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Soil CO_2 Efflux Measurements by Alkali Absorption and Infrared Gas Analyzer in the Brazilian Semiarid Region

Carlas Renata Prissila Costa Ferreira[1], Antonio Celso Dantas Antonino[2], Everardo Valadares de Sá Barreto Sampaio[2], Karina Guedes Correia[3], José Romualdo de Sousa Lima[4]*, Willames de Albuquerque Soares[5] and Rômulo Simões Cezar Menezes[2]

[1] Universidade Federal de Pernambuco, Departamento de Energia Nuclear, Programa de Pós-Graduação em Tecnologias Energéticas e Nucleares, Recife, Pernambuco, Brasil.
[2] Universidade Federal de Pernambuco, Departamento de Energia Nuclear, Recife, Pernambuco, Brasil.
[3] Centro Universitário de João Pessoa, João Pessoa, Paraíba, Brasil.
[4] Universidade Federal Rural de Pernambuco, Unidade Acadêmica de Garanhuns, Garanhuns, Pernambuco, Brasil.
[5] Universidade de Pernambuco, Escola Politécnica de Pernambuco, Recife, Pernambuco, Brasil.

*** Corresponding author:**
E-mail: romualdo.lima@ufrpe.br

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ABSTRACT: The CO_2 emission from the soil surface, commonly referred to as soil CO_2 efflux (ECO_2) or soil respiration, is the sum of processes that include root respiration and microbial activity. Measuring this evolution is important to establish sustainable land use models and to estimate global fluxes of carbon, which affect climate change. Despite its importance, few measurements have been made in areas of the semiarid Brazilian Northeast region, and most of them were made using the alkali absorption method (AA), which can underestimate ECO_2. Measurements using AA were compared to measurements using the infrared gas analyzer method (IRGA) over ten months (in rainy and dry seasons), during the day and night, in areas of *Caatinga* (xeric shrubland and thorn forest) and pasture in the Agreste region of the state of Pernambuco. The ECO_2 measurements from AA varied little from night to day and throughout the year or in the rainy and dry seasons. However, those obtained from IRGA were higher in the rainy than in the dry season, but also without significant differences from day to night. The values of both methods were similar in the dry season, but in the rainy season they were higher with the IRGA. Therefore, AA seems to have little sensitivity to seasonal variations, in contrast with measurements from the IRGA, and it may underestimate soil ECO_2 when it attains higher values. This result indicates that some of the soil ECO_2 values determined in areas of the Brazilian semiarid region, and consequently annual C losses, may have been underestimated.

Keywords: CO_2 emission, rainy season, dry season, *Caatinga*, pasture.

INTRODUCTION

The carbon cycle has received increased attention due to escalating concentrations of atmospheric carbon gas and its relationship with global warming and climatic changes (IPCC, 2014). Soil CO_2 efflux (ECO_2), also referred to as soil respiration, is an important component of the global carbon balance, returning about 80×10^9 tons of C to the atmosphere each year (Raich et al., 2002). Therefore, ECO_2 can have a large influence on global atmospheric CO_2 (Ryan and Law, 2005; Zheng et al., 2014) and the increase of ECO_2 may promote disturbing climatic changes (Pinto-Junior et al., 2009; Panosso et al., 2012).

Several studies have quantified ECO_2 in specific areas in Brazil (Valentini et al., 2008; Panosso et al., 2009; Souto et al., 2009; Araujo et al., 2011; Ivo and Salcedo, 2012; Correia et al., 2015; Holanda et al., 2015) and in other countries (Deng et al., 2012; Chen et al., 2014; Zheng et al., 2014). These studies used different methodologies to quantify ECO_2, the most common ones being CO_2 absorption in an alkaline solution (AA), in general 0.5 or 1.0 mol L^{-1} KOH or NaOH (Souto et al., 2009; Araujo et al., 2011; Correia et al., 2015). Other studies adopt dynamic measurement made with an infrared gas analyzer (Valentini et al., 2008; Panosso et al., 2009). These methodologies differ in precision, applicability, and spatial and temporal resolution (Janssens et al., 2000; Yim et al., 2002).

The infrared gas analyzer (IRGA) measurement has been considered to be more precise but depends on the availability of relatively expensive equipment and, in general, is conducted over short periods of time (Rochette and Eller, 1991; Haynes and Gower, 1995). The AA methodology is cheaper since it requires only one simple piece of equipment (pH meter) and can integrate CO_2 evolution for many hours, which facilitates nocturnal measurements. These differences are probably the reason why AA has been the most commonly used methodology for ECO_2 measurement in the Northeast region of Brazil (Souto et al., 2009; Araujo et al., 2011; Ivo and Salcedo, 2012; Correia et al., 2015; Holanda et al., 2015). However, some reports point to possible overestimation of low ECO_2 fluxes and underestimation of high fluxes by the AA method (Janssens et al., 2000), although it can be reliably used in the intermediate flux range (Davidson et al., 2002).

Considering that both IRGA and AA methods have been used world-widely, our hypothesis is that they provide similar results when measuring ECO_2 under *Caatinga* or pasture and in different seasons in the Brazilian Northeastern semiarid region. Therefore, we aimed to compare ECO_2 measurements using the AA and IRGA methodologies applied to soils under *Caatinga* (xeric shrubland and thorn forest) and pasture vegetation in the Agreste region of the state of Pernambuco, Brazil.

MATERIALS AND METHODS

The experiment was conducted in two areas, one cultivated with the grass *Brachiaria decumbens* Stapf. and the other a neighboring *Caatinga* area, both located on the Riacho do Papagaio farm (8° 48’ 34.2’’ S; 36° 24’ 29.3’’ W and 702 m altitude) in the municipality of São João in the southern portion of the Agreste mesoregion of Pernambuco. Average annual rainfall is 782 mm, concentrated mainly from May to August (Silva et al., 2014). The climate is predominantly hot and humid, As’ according to the Köppen classification system (Alvares et al., 2014), with dry summers and average temperatures ranging from 18.8 to 22.6 °C (Borges Júnior et al., 2012). The native vegetation is classified as hypo xerophilic *Caatinga* (Santos et al., 2012), and the soil in the experimental area is a sandy *Neossolo Regolítico Eutrófico típico* (Santos et al., 2012) or Entisol Arent (Soil Survey Staff, 2014).

The experimental area was deforested in the 1950s. Part of the area was not cropped, and the native *Caatinga* vegetation has been spontaneously regenerating ever since. Another part of the area was planted with Guatemala grass, followed by cassava and beans (1974-1980), corn (1981-1999), and Brachiaria grass (since 2000) (Almeida et al., 2015). In the pasture area, 24 points were marked for soil ECO_2 determination along two perpendicular transects in a cross shape (one with 14 and the other with 10 points); each point was 15 m distant from the next. In the *Caatinga* area, 12 points were marked along two winding trails.

The experiment followed a completely randomized design with two treatments (the AA and IRGA methods) and 24 replicates in the pasture area and 12 in the *Caatinga* area.

At each point, ECO_2 measurements were made with IRGA between 6:00 and 11:00 a.m. and between 6:00 and 11:00 p.m. once a month from April 2012 to January 2013. The period from April to August 2012 corresponded to the rainy season (total rainfall of 160 mm) and from September 2012 to January 2013 to the dry season (39 mm rainfall). It is important to note that 2012 was a drought year; therefore, rains during the rainy season were below average.

The IRGA apparatus (Licor LI-6400-09) has a gas retention chamber of 991 cm^3, covering a soil surface area of 71.6 cm^2, an infrared irradiator, and a measurement chamber, also described as an optical path and filter plus a detector. The infrared signal traverses the measurement chamber, which is filled with the sampled gas, and it is measured by the detector. The CO_2 emission is calculated by the linear regression of the increase in CO_2 concentration inside the chamber along the measurement period (Davidson et al., 2002). Before beginning measurement, the CO_2 concentration near the soil surface was registered (about 350 μmol mol^{-1}), and this value was introduced in the software system of the apparatus to function as a reference value. At the beginning of each measurement, part of the gas chamber was inserted into the soil, and the CO_2 concentration inside the chamber was reduced to 10 μmol mol^{-1}, flushing the gas through a mixture of calcium oxide and sodium hydroxide. After this reduction, the subsequent CO_2 concentration increases inside the chamber, due to soil emission, were measured every 2.5 seconds, for a total period of 90 seconds, at which time the concentration inside the chamber was approximately 10 μmol mol^{-1} above the reference CO_2 concentration value. After this period, the LI-6400-09 software system calculated the linear regression between the CO_2 emission and its concentration inside the chamber, and the emission was assumed to have the value registered when the concentration inside the chamber was equal to the concentration of the reference value.

On the same days and in the periods when the IRGA measurements were made, AA measurements of soil ECO_2 were made at points close to those marked for the IRGA measurements. The CO_2 evolved from an area of 390.57 cm^2 was absorbed by a 0.5 mol L^{-1} KOH solution placed in a 100-mL beaker with a 4.2 cm diameter opening (13.9 cm^2) under a cylindrical bucket with a 22.3 cm diameter opening and 21 cm height, which was inserted 2 to 3 cm inside the soil to avoid direct gas exchange with the atmosphere. Measurements were made separately during the day (from 6:00 a.m. to 6:00 p.m.) and during the night (from 6:00 p.m. to 6:00 a.m.), integrating the emissions for each 12-hour period. During each measurement, three control measurements were made, placing beakers with 10 mL KOH inside buckets that were hermetically sealed. After each measurement period, the KOH solution was transferred to hermetically sealed flasks to avoid exchange with the atmosphere, and the flasks were taken to the laboratory, where CO_2 was determined by titration with 0.1 mol L^{-1} HCl using 1 % phenolphthalein and methyl orange as pH indicators. The ECO_2 value (mg CO_2 m^{-2} h^{-1}) was converted to μmol CO_2 m^{-2} s^{-1} through multiplication by 0.0063.

In addition to the ECO_2 measurements, ten soil samples were also collected in each area (pasture and *Caatinga*) from the 0.00-0.20 m surface layer to determine some soil properties: total organic carbon (TOC), texture, soil bulk density (SD), total porosity (TP), and macro- and micro-porosity. These determinations were made following the methodologies described by Donagema et al. (2011). The data were subjected to analysis of variance (F-test), and the averages compared by the Tukey test at the 5 % probability level. The identity test proposed by Leite and Oliveira (2002) was used to test if ECO_2 data obtained by the AA and IRGA methodologies were similar. Since the aim of the study was to compare the two methodologies, data from pasture were not compared to *Caatinga*, but only data from one methodology were compared to data from the other methodology in each area separately. These comparisons were made considering the whole period (April 2012 to January 2013), the rainy (April to August 2012) and the dry (August 2012 to January 2013) periods, and the diurnal and nocturnal periods.

RESULTS AND DISCUSSION

Soils from the two areas were not significantly different regarding their particle size fractions, both were classified as sandy. They were also similar regarding soil bulk density (SD), total porosity (TP), macroporosity (Ma), and microporosity (Mi). However, the *Caatinga* soil had a higher total organic carbon (TOC) content than the pasture soil (Table 1). Both contents were within the range usually found in sandy soils of semiarid regions (Santos et al., 2012; Medeiros et al., 2015) and the higher *Caatinga* TOC was not enough to cause a different pore size distribution compared to that from pasture soil. As expected for sandy soils (Santos et al., 2012; Silva et al., 2014; Almeida et al., 2015), macropores predominated over micropores. Texture can influence soil CO_2 movement because it alters porosity (Bouma and Bryla, 2000); more macropores favor soil respiration due to greater penetration of air.

Table 1. Soil physical properties and total organic carbon in areas of *Caatinga* and pasture, in the municipality of São João, Pernambuco, Brazil

	Sand	Silt	Clay	SD	TP	Ma	Mi	TOC
	———	$g\ kg^{-1}$	———	$Mg\ m^{-3}$	———	$m^3\ m^{-3}$	———	$g\ kg^{-1}$
Pasture	888a	72a	41a	1.51a	0.431a	0.315a	0.116a	5.1b
Caatinga	909a	51a	40a	1.50a	0.433a	0.304a	0.129a	8.3a

SD: soil bulk density; TP: total porosity; Ma: macro-porosity; Mi: micro-porosity; TOC: total organic carbon. Sand, silt, clay, SD, TP, Ma, Mi, and TOC were determined following the methodologies described by Donagema et al. (2011). Averages followed by the same letter in the column are not significantly different by the Tukey test at the 5 % probability level.

Monthly variations in soil ECO_2 values obtained from the AA methodology were small, both for diurnal and nocturnal measurements and for *Caatinga* and pasture, with average values around 1.05 μmol CO_2 m^{-2} s^{1} (Figure 1). Values obtained from the IRGA methodology were also similar for the diurnal and nocturnal periods and *Caatinga* and pasture areas, but they differed significantly between the rainy and the dry seasons, both in the *Caatinga* and pasture areas (Figure 1 and Table 2).

Differences between CO_2 evolution during the day and the night may be caused by lower night temperatures, which would result in higher relative humidity and, consequently, higher soil microbial activity (Souto et al., 2009; Holanda et al., 2015). However, no differences were found between day and night in either of the two methods (Table 2). It is possible that, in this region, even at night, soil temperatures remain high enough not to cause effects different from those observed during the day. In addition, soil in the areas has a sandy texture, and its high macroporosity (Table 1) may lead to high diffusion and aeration.

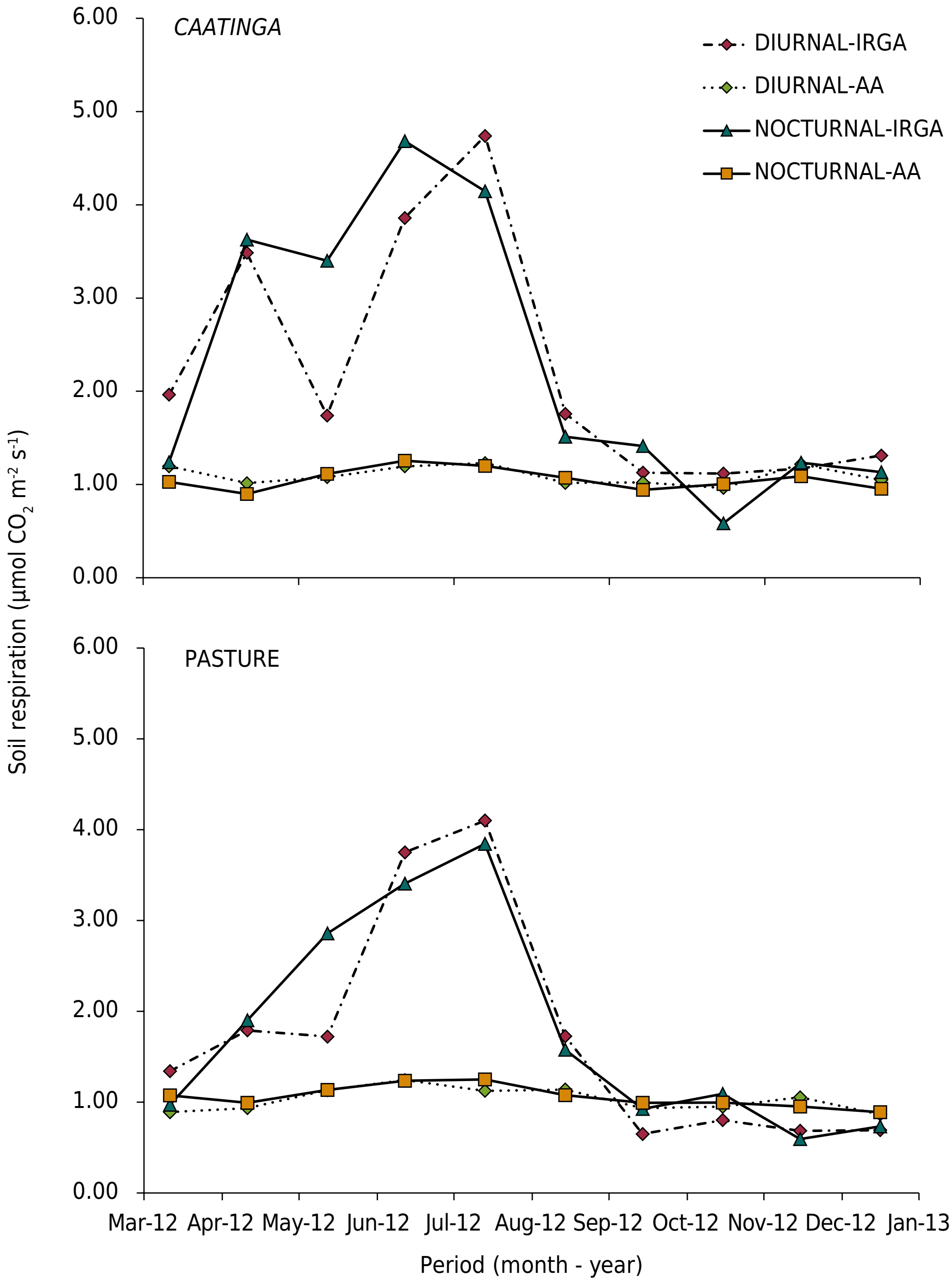

Figure 1. Average soil ECO_2 values, in diurnal and nocturnal periods, measured by infrared gas analyzer and alkali absorption methodologies, in areas of *Caatinga* and pasture, in the municipality of São João, Pernambuco, Brazil

These results confirm those of Correia et al. (2015) who measured ECO_2 using the AA method in *Caatinga* and pasture areas in the semiarid region of the state of Paraíba under climatic and soil conditions similar to those of the present study. Several authors (Souto et al., 2009; Correia et al., 2015; Holanda et al., 2015) report that high soil temperatures limit microbial activity in *Caatinga* soils because they reduce microbial populations and, consequently, reduce the intensity of organic residue decomposition and soil ECO_2.

Soil ECO_2 values determined by IRGA were two to four times higher than those by AA when measured during the rainy season, from April to August 2012, whereas in the dry season (September 2012 to January 2013), values obtained from both methods were similar (Table 2). Considering that ECO_2 varies as a function of climate and soil variables, such

Table 2. Summary of statistical analysis, according to the methodology proposed by Leite and Oliveira (2002), comparing infrared gas analyzer (IRGA) and alkali absorption (AA) methodologies for ECO_2 measurements in areas of *Caatinga* and pasture, in the municipality of São João, Pernambuco, Brazil

	ECO_2 average		Statistical tests			
	μmol m^{-2} s^{-1}					
Period/ Data	IRGA	AA	F(Ho)	$t\bar{e}$	RYjY1≥ (1-|$\bar{e}$|)	Conclusion
Caatinga diurnal						
All	2.22±1.34	1.03±0.10	9237.9*	7.256*	No	Yj ≠ Y1
Rainy	3.16±1.29	1.02±0.09	11158*	15.701*	Yes	Yj ≠ Y1
Dry	1.28±0.40	1.05±0.10	233.8*	1.895ns	No	Yj ≠ Y1
Caatinga nocturnal						
All	2.32±1.47	1.06±0.12	8096.9*	4.535*	No	Yj ≠ Y1
Rainy	3.47±1.21	1.10±0.14	5922.2*	15.605*	Yes	Yj ≠ Y1
Dry	1.18±0.44	1.02±0.09	299.5*	0.126ns	No	Yj ≠ Y1
Pasture diurnal						
All	1.80±1.41	1.04±0.18	2142.1*	1.133ns	No	Yj ≠ Y1
Rainy	2.69±1.48	1.09±0.14	1663.1*	10.443*	No	Yj ≠ Y1
Dry	1.03±0.40	0.91±0.09	97.3*	2.662*	No	Yj ≠ Y1
Pasture nocturnal						
All	1.77±1.16	1.06±0.13	4944.4*	2.112ns	No	Yj ≠ Y1
Rainy	2.55±1.15	1.14±0.15	3453.4*	6.041*	No	Yj ≠ Y1
Dry	1.06±0.44	0.98±0.10	465.6*	1.660ns	No	Yj ≠ Y1

ns: not significant; *: significant at the 5 % probability level; Yj ≠ Y1: the AA methodology differs from the IRGA methodology.

as rainfall, air temperature, and soil moisture and temperature (Davidson et al., 2000; Deng et al., 2012; Chen et al., 2014), it was expected that ECO_2 values measured by AA would change significantly from the wet to the dry season, as they did when measured by IRGA (Figure 1 and Table 2). However, the average values measured by AA were only 10 % higher in the rainy than in the dry season, whereas those measured by IRGA were more than 260 % higher in the rainy season. Therefore, the IRGA methodology is more sensitive to seasonal variations than the AA methodology. Martins et al. (2010) stated that higher water availability during the rainy season should result in higher biological activity. Therefore, higher ECO_2 should also have been registered during the rainy season in this area in the Agreste region of the state of Pernambuco, which was only observed using the IRGA methodology (Figure 1 and Table 2).

Measurements performed in other *Caatinga* areas (Souto et al., 2009; Correia et al., 2015; Holanda et al., 2015), all of them using the AA methodology, registered small (0.28 μmol CO_2 m^{-2} s^{-1}) and high amplitudes (0.75 μmol CO_2 m^{-2} s^{-1}) between maximum (1.00 to 1.32 μmol CO_2 m^{-2} s^{-1}) and minimum values (0.57 to 0.72 μmol CO_2 m^{-2} s^{-1}). It is not possible to know if the similarity of ECO_2 values throughout the year, reported by some of these articles, was due to absence of environmental variables that substantially altered root respiration and soil microbial activity, or if it was caused by a limitation of the methodology. Nonetheless, in this study, it is more likely that rainfall variation was enough to explain the high variations in ECO_2 obtained from the IRGA methodology.

Considering the extreme difference in water availability usually observed between the rainy and the dry season in the Northeastern semi-arid region, and the effect that this availability has on root and soil microbial activities (Souto et al., 2009), large differences in ECO_2 are expected. Therefore, a doubt arises as to whether AA measurements during the rainy season of the semiarid region were not underestimated. In other Brazilian regions where measurements were also made with the AA methodology (Assis Júnior et al., 2003; Valentini et al., 2015), including forest and *Cerrado* (Brazilian tropical savanna) areas, there are no cases of low amplitude in ECO_2 values. The highest values were at least 50 % higher than the lowest, but were as much as eight times higher.

Since no ECO_2 measurements made with IRGA in the Northeast region were found in the literature beyond those of the present study, comparisons are not possible. However, measurements in forest, *Cerrado*, and agricultural areas in other Brazilian regions (Sotta et al., 2004; Valentini et al., 2008; Panosso et al., 2009; Pinto-Junior et al., 2009; Zanchi et al., 2012) report higher amplitudes (2.51 to 6.16 μmol CO_2 m^{-2} s^{-1}) between maximum (3.96 to 10.51 μmol CO_2 m^{-2} s^{-1}) and minimum (1.45 to 4.35 μmol CO_2 m^{-2} s^{-1}) values than those measured with the AA methodology. The smallest difference (about 50 %) occurred in a sugarcane field in Mato Grosso do Sul (Moitinho et al., 2013); all other maximum values were two to five times higher than the minimum values. These findings suggest that the IRGA methodology has higher sensitivity in detecting ECO_2 differences than the AA methodology. However, these are comparisons derived from few studies and under different climate and soil conditions, and it cannot be confirmed that the AA methodology was not adequate under the conditions in which it was used.

Several studies point to advantages in using the AA methodology: Assis Júnior et al. (2003) and Souto et al. (2009) highlighted that it is a simple, low cost, and sensitive method that can be applied simultaneously at many points and that can integrate CO_2 evolutions over many hours (even 24 hours), therefore reducing spatial and temporal variability. If it is not possible to use an IRGA, which requires equipment that is expensive, has high maintenance costs, and demands a specially trained operator, use of the AA methodology is justified. However, many authors, especially those working in other countries, have claimed that the AA methodology can underestimate ECO_2 values (Haynes and Gower, 1995; Jensen et al., 1996; Yim et al., 2003).

There are several explanations for these underestimations. The underestimations might result from the measuring chamber remaining in the same spot for a long time, which could disturb the micro-climate, demands the daily temperature amplitude (Jensen et al., 1996). However, Hendry et al. (2001) performed measurements for periods longer than 24 hours and reported values similar to those obtained from other methodologies. Absorption of CO_2 by the alkaline solution could also decrease over the measurement period due to low diffusion (Freijer and Bouten, 1991), the CO_2 concentration inside the chamber decreasing the diffusion gradient between the soil and air inside the chamber, reducing the flux absorbed by the alkaline solution. Under laboratory conditions, Freijer and Bouten (1991) observed that static absorption was not sufficient to react with all the respired CO_2, due to lack of contact between the solution and the entire CO_2 volume inside the chamber. One or more of these problems probably occurred in the present study since the ECO_2 values obtained by the AA methodology were underestimated and did not reflect the changes that happened from the dry to the rainy season. However, it is not possible to know for sure if those problems occurred during the present study. Considering that the methodology was applied in a way similar to other studies using AA, similar problems could have occurred in these other studies, which were not detected due to the absence of comparisons with other methodologies.

The IRGA methodology has been criticized because it does not integrate CO_2 fluxes over long time periods, as the AA methodology does, since measurements last only a few minutes (Davidson et al., 2002). To extend these periods, it would be necessary to perform a large number of measurements (Panosso et al., 2012). According to Perez-Quezada et al. (2016), this is possible, and despite the high cost of the equipment, the IRGA methodology has a better cost/benefit ratio than others.

CONCLUSIONS

Soil ECO_2 measurements with the infrared gas analyzer (IRGA) methodology were more sensitive to seasonal variations than those with the alkaline absorption (AA) methodology. Soil ECO_2 values measured with the IRGA were significantly higher during the rainy than the dry season, differing from those obtained from AA, which had little or no differences between seasons.

In sandy soils, under semiarid conditions, the IRGA methodology is recommended, since the AA methodology underestimates ECO_2 during periods of high CO_2 evolution.

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