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SOIL CO_2 FLUX: A METHOD COMPARISON OF CLOSED STATIC CHAMBERS IN A SUGARCANE FIELD[1]

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SUMMARY

A large variety of techniques have been used to measure soil CO_2 released from the soil surface, and much of the variability observed between locations must be attributed to the different methods used by the investigators. Therefore, a minimum protocol of measurement procedures should be established. The objectives of this study were (a) to compare different absorption areas, concentrations and volumes of the alkali trapping solution used in closed static chambers (CSC), and (b) to compare both, the optimized alkali trapping solution and the soda-lime trapping using CSC to measure soil respiration in sugarcane areas. Three CO_2 absorption areas were evaluated (7; 15 and 20 % of the soil emission area or chamber); two volumes of NaOH (40 and 80 mL) at three concentrations (0.1, 0.25 and 0.5 mol L^{-1}). Three different types of alkaline traps were tested: (a), 80 mL of 0.5 mol L^{-1} NaOH in glass containers, absorption area 15 % (V0.5); (b) 40 mL of 2 mol L^{-1} NaOH retained in a sponge, absorption area 80 % (S2) and (c) 40 g soda lime, absorption area 15 % (SL). NaOH concentrations of 0.5 mol L^{-1} or lower underestimated the soil CO_2-C flux or CO_2 flux. The lower limit of the alkali trap absorption area should be a minimum of 20 % of the area covered by the chamber. The 2 mol L^{-1} NaOH solution trap (S2) was the most efficient (highest accuracy and highest CO_2 fluxes) in measuring soil respiration.

Index terms: alkaline traps, carbon, soil respiration.

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RESUMO: *FLUXO DE CO_2 NO SOLO: UMA COMPARAÇÃO DE MÉTODOS ESTÁTICOS EM ÁREA DE CULTIVO DE CANA-DE-AÇÚCAR*

Uma grande variedade de técnicas tem sido aplicada na medição do fluxo de CO_2 dos solos, e muito da variabilidade observada entre locais deve ser atribuída às diferenças metodológicas utilizadas pelos investigadores, sendo importante a busca de um protocolo mínimo nos procedimentos dessas medições. O objetivo do presente estudo foi: (a) comparar diferentes áreas de captação, concentrações e volumes de solução alcalina para uso em câmaras estáticas fechadas (CSC); e (b) comparar duas técnicas de absorção alcalina de CO_2, armadilhas com solução alcalina de NaOH e armadilhas com mistura granulada de $Ca(OH)_2$, usando CSC, para medição da respiração do solo, em áreas de cultivo de cana-de-açúcar. Foram avaliadas três áreas de captação de CO_2: 7, 15 e 20 % da área de emissão do solo (câmara); dois volumes de NaOH: 40 e 80 mL; e três concentrações: 0,1, 0,25 e 0,5 mol L^{-1}. Três diferentes tipos de armadilhas alcalinas foram testados: (a) NaOH, 0,5 mol L^{-1}, 80 mL em recipientes de vidro, área de captação de 15 % (V0.5); (b) NaOH, 2 mol L^{-1}, utilizando-se 40 mL retidos em espuma, área de captação de 80 % (S2); e (c) cal sodada 40 g em uma área de 15 % (SL). Baixas concentrações de NaOH subestimaram o fluxo de CO_2 do solo, recomendando-se concentrações ≥ 0,5 mol L^{-1}.O limite inferior para a área de captação do CO_2 deve ser ≥ 20 % da área coberta pela câmara. A armadilha com solução de NaOH 2 mol L^{-1} (S2) foi a mais eficiente (maior exatidão e maiores fluxos) nas medições da respiração do solo.

Termos de indexação: armadilhas alcalinas, carbono, respiração do solo.

INTRODUCTION

Carbon dioxide emissions increase global warming, and to understand how the global C cycle responds to human intervention and to climate change itself it is necessary to quantify C pools and fluxes, including soil respiration. The latter accounts for a great part of the total respiration of the biosphere and is the second largest flux of terrestrial ecosystems (Luo & Zhou, 2006). The soil-atmosphere CO_2 flux results from the respiration of plant roots and microorganisms. This flux provides information about (i) the physiological state or catabolic potential of soil microbes; (ii) the decomposition of specific organic substrates in the soil (iii) soil biomass, and (iv) the relative contribution of microbes, fauna, plant roots and abiotic sources to the total flux of soil C (Zibilske, 1994).

Studies in this area show that a great variety of techniques have been used to measure the levels of soil CO_2 fluxes (Raich & Nidelhoffer, 1989; Raich & Schlesinger, 1992; Jensen et al., 1996). According to some of these authors, much of the variability observed between locations must be attributed to the differences of methods used by the investigators. According to Luo & Zhou (2006), there is still no universal consensus about which method is the best and which could be used as the “gold standard” to measure soil respiration. These authors also state that, in spite of this, many comparison studies state that the dynamic open method has been indicated as the most reliable, though rather complicated in terms of controlling the pressure within the chamber, requiring substantial technical investment.

In this sense, comparisons between static absorption methods in an alkali medium with dynamic methods have shown that, when the flux of CO_2 is high, the measurements in the closed static chambers (CSC) underestimate values in relation to the dynamic chambers (Jensen et al., 1996; Chavez et al., 2009). A variant of this method (CSC) is a procedure where gas is sampled from the chambers with syringes, for laboratory analysis using a gas chromatograph. According to Lou & Zhou (2006), gas chromatography can also underestimate the CO_2 flow rate in comparison with other methods by up to 45 %. These authors explain that building up CO_2 concentrations in the closed chamber, with a reduction of the CO_2 concentration gradient and, then, decrease in the CO_2 release, can be one explanation of this underestimation. So, the measurement period also significantly affects the flux rates. The advantage of the method is that the fluxes of several gas species can be measured from the same gas sample.

However, when some features of the chamber design, such as the absorption area and the alkaline medium, are observed, the differences between these static and dynamic methods are reduced or compensated (Yim et al., 2002). In view of this possibility and the difficulty of obtaining devices for a dynamic determination of soil CO_2 fluxes, it is necessary to improve one of the most popular types of methods used to determine CO_2, the static and based on the absorption of CO_2 through an alkaline medium, in its solid or liquid form, followed by gravimetric, conductivity, or titration analyses.

One of the fundamental aspects of determining these fluxes is the area of CO_2 absorption. Research addressing the determination of global carbon fluxes as of Raich & Nadelhoffer (1989) and Raich & Schlesinger (1992) disregards data generated when the surface area of the container holding the alkaline absorbent is less than 6 and 5 % of the area covered by the chamber. These data lead to low estimates of soil respiration. Yim et al. (2002) suggested that a high ability to absorb CO_2 can be maintained in these static chambers by using sponges saturated with an alkaline solution, provided they cover about the same area of the chambers. Similarly, the concentration of alkaline solutions may also vary considerably. The recommended values range from 0.1 mol L^{-1} (Anderson & Ingram, 1993), to 0.2; 0.5 (Jensen et al., 1996; Mendonça & Matos, 2005); 1.0 (Chavez et al., 2009) and 2 mol L^{-1} (Yim et al., 2002), which may interfere with the establishment of the CO_2 concentration gradient, by processes that occur at the gas-liquid interface. Low soil respiration may also be estimated as a consequence of a deeper settlement of the chambers, resulting from cutting the roots (Raich & Nidelhoffer, 1989) or from the lateral CO_2 flux (Hutchinson & Rochete, 2003).

The objectives of the study were to: (a) compare different absorption areas, concentrations and volumes of the alkali solution used in CSC, and (b) compare the optimized alkali trapping solution and the soda-lime trapping using CSC, to measure soil respiration in sugarcane areas.

MATERIAL AND METHODS

Locations, soil and climate

The study was conducted in an area of the sugar cane mill Triunfo, in the city of Boca da Mata, Alagoas (09°40' S; 36° 08' W). The soil in the area was classified as Ultisol, with sandy loam (172 g kg^{-1} clay) in the 0–20 cm layer to sandy clay texture (392 g kg^{-1} clay) in deeper layers. The average annual rainfall is 1600 mm, concentrated between May and September, and the air temperature about 28 °C. Sugarcane is the main crop in the region, and was harvested in the experimental area without burning the leaves.

CO_2 flux measurements

The closed chamber to measure the CO_2 flux had an absorption area of 346.2 cm^2 (diameter 21 cm, height 9 cm). Following straw removal, the chambers were placed on the soil surface, covering the alkali trap (NaOH), and were then closed with soil. The traps were suspended by a wire at a height of two centimeters above the ground. The alkali traps were left in the field for periods of 24 h, thus avoiding daily fluctuations in the CO_2 flux estimates. The alkali was transferred to Falcon plastic tubes, with lids reinforced by polytetrafluoroethylene (PTFE) film, and taken to the laboratory. On the same day or one day after sampling, total CO_2 was determined by titration with HCl from pH 8.3 to 3.7 (Sampaio & Salcedo, 1982). In traps where soda lime was used, gravimetric values were used for the determination, and the lime was oven-heated to 105 °C, for 24 h. The amount of CO_2 retained was corrected considering the CO_2 of the chamber environment, using traps in closed chambers, which were not exposed to the soil.

Experiment 1: This experiment was arranged in a completely randomized design with three replicates and consisted of three treatments (alkali volume, alkali concentration and absorption area) in an incomplete factorial design: two alkali volumes (40 and 80 mL), combined with three alkali concentrations (0.1; 0.25 and 0.5 mol L^{-1}). The factorial combinations using the 40 mL volume were combined with three CO_2 absorption areas: 7 % (vessel height 5.0 cm, diameter 5.5 cm); 15 % (height 5.0 cm, diameter 8.0 cm); and 20 % (height 1.5 cm, diameter 9.5 cm) of the soil emission area - chamber area) while those with 80 mL alkali volume were combined with only two absorption areas (7 and 15 %). The third treatment could not be tested because no vessel with an absorption area 20 % and 80 mL capacity was available.

Experiment 2: After testing and selecting the volume of 80 mL of 0.5 mol L^{-1} NaOH, different types of traps were compared in a second experiment, assessed on seven dates during sugarcane growth (05/15/2009, 06/05/2009, 01/12/2010, 03/18/2010, 04/07/2010, 04/23/2010 and 06/18/2010) when soil moisture at the surface (0-10cm) was 0.17, 0.17, 0.10, 0.08, 0.13, 0.14 and 0.28 cm^3 cm^{-3}. Three alkali traps were compared: (a) 80 mL of 0.5 mol L^{-1} NaOH placed in a shallow glass (height 5.0 cm, diameter 8.0 cm) with an absorption area of 15 % of the chamber area (346 cm^2) (V0.5); (b) a wetted sponge (height 3.0 cm, diameter 13.0 cm) with 40 mL of 2 mol L^{-1} NaOH, with an absorption area of 80 % (chamber area = 177 cm^2) (S2) and (c) 40 g of soda lime,a granulate mixture of hydrated lime ($Ca(OH)_2$,approximately 80 %) with a small quantity of sodium hydroxide (NaOH,3.7 %), placed in a shallow glass (height 5.0 cm, diameter 8.0 cm) with an absorption area of 15 % (chamber area = 346 cm^2) (SL). The CO_2 flux values of the V0.5 trap were lower on the first two dates, so on the following dates only S2 and SL were compared, with nine replicates per treatment.

Data were subjected to analysis of variance and the means compared using the Tukey test, at 5 %.

RESULTS

Experiment 1

The average CO_2 flux ranged between 19 and 92 mg m^{-2} h^{-1} of CO_2-C, with a maximum coefficient of variation (CV) of 24.8 % (Figure 1). The CO_2 flux increased more than 65 % with the increase in the alkali trap concentration. An average increases in CO_2 fluxes due to changes in alkali volume and in absorption area ranged between 27 and 21 %, respectively. Curve trends (Figure 1) indicate a tendency towards stabilization of the flux measured by the alkali trap with 0.5 mol L^{-1}.

At a volume of 40 mL NaOH, a significant interaction was observed (Figure 1) between concentration and absorption areas ($p < 0.05$), since at lower alkali concentrations, the absorption area had no effect on the CO_2 flux, whereas for the 0.5 mol L^{-1} concentration, significant increases in CO_2 fluxes were observed with the area increase from 7 to 15 % and from 15 to 20 %. Therefore, no significant differences among absorption areas were found at 0.1 mol L^{-1} NaOH, with CO_2 fluxes from 19.3 to 23.5 mg m^{-2} h^{-1} CO_2-C. In contrast, differences were significant at 0.25 mol L^{-1} (7 and 15 %), and 0.5 mol L^{-1} (7, 15 and 20 %).

With the increase in alkali volume to 80 mL, both treatment combinations had significant effects on the measured CO_2 flux ($p < 0.05$). However, fluxes were lower in the treatment that combined a smaller absorption area with a lower concentration (Figure 1).

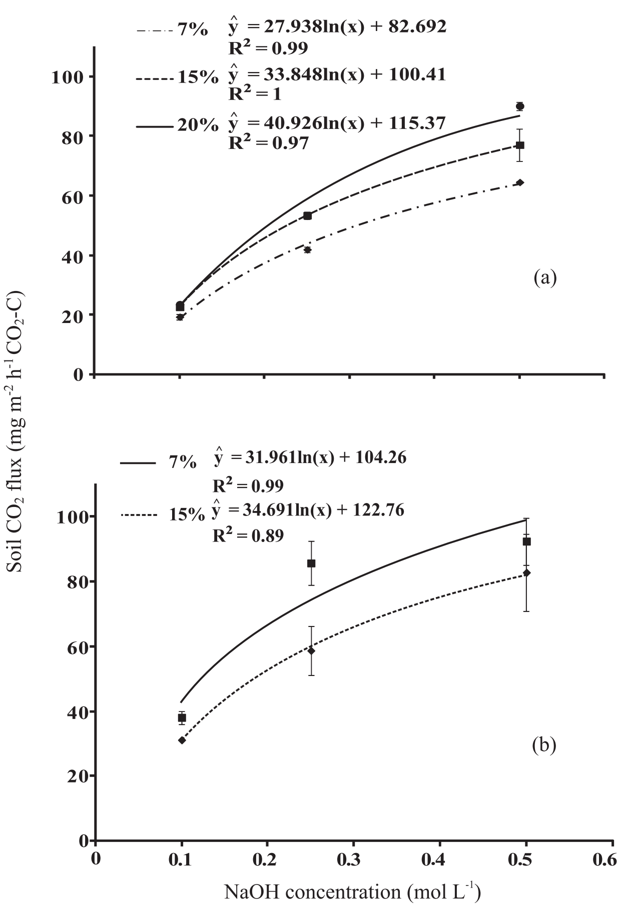

Figure 1. Soil CO_2 flux as a function of the NaOH concentration (0.1, 0.25 and 0.5 mol L^{-1}) and the absorption area (7, 15 and 20% of the chamber area), using 40 mL (a) and 80 mL (b) NaOH in a sugarcane area. Vertical bars represent the mean standard error.

Experiment 2

In the first two tests, a significant difference ($p < 0.05$) was found between the trap types (Figure 2). On the first date, the CO_2 fluxes were 80, 160 and 215 mg m^{-2} h^{-1} of CO_2-C, for V0.5; S2 and SL, respectively. In the second test, much lower values (53 mg m^{-2} h^{-1} of CO_2-C) were obtained once more for the V0.5 trap, whereas the S2 trap measured fluxes of 140 mg m^{-2} h^{-1} of CO_2-C and the SL of 143 mg m^{-2} h^{-1} of CO_2-C.

Because of the low fluxes measured with trap V0.5, its use was discontinued. The additional measures with traps S2 and SL (Figure 3) showed no consistent pattern in soil CO_2 fluxes for either method. However, trap S2 showed a greater frequency of higher fluxes than SL. The average values for all sampling dates were 169 and

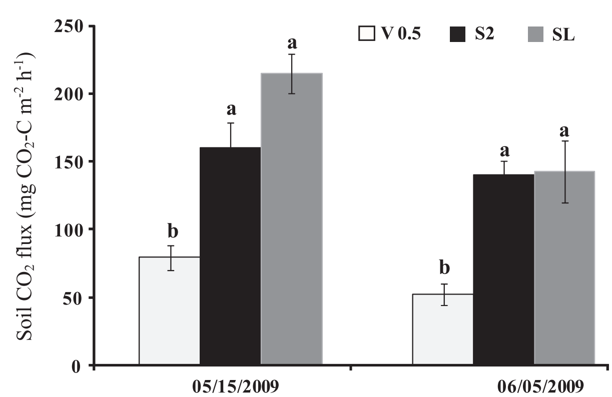

Figure 2. Soil CO_2 flux as a function of alkali traps: V0.5 (80 mL of 0.5 mol L^{-1} NaOH in glass container), S2 (sponge 40 mL of NaOH, 2 mol L^{-1}), SL (40 g of soda lime in glass container) in sugarcane areas. Vertical bars represent the mean standard error. On each date, the averages followed by the same letter showed no difference by the Tukey test at 5 %.

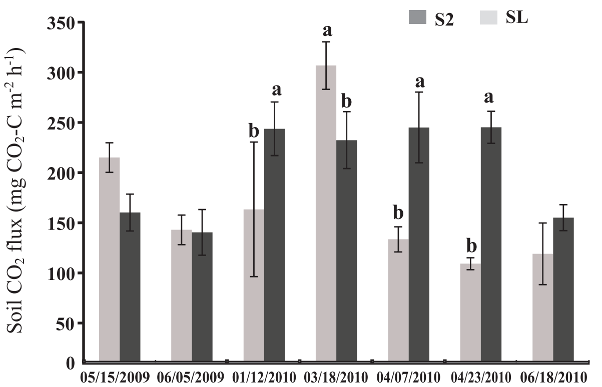

Figure 3. Soil CO_2 flux as a function of methods: S2 (sponge, 40 mL NaOH, 2 mol L^{-1}), SL (40 g of soda lime in a glass container) in sugarcane areas. Vertical bars represent the mean standard error. On each date, the averages followed by the same letter showed no difference by the Tukey test at 5 %.

203 mg m^{-2} h^{-1} of CO_2-C for SL and S2, respectively. The CV values for SL were around 59 %, ranging between 18 and 100 % and lower for S2, around 40 %, ranging between 20 and 45 %.

DISCUSSION

The lowest fluxes measured in traps with lower NaOH concentrations (0.1 and 0.25 mol L^{-1}) and smaller absorption area (7 % of the chamber area) indicate that the recommendation of 0.1 mol L^{-1}, for field measurements (Anderson & Ingram, 1993) will underestimate the CO_2 flux, as do absorption areas close to 7 %. Raich & Nadelhoffer (1989) claimed that in static systems, soil CO_2 flux estimates are consistently low, when the absorption area of the alkali traps is less than 6 % of the chamber area. In this sense, when Raich & Schlesinger (1992) estimated the global CO_2 flux of terrestrial ecosystems, they excluded all published soil respiration data using alkali trap areas < 5 % of chamber areas. An even higher limit, of about 16 %, was mentioned by Yim et al. (2002) as the cause of underestimated soil CO_2 fluxes. Results of the present study show the need to use alkali trap areas even greater than this 16 % limit, with alkali concentrations of ≥ 0.5 mol L^{-1}. Hutchinson & Rochette (2003), regarding the optimization of measurements using static methods, suggested a 20 % ratio between the trap area and emission area of soil CO_2.

The coefficients of variation (CV) of the CO_2 fluxes were within the limits established in other studies (Jensen et al., 1996; Yim et al., 2002). According to Jensen et al. (1996), the CV values are typically within an interval of 10–60 %, occasionally reaching over 100 %, being 1.5–2 times greater in the dynamic than the static methods. In areas where sugarcane is harvested raw and burned, Panosso et al., (2009) found CV values between 22 and 63.5 %, using the dynamic method.

Some aspects related to the use of soda lime should be observed, in an attempt to reduce the high CVs. As the determination is based on a difference in mass, the container used in the oven should be the same as in the field, thus avoiding the transfer of soda lime among containers and a potential loss of material. Another aspect is related to the CO_2 absorption capacity of lime. This absorption capacity decreases every time the lime is used, requiring a change of the soda lime when a threshold of 28 % of the original dry weight is reached. It is, however, recommended that soda lime should be disposed of when the total weight (after drying) is 7 % of the original value, i.e., a accurate weighing and drying are prerequisites for an adequate CO_2 flux estimation based on soda lime (Zibilske, 1994).

The estimates of CO_2 fluxes obtained by the S2 and SL traps are similar to those found by Campos (2003), who monitored soil CO_2 flux using gas chromatography to determine the C balance in sugarcane. The fluxes determined by the alkali traps are closer to those presented by this author for the rainy months in São Paulo. On the other hand, the monitoring performed by Panosso et al. (2009) using dynamic chambers in raw sugar cane areas, measured approximately 85 mg m^{-2} h^{-1} of CO_2-C, which is lower than the CO_2 values in this study. It is likely that the differences were related to the lower soil temperature during the monitoring period (16–24 ºC, Panosso et al., 2009), whereas in the present study temperatures oscillated between 27 and 30 ºC. However, if compared to the fluxes determined by La Scala Jr. et al. (2006) (2.18–1.11 g m^{-2} h^{-1} CO_2-C), immediately after soil tillage for sugarcane planting, the fluxes of the present study were lower, as they were measured in the middle of the ratoon cane. These contrasts show that the environmental conditions (climate, soil, land relief), crop management (soil preparation, burning, phase of the crop cycle), besides the CO_2 quantification method itself, are important aspects when comparing fluxes.

In a forest area, it was found that the S2 alkali trap showed similar fluxes compared to the dynamic method, when determining the annual average soil CO_2 flux (Yim et al., 2002). According to the authors, the highest values estimated by the static method in the period in which the flux was below 300 mg h^{-1} m^{-2} CO_2 (81 mg CO_2-C), were compensated by the underestimation during the summer months, when the values were above 300 mg. The CSC overestimation in low fluxes could be caused by a reduction in the CO_2 concentration in the chamber, where the CO_2 absorption rate of the NaOH solution is higher than the production rate in the soil (Yim et al., 2002). The greater CO_2 gradient between the chamber and the soil would increase the rate of gas diffusion into the chamber. In addition, according to these authors, a low CO_2 concentration in the chamber would accelerate the microbial respiratory activity, thus increasing the measured fluxes.

The high concentration of NaOH solution in the E2 trap (2 mol L^{-1}), along with an absorption area of 80 %, are likely to have caused a greater CO_2 gradient in the chamber, when the flux was smaller. Yim et al. (2002) commented that this method, compared to other static methods, apparently tends to overestimate CO_2 fluxes more at low soil respiration, but underestimate them less at high soil respiration. However, the generation of this gradient is not necessarily a drawback, since, for Hutchinson & Rochette (2003), the main advantage of the E2 trap is that it allows enough time to adjust to the CO_2 diffusion gradient, so the gas absorption rate of the alkaline trap can reach a balance close to the subsurface production rates. Also according to these authors, the method represents a simple and cost-effective way of making reliable soil respiration assessments, which are rapid – usually last 24 h, even at remote locations. This allows the conclusion that the potential drawbacks of static chamber systems are frequently exaggerated.

In their review on assessment and estimation methods of CO_2 fluxes, Luo & Zhou (2006) stated that the alkali trap method is considered efficient and cost-effective, which makes it appropriate for the numerous field measurements required in view of the vast spatial heterogeneity of surface CO_2 fluxes.

CONCLUSIONS

1. NaOH concentrations of 0.5 mol L^{-1} or lower underestimated the soil CO_2 flux.

2. The lower limit of the alkali trap absorption area should be 20 % of the area covered by the chamber.

3. The 2 mol L^{-1} NaOH solution trap was the most efficient (greatest accuracy and highest CO_2 flux) in soil respiration measurement.

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