicates either the isotope $^{12}CO_2$ or $^{13}CO_2$, P is atmospheric pressure (98 500 Pa), V is the volume of the chamber, A is the surface area of the chamber (0.216 m^2), T_a is the air temperature (K), and R is the gas constant (8.3144 m^3 Pa K^{-1} mol^{-1}). For this study, sign convention defines a positive flux leaving the surface. A best fit linear regression was used to determine the rate of concentration change within the chamber headspace during a single sampling event. The carbon isotope composition of soil respiration (δ_{R_s}) was calculated using

$$\delta_{R_s} = \left(\frac{F_{Rs}^{13}/F_{Rs}^{12}}{R_{VPBD}} - 1 \right) \times 1000 \quad (3)$$

Linear regressions with an r^2 coefficient less than 0.95 were excluded from the analysis. Uncertainty in the chamber measurement of δ_{R_s} averaged ±0.4‰ in the 2009 corn growing season. Chamber air analyzed by the TDL was not returned to the chamber. To compensate, N_2 gas flowing at $1.48 \times 10^{-4}\,\mathrm{mol\,s^{-1}}$ (200 mL min^{-1}) was added to the chamber return line. Replacement of air sampled by the TDL with N_2 caused a dilution of the chamber CO_2 concentration, causing a slight underestimation of the flux measurement. A correction factor was obtained from the product of the N_2 flow rate into the chamber and the mean chamber concentration of either $^{12}CO_2$ or $^{13}CO_2$ (Griffis et al., 2011). Using this method, the theoretical dilution flux was calculated at −0.21 and −0.0022 μmol m^{-2} s^{-1} for F^{12} and F^{13}, respectively. This offset was added to each flux measurement. The dilution correction altered the isotope ratio of the flux slightly, with a median enrichment of about 0.4‰.

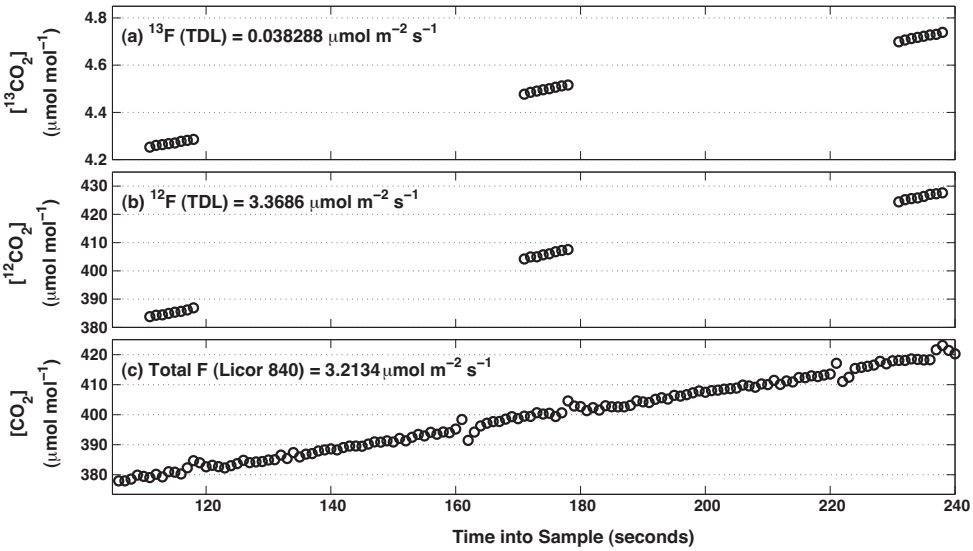

Fig. 2. Example of a typical soil chamber measurement in the 2009 corn growing season showing TDL concentration measurements of (a) $^{13}CO_2$ and (b) $^{12}CO_2$. A Li-Cor 840 IRGA measured the total CO_2 concentration (c). The δ_{R_s} value for this sample is −16.6‰.

2.3. Partitioning ecosystem and aboveground plant respiration

We estimated aboveground plant respiration ($F_{R,ag}$) and its carbon isotope composition ($\delta_{R,ag}$) using a simple mass balance approach to understand their relative contributions to F_R and δ_R, respectively, using

$$F_R = F_{R,ag} + F_{R_s} \tag{4a}$$

$$\delta_R F_R = \delta_{R,ag}(F_R - F_{R_s}) + \delta_{R_s} F_{R_s} \tag{4b}$$

Here, $F_{R,ag}$ was estimated by taking the difference between nighttime (22:00–04:00 h) mean values of F_R (measured with eddy covariance) and F_{R_s} (measured with soil chambers), leaving the isotope signature of aboveground respiration ($\delta_{R,ag}$) as the only unknown term. Chamber measurements of F_{R_s} and EC measurements of F_R taken shortly after corn planting were generally within 0.5 μmol m^2 s^{-1}, showing little measurement bias between the methods and indicating that partitioning later in the growing season should be reasonable.

3. Results and discussion

3.1. Climate and net CO_2 exchange

Nightly average soil temperature (T_s, 5 cm depth) is shown in Fig. 3a. Although the timing of winter thaw varied from year to year, the annual T_s pattern was relatively consistent, reaching a maximum of 25 °C around Day of Year (DOY) 200 (July 19). Volumetric soil water content was highest in the early spring after winter thaw, ranging from 0.3 to 0.4 m^3 m^{-3} before gradually decreasing to <0.20 m^3 m^{-3} in the mid to late summer (Fig. 3b).

Peak photosynthetic uptake in corn growing seasons occurred from approximately DOY 175–225, causing daytime F_N to reach values of −50 μmol m^{-2} s^{-1} (Fig. 4a). After peak uptake, F_N gradually became less negative at −20 to −10 μmol m^{-2} s^{-1}. Peak

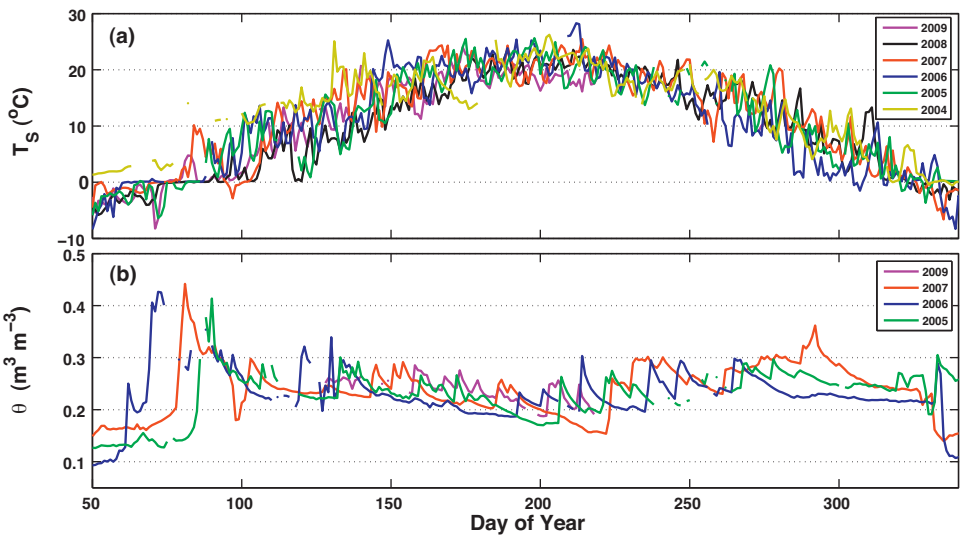

Fig. 3. (a) Nightly averages of soil temperature at the 5 cm depth. (b) Nightly averages of soil water content at the 10 cm depth for the 2005 through 2007 growing seasons.

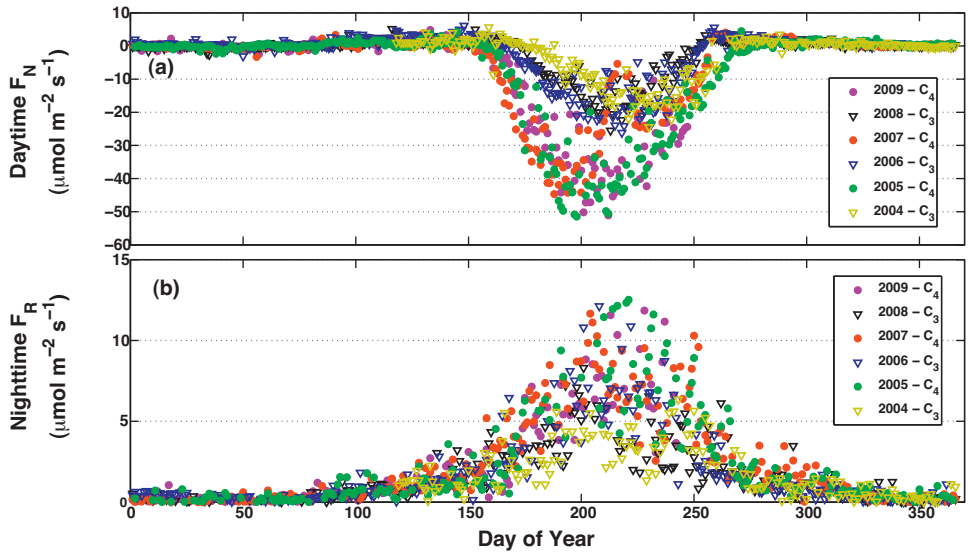

Fig. 4. Eddy covariance data showing (a) daytime averages of F_N (10:00–16:00 h) and (b) nighttime averages of F_R (22:00–04:00 h) for the 2004 through 2009 corn (circles) and soybean (inverted triangles) growing seasons.

uptake in soybean seasons occurred from DOY 200 to 240. Daytime F_N in peak soybean growth was much less than in corn years, reaching −25 μmol m^{-2} s^{-1}. After DOY 240, F_N became less negative at −15 to −10 μmol m^{-2} s^{-1} in soybean seasons. Nighttime (22:00–04:00 h) F_R steadily increased after leaf emergence, with a peak rate close to 12.0 μmol m^{-2} s^{-1} in corn seasons. On average, peak nighttime F_R in corn years occurred on about DOY 225 (Fig. 4b), lagging peak uptake by approximately 10–15 days. The F_R pattern varied among soybean seasons, peaking as early as DOY 200 at 7.1 μmol m^{-2} s^{-1} and as late as DOY 240 in 2004 at 6.8 μmol m^{-2} s^{-1}. No discernible pattern was observed between peak F_R and peak F_P in soybean growing seasons.

3.2. Diel and seasonal variation in soil respiration

Soil respiration (F_{R_s}) was measured with two automated chambers beginning on DOY 140 and ending on DOY 220 (early to peak growth) of the 2009 corn growing season. Reported values of F_{R_s} represent the half hourly mean of the two chamber measurements. Measurement of F_{R_s} concluded prematurely due to damage suffered from a lightning strike on DOY 220. In the early growth period (DOY 140–155, crop height <0.2 m), F_{R_s} was predominantly heterotrophic and exhibited a consistent diel pattern with average daytime and nighttime values at 2.8 and 1.6 μmol m^{-2} s^{-1}, respectively (Fig. 5a). Chamber measurements indicated that F_{R_s} slightly lagged surface temperature in this period but their respective diel ensembles showed a strong correlation (r^2 = 0.93, n = 24) (Fig. 5b). The hysteresis loop inverted below the 5 cm depth, with peak 5 cm and 10 cm soil temperatures lagging peak F_{R_s} by 1 and 2 h, respectively (Figs. 5c and d).

As the corn entered rapid development beginning on about DOY 175, F_{R_s} gradually increased with average daytime and nighttime values of 3.9 and 2.8 μmol m^{-2} s^{-1}, respectively. Peak F_{R_s} continued to slightly lag peak surface temperature in this period and their respective diel ensembles exhibited a strong overall relationship (r^2 = 0.95, n = 24). Also, the diel ensembles of F_{R_s} and 5 cm

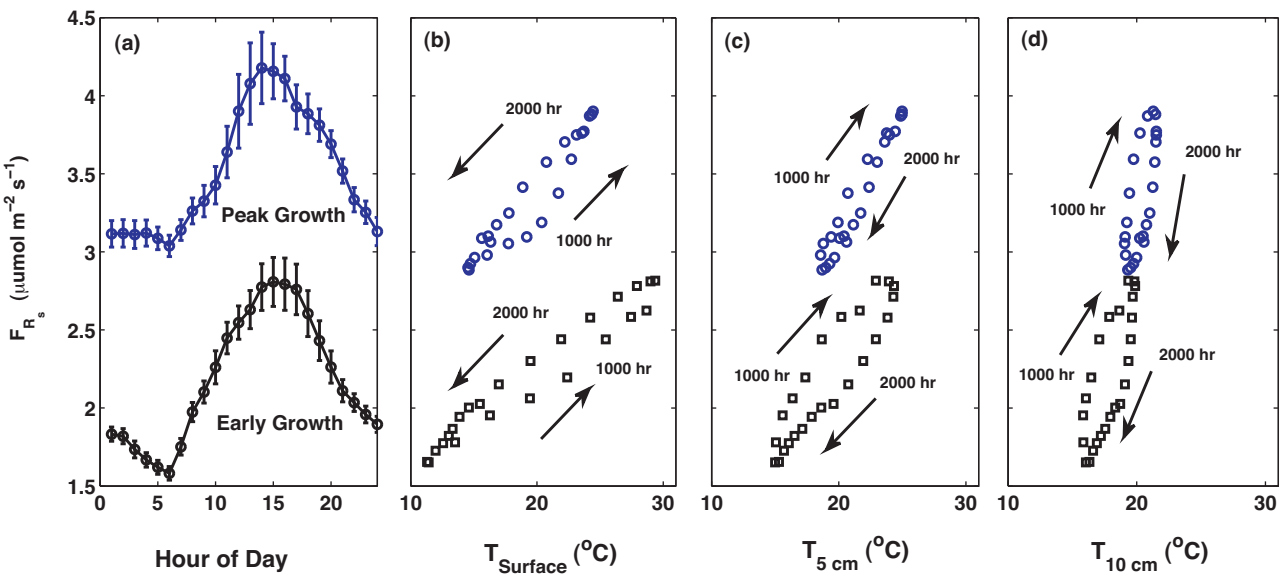

Fig. 5. Diel ensembles of (a) soil respiration (F_{R_s}) and its relationship to (b) surface temperature, (c) 5 cm soil temperature, and (d) 10 cm temperature in early and peak growth for the 2009 corn growing season at RROC. All diel ensembles represent 15 days of continuous data from either DOY 142 to 157 during early growth or DOY 179 to 194 during peak growth. The black squares represent values from early growth while blue circles represent values from peak growth.

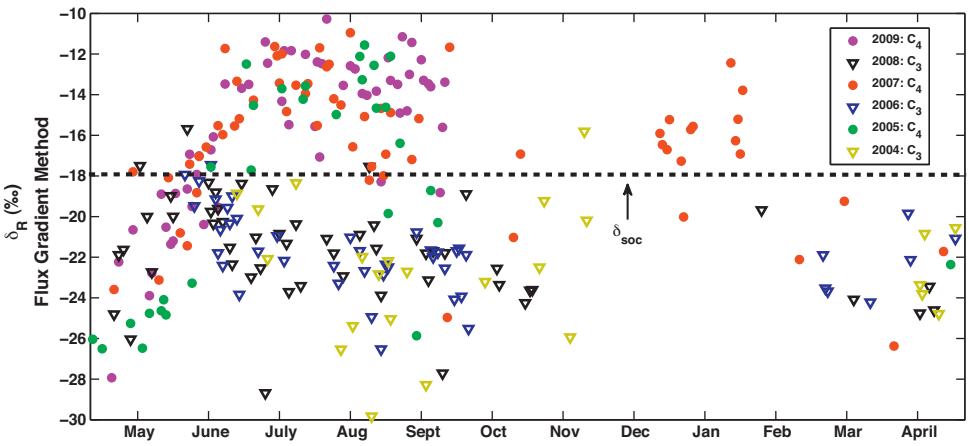

Fig. 6. Nightly values of δ_R for the 2004–2009 corn (circles) and soybean (inverted triangles) growing seasons. The dashed line at −18‰ represents the average isotope composition of soil organic carbon at RROC. Each growing season began at the time of planting (May) and ended prior to the planting of the next crop.

soil temperature showed a stronger relationship in peak growth (r^2 = 0.85, n = 24) than in early growth (r^2 = 0.65, n = 24). Inversion of the hysteresis loops continued to occur at a depth of 5 cm in peak growth which is much shallower than the 20 cm depth observed by Gaumont-Guay et al. (2006) in a boreal aspen stand. This difference may be partially due to dissimilarities in the temperature and CO_2 production profiles between these ecosystems. However, Phillips et al. (2010) and Riveros-Iregui et al. (2011) found that hysteresis loops between soil temperature and F_{R_s} can vary independent of temperature and CO_2 production by changing physical properties such as soil moisture and thermal diffusivity.

3.3. Interannual and seasonal variation in the carbon isotope composition of ecosystem respiration

Flux-gradient measurements revealed major shifts in nighttime δ_R during the course of the corn and soybean growing seasons (Fig. 6). As F_{Ra} began to increase in the early stages of peak growth, the δ_R signal equilibrated toward the photosynthetic signature of the current crop, carrying a strong C_4 signal in corn growing seasons and a strong C_3 signal in soybean growing seasons. In corn growing seasons, δ_R enriched from a pre-growing season value of about −22‰ to a peak value of about −11‰ occurring as early as July 18 (DOY 200) in 2005 and as late as August 17 (DOY 230) in 2009. After peak growth, δ_R was highly variable but exhibited a decrease to about −18‰, likely due to a decrease in F_{Ra}. Although data are limited in the winter following corn growing seasons, δ_R was variable but slightly enriched relative to δ_{SOM} in the upper soil. Values of δ_R during this period ranged from −20 to −15‰ and may have been influenced by the microbial decomposition of fresh corn residue and/or isotopically heavy SOM in deeper, warmer soil layers. As the soybean plants developed δ_R gradually became depleted from −18‰ to about −22‰ due to increased C_3 plant respiration. After about July 10 (DOY 192), δ_R was highly variable in all soybean growing seasons but exhibited a strong C_3 signal, ranging from −28 to −21‰.

Surprisingly, δ_R in the spring months (March to May) was consistently about −23 ± 3 ‰ regardless of the crop present in the previous growing season. One possible explanation for this anomaly is that at low soil temperatures, corn residue (i.e. stalks, leaves, and roots) may be more resistant to decomposition by soil microbial communities than soybean residue (Ajwa and Tabatabai, 1994). Archival photographs and personal observations of the agricultural field in the spring following corn growing seasons showed an abundance of corn residue. Following soybean seasons, however, soybean residue was nearly absent. Soil chamber measurements showed an enrichment in δ_{R_s} from early to peak growth of the 2009 corn season (Fig. 7b). In early growth, nightly average δ_{R_s} values ranged between −24 and −20‰ and were consistent with flux-gradient measurements of δ_R taken during the same time

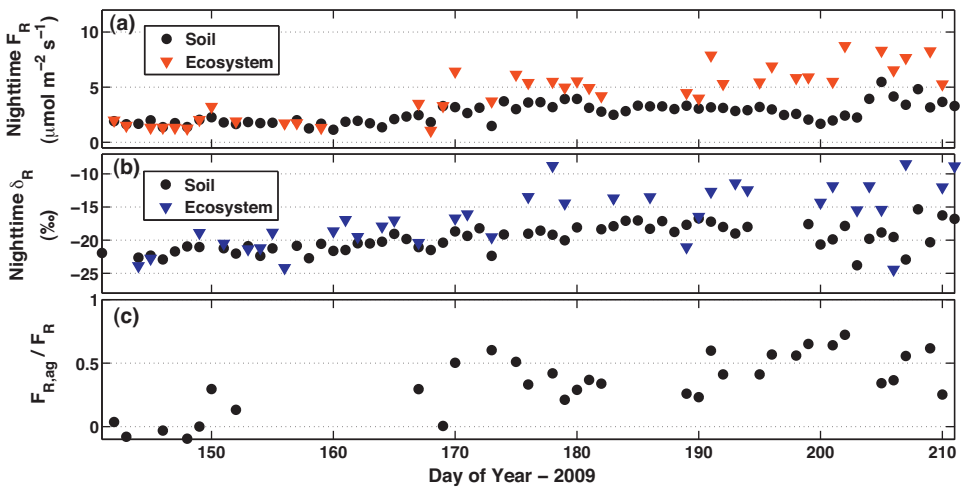

Fig. 7. Nighttime averages of (a) F_{R_s} (black circles) and F_R (red triangles), (b) δ_{R_s} (black circles) and δ_R (blue triangles), and (c) the ratio of aboveground plant respiration ($F_{R,ag}$) to F_R during the early to mid 2009 corn growing season at RROC.

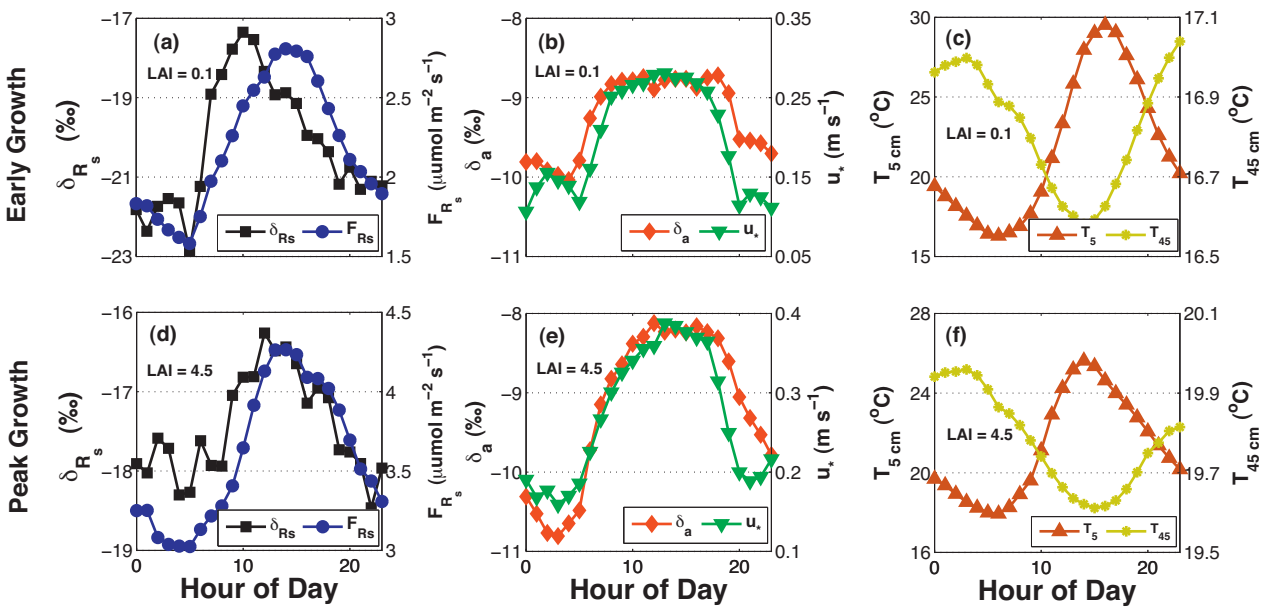

Fig. 8. Diel ensembles of δ_{R_s} (black squares), F_{R_s} (blue circles), friction velocity (u_*, green inverted triangles), the carbon isotope composition of ambient CO_2 (δ_a, red diamonds), and soil temperature at the 5 cm (T_5, brown triangles) and 45 cm (T_{45}, yellow stars) depths in early growth (a–c) and peak growth (d–f). All diel ensembles represent 15 days of continuous data from either DOY 142 to 157 during early growth or DOY 179 to 194 during peak growth. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

period (within 2‰). In addition, EC measurements of F_R and soil chamber measurements of F_{R_s} during the nighttime were in good agreement (within 0.5 μmol m^2 s^{-1}), indicating little contribution from aboveground plant respiration to the total respiratory flux (Fig. 7a).

As the corn entered rapid development on about June 15 (DOY 166) and root respiration increased, nightly δ_{R_s} values gradually enriched, reaching about −16‰ by July 29 (DOY 210). Nightly flux-gradient measurements of δ_R taken above the corn canopy were generally 1–6‰ more enriched than soil chamber values of δ_{R_s} after about June 15, indicating an increase in ^{13}C-enriched aboveground respiration from the corn leaves and shoots. Supporting this hypothesis are EC measurements of nighttime F_R that were consistently 2–5 μmol m^{-2} s^{-1} greater than soil chamber measurements of F_{R_s} in this period (Fig. 7a).

Using Eq. (4a), $F_{R,ag}$ was estimated and showed an increasing contribution to total F_R as the growing season progressed from early to peak growth. The $F_{R,ag}/F_R$ ratio was highly variable, ranging from 0.20 to 0.75 in peak growth with a mean value of 0.43 (Fig. 7c). The high variability in this ratio was likely due to the complex relationships between climate, soil respiration (both heterotrophic and autotrophic), the rapid development of the corn crop, and the inherent noise in the EC and chamber flux measurements. For example, from July 9 to 20 (DOY 190–201) $F_{R,ag}/F_R$ steadily increased from 0.23 to 0.72, corresponding to a decrease in soil respiration. Values of δ_{R_s} during this period ranged from −16 to −19‰. After a 20 mm rain event on DOY 202, nighttime soil respiration increased sharply over the next 48 h from 2.2 to 5.4 μmol m^{-2} s^{-1}. The increase in soil respiration caused $F_{R,ag}/F_R$ to decrease from 0.72 to 0.35 during this time period. However, δ_{R_s} decreased to near −20‰ after the rain event, suggesting that the sharp increase in soil respiration may have stimulated heterotrophic contributions. Unger et al. (2010) observed that rapid F_{R_s} pulses associated with the rewetting of dry soils ("Birch" effect) were from microbial sources. Also, microbial moisture sensitivity was observed by Gaumont-Guay et al. (2008) who found that an increase in soil water content during spring thaw induced a small and sustained increase in heterotrophic respiration but had no effect on autotrophic respiration in a black spruce stand.

The carbon isotope composition of aboveground plant respiration ($\delta_{R,ag}$) was estimated and averaged −10.2 ±3‰ in the peak growth period. Here, $\delta_{R,ag}$ was not measured directly (Eq. (4b)). Although highly variable, this mean value is slightly more enriched than the average carbon isotope ratio of −12‰ for C_4 plants (Farquhar et al., 1989; Lai et al., 2003; Zhang et al., 2006). Enriched values of $\delta_{R,ag}$ on the leaf-level have been shown in several studies (Duranceau et al., 1999; Ghashghaie et al., 2003; Klumpp et al., 2005; Barbour et al., 2007, 2011b; Gessler et al., 2008), although very few have examined $\delta_{R,ag}$ from C_4 vegetation.

3.4. Diel variation in the carbon isotope composition of soil respiration

In the early growth period of the 2009 corn growing season, δ_{R_s} exhibited a strong diel pattern consisting of a sharp morning enrichment (06:00–10:00 h) followed by a gradual depletion throughout the afternoon and evening. Values of δ_{R_s} in early growth averaged about −21.5‰ in the nighttime with daytime values more enriched at about −18.5‰. Peak enrichment in δ_{R_s} occurred 3–4 h before peak values of F_{R_s} and 5 cm soil temperature (Fig. 8a and b). Daytime enrichment in δ_{R_s} in early growth was most pronounced under dry soil conditions and diminished during and shortly after heavy rain events. For example, from May 30 to June 4 (DOY 150–155), soil conditions were dry and δ_{R_s} showed strong daytime enrichment (Fig. 9a). Rain events on DOY 157 (34 mm) and 159 (14 mm) saturated the upper soil, causing the water content to reach 0.35 m^3 m^{-3} and remain over 0.3 m^3 m^{-3} until DOY 162 (Fig. 9d). During this period, the pattern of daytime enrichment in δ_{R_s} was diminished. After DOY 162 the soil water content decreased and daytime enrichment of δ_{R_s} became more evident.

We have proposed two hypotheses to explain the diel variability in δ_{R_s} in the early growth period. The first hypothesis is that the enriched values of δ_{R_s} were caused by contributions from microbial decomposition of SOM in deeper, isotopically heavier soil layers. At our agricultural field site, the 0–40 cm soil layer is well mixed due to annual tillage, causing δ_{SOM} to be between the C_3 and C_4 end members at about −18‰ (Griffis et al., 2005). Below the tillage zone δ_{SOM} is more enriched at about −14‰. Soil temperature data

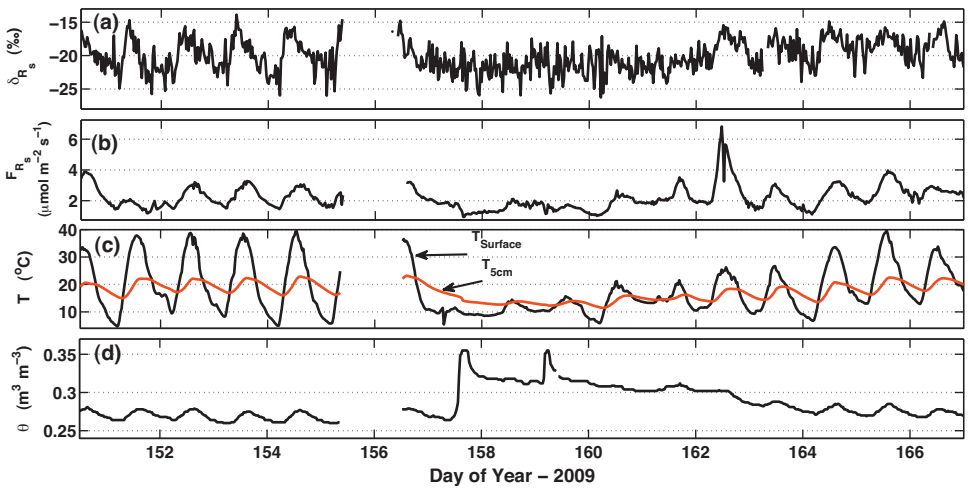

Fig. 9. Half hourly measurements of (a) δ_{R_s} and (b) F_{R_s} during early growth of the 2009 corn growing season at RROC. Also plotted are half hourly measurements of (c) temperature at the surface (black line) and at the 5 cm soil depth (red line), and (d) soil moisture at the 10 cm depth. Two significant rain events occurred on DOY 157 and 159 which saturated the upper soil until DOY 162. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

(Fig. 8c) signify that peak temperature at the 45 cm depth lags the peak temperature near the surface by 12–15 h. This temperature lag means that at 06:00 h (when δ_{R_s} began to show a sharp enrichment) the temperature at the 45 cm depth was near its highest (16.9 °C) when the temperature at the surface was near its lowest (10.0 °C). Diel variability in microbial activity may have occurred within the soil profile because of this temperature lag, producing varying contributions to both $^{13}F_{R_s}$ and $^{12}F_{R_s}$ that could have significantly altered δ_{R_s}. The use of a simple mixing model shows that when F_{R_s} is 2.5 μmol m^{-2} s^{-1} and the isotope composition of the upper soil is −22‰ (with fresh soybean residue included), 50% of the total flux would have to come from below the 45 cm depth to increase the carbon isotope composition of F_{R_s} by 4‰.

The second hypothesis is that variability in δ_{R_s} was caused by turbulence and/or non-steady state diffusion during chamber sampling. In the early growth period (LAI = 0.1, crop height <0.2 m), the chambers were exposed directly to wind, which may have resulted in advection and diminished diffusive gas transport in the upper soil layer. Because the transport was not purely diffusional the kinetic fractionation effect may have been less than the theoretical value of 4.4‰, especially during periods when the respiration rate was low. Ensemble averages of δ_{R_s} during the period of morning enrichment (06:00–12:00 h) showed strong relationships with friction velocity (r^2 = 0.92, n = 7, Fig. 8b) and wind speed (r^2 = 0.87, n = 7). Also, infiltration of atmospheric CO_2 may have been diminished when the upper soil was saturated during and shortly after rain events, explaining why daytime enrichment in δ_{R_s} was not observed during these periods (Fig. 9). Millard et al. (2008) found that dry, porous soils with low respiration rates were highly susceptible to infiltration of atmospheric CO_2. Midwood and Millard (2011) found that mixing of atmospheric CO_2 with soil CO_2 can be problematic during chamber sampling in dry soils and can dramatically alter the δ_{R_s}. Further, Kayler et al. (2010) demonstrated that advection could produce a 1‰ variation on δ_{R_s}, suggesting that advection plays a minor role in a forest ecosystem. For our study, however, the unique conditions of an agricultural field in the early growth period may have resulted in stronger advective effects.

The use of non-steady state (NSS) chambers may have also contributed to the variability in δ_{R_s} by creating disturbances to the natural diffusive processes in the upper soil. Nickerson and Risk (2009) concluded that dynamic fractionation exists under NSS diffusive conditions and will be more pronounced in environments such as agricultural systems where the rate production and diffusive parameters vary significantly on the diel timescale. Both Bowling and Massman (2011) and Risk and Kellman (2008) found that under NSS diffusive conditions the kinetic fractionation factor was likely less than 4.4‰, causing measured values of δ_R to be depleted relative to the source. Their studies, however, measured δ_R using the Keeling regression method (Keeling, 1958), which has been shown to be unreliable under NSS conditions (Risk and Kellman, 2008; Nickerson and Risk, 2009; Midwood and Millard, 2011). An enrichment in δ_{R_s} was observed by Susfalk et al. (2002) due to the lateral diffusion of near-surface CO_2 into the chamber collar during chamber sampling. Their study concluded that lateral diffusion occurred due to a pressure gradient established by the soil chamber and was most pronounced in coarse soil with low soil moisture content. For our study, it is likely that NSS conditions were created by both advection and the soil chamber footprint which contributed to variability in δ_{R_s} in the early growth period. However, quantification of these influences on the chamber measurement of δ_{R_s} is made difficult because continuous measurement of subsurface $^{12}CO_2$ and $^{13}CO_2$ concentrations was not made during the experiment.

The δ_{R_s} signal in peak growth was up to 4‰ more enriched than in early growth (Fig. 8c). Values of δ_{R_s} in peak growth exhibited a strong diel pattern but with less variability, with nighttime and daytime values at −18.0 and −16.5‰, respectively. Peak enrichment of δ_{R_s} was also shifted 2–3 h later in the afternoon, occurring between 12:00 and 14:00 h. Diel variability in the δ_{R_s} signal in peak growth may have been influenced by corn root respiration. Although Bathellier et al. (2009) observed little change in the carbon isotope composition of root respiration, Huck et al. (1962) observed that the root respiration rate in corn plants was highest in the daytime due to the presence of light. Harris and van Bavel (1957) found that corn root respiration was highest in the afternoon (16:00 h) and that root respiration increased with the age of the plant. An increase in daytime root respiration could help explain why peak enrichment in δ_{R_s} occurred in the afternoon in the peak growth period. The diel ensembles of F_{R_s} and δ_{R_s} exhibited a stronger relationship in peak growth (r^2 = 0.78, n = 24) than in early growth (r^2 = 0.45, n = 24), indicating an influence from corn root respiration. In addition, a stronger relationship between the diel ensembles of δ_{R_s} and 5 cm soil temperature was observed in peak growth (r^2 = 0.45, n = 24) than in early growth (r^2 = 0.05, n = 24) (Fig. 8d). Assuming soil temperature can be used as a proxy for microbial activity, δ_{R_s} may have been influenced by microbial consumption of corn root exudates. The lower diel variability in δ_{R_s} observed in peak growth could also be due to the sheltering effect of

the tall corn canopy that diminished abiotic influences on the chamber measurements. Overall, chamber measurement of δ_{R_s} in the peak growth period appeared to be less influenced by abiotic processes when compared to the early growth period which allowed for the biotic processes influencing δ_{R_s} to be examined more clearly.

4. Conclusions

1 Flux-gradient measurements indicated that δ_R had a very consistent annual pattern in both soybean and corn growing seasons due to contributions from F_{Ra}, showing a strong C_4 signal during corn years and a C_3 signal during soybean years. In the spring, δ_R exhibited a strong C_3 signal regardless of the crop grown in the previous season. This anomaly may be due to corn residue being more resistant to microbial decomposition than soybean residue, especially when soil temperatures are low.

2 Chamber measurements of F_{R_s} and δ_{R_s} in the early growth period were in good agreement with EC measurements of F_R and flux gradient measurements of δ_R during the nighttime but showed a significant departure during the daytime. This departure was likely the result of wind gusts affecting the highly exposed soil chambers during sampling.

3 In the peak corn growth period, nighttime δ_R measurements taken above the canopy were consistently 1–6‰ more enriched than δ_{R_s}. The relatively enriched signal above the canopy was likely explained by the strong influence of $F_{R,ag}$ on δ_R, which accounted for an average of 43% of F_R in the peak growth period.

4 Strong diel variability in δ_{R_s} was observed throughout the corn growing season. In the early growth period, δ_{R_s} showed a sharp morning enrichment of up to 4‰ followed a gradual depletion throughout the afternoon and evening. Daytime enrichment in δ_{R_s} was most pronounced during dry soil conditions and was not observed when the upper soil was near saturation. In the peak growth period, the diel pattern of δ_{R_s} was strongly correlated to F_{R_s}, suggesting an influence from corn root respiration.

5 Based on our observations, chamber measurement of δ_{R_s} in the early growth period was least impacted by abiotic processes in the nighttime when turbulence was low (u_* <0.1 m s^{-1}). In the peak growth period, the sheltering effect of the corn plants and higher soil respiration rates appeared to diminish the influence of abiotic processes and allowed for biotic influences on δ_{R_s} variability to be examined more clearly.

Acknowledgements

We thank Matt Erickson and Bill Breiter for their technical assistance at the field site. We also thank Zoran Nesic and Dr. T.A. Black, Biometeorology and Soil Physics Group, University of British Columbia, for their help with the implementation of the automated chamber system. We acknowledge the very helpful comments and criticisms of two anonymous reviewers and the guest editor. Funding for this research has been provided by the National Science Foundation, ATM-0546476 (T.G.) and the Office of Science (BER) U.S. Department of Energy, DE-FG02-06ER64316 (T.G. and J.M.B.).

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