



Development and field application of a low-cost device for sampling greenhouse gas fluxes at the air-water interface in limnological studies

Desenvolvimento e aplicação de dispositivo de baixo custo para a amostragem de fluxos de gases de efeito estufa na interface ar-água em estudos limnológicos

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Cite as: Coelho, L.H.G. et al. Development and field application of a low-cost device for sampling greenhouse gas fluxes at the air-water interface in limnological studies. *Acta Limnológica Brasiliensia*, 2025, vol. 37, e28. <https://doi.org/10.1590/S2179-975X4023>

Abstract: Aim: The objective of the present study was to develop and field-apply a simple device for sampling greenhouse gas flows (CO_2 and CH_4) at the air-water interface. **Methods:** The device consisted of a cylindrical chamber made of high-density polyethylene with a valve to collect gases. The chamber sealing materials (silicone rubber and epoxy resin) and the system configuration (area/volume ratio A/V, and influence of ventilation) were evaluated. The device was applied in five field campaigns ($n = 45$). The samples were stored in gasometric containers until analysis by gas chromatography with flame ionization detection. **Results:** The epoxy resin sealed the chambers better, while the non-vented device with a higher A/V ratio showed better mixing with fewer uncertainties in gas diffusion through boundary layer disturbance. The flow rates of the target gases varied greatly, from below the limit of quantification for CH_4 ($< 0.062 \text{ mg m}^{-2} \text{ min}^{-1}$) to $0.214 \text{ mg m}^{-2} \text{ min}^{-1}$, and from 0.3 to $42.3 \text{ mg m}^{-2} \text{ min}^{-1}$ for CO_2 . **Conclusions:** These chambers minimize disturbances to the water body and the natural gas exchange processes obtaining more representative data on the natural emissions. Our floating chamber device proved robustness and versatility for determining gas flows at the air-water interface. However, its use must be evaluated in preliminary field work to define the sampling interval time, uncertainties and main analytical challenges to be overcome.

Keywords: carbon dioxide (CO_2); diffusive chamber; GHGs; methane (CH_4); wastewater pollution.

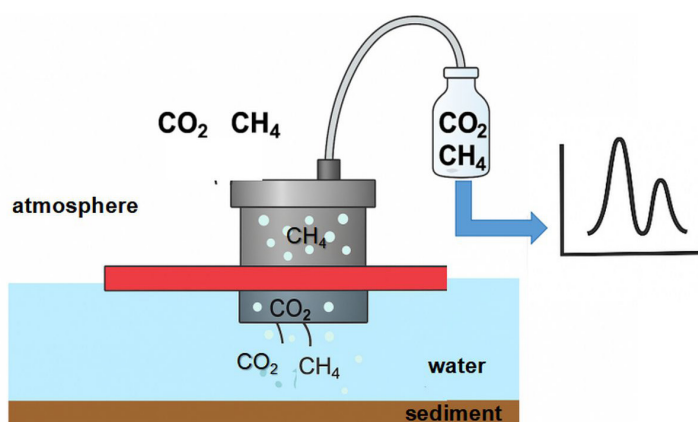
Resumo: Objetivo: O presente estudo teve por objetivo o desenvolvimento e aplicação em campo de um dispositivo simples para amostragem de fluxos de gases de efeito estufa (CO_2 e CH_4) na interface ar-água. **Métodos:** O dispositivo era composto por uma câmara cilíndrica de polietileno de alta densidade com válvula para coleta dos gases. Foram avaliados os materiais de vedação da câmara (borracha de silicone e resina epóxi) e a configuração do sistema (relação área/volume A/V e influência da ventilação). O dispositivo foi aplicado em cinco campanhas de campo ($n = 45$). As amostras foram armazenadas em recipientes gasométricos até a análise por cromatografia gasosa com detecção de ionização em chama. **Resultados:** A resina epóxi selou melhor as câmaras, enquanto o dispositivo não ventilado e com relação A/V mais alta apresentou melhor mistura com menos incertezas na difusão



do gás através da perturbação da camada limite. As taxas de fluxo dos gases alvo variaram muito, desde abaixo do limite de quantificação para CH_4 ($< 0,062 \text{ mg m}^{-2} \text{ min}^{-1}$) a $0,214 \text{ mg m}^{-2} \text{ min}^{-1}$, e de $0,3$ a $42,3 \text{ mg m}^{-2} \text{ min}^{-1}$ para CO_2 . **Conclusões:** Estas câmaras minimizam as perturbações do corpo hídrico e dos processos de troca de gases naturais, obtendo dados mais representativos sobre as emissões naturais. Nosso dispositivo de câmara flutuante mostrou-se robusto e versátil para determinar fluxos de gases na interface ar-água. Entretanto, seu uso deve ser avaliado em trabalhos preliminares de campo para definição do tempo de intervalo de amostragem, incertezas e principais desafios analíticos a serem superados.

Palavras-chave: dióxido de carbono (CO_2); câmara difusional; GEE; metano (CH_4); poluição por águas residuárias.

Graphical Abstract



1. Introduction

Gas flowrates on the air-water interface can be estimated by several methods (Cole et al., 2010): (i) floating chambers for accurate and representative measurements of gas emissions; (ii) boundary layer approach supported by gas concentrations in water and air, wind velocity, and temperature measurements; (iii) Eddy covariance technique, which requires vertical velocity measurement fluctuations in combination with gas concentrations variation in high temporal resolution using automatic devices; and (iv) adding an inert gas marker (e.g. SF_6) and observing its decrease with time to estimate the transfer rate.

The floating chamber method is the most used technique for studies in aquatic environments due to its low cost, operational simplicity, and ease of implementation in several sites, especially in remote locations or with difficult access (Kutzbach et al., 2007). Such advantages spatially increase the number of measurements, giving greater coverage and quality of measures compared to other techniques (Cole et al., 2010; Martinez-Cruz et al., 2015; Mannich et al., 2019).

In these systems, the diffusive gas flows are estimated by the increase in the concentration of the gas confined within the chamber for a while in a given cross-section (UNESCO, 2010; Mannich et al., 2019). Approaches reported for gas sampling in the air-atmosphere interface are based on containment methods. These strategies involve the usage of a specific floating sampling device that hoods and isolates a portion of the subsurface water and, consequently, collects the gas diffused into the atmosphere. The more consolidated technique is the U. S. Environmental Protection Agency method (Klenbusch, 1986), which requires the use of a floating chamber flushed with a neutral gas flow (e.g. N_2 or purified air) (Gebert et al., 2011; Di Trapani et al., 2013). Another way adopted by the United Kingdom Environment Agency (SEPA, 2004) involves the adaptation of a field static chamber in which the increase of gas concentrations is measured over time.

Environmental regulations and standards for using floating chambers to collect methane may vary depending on the jurisdiction and specific usage.

The IPCC provides standardized methodologies for measuring and reporting greenhouse gas emissions. Floating chambers must comply with these guidelines to ensure data accuracy and comparability. The International Organization for Standardization (ISO) standard specifies principles and requirements for quantifying and reporting greenhouse gas emissions and removals. Using floating cameras must be aligned with these principles to ensure data quality (ISO, 2018). Also, ASTM International provides standardized methods for measuring GHG emissions from landfills, which can be adapted for use in floating chambers in bodies of water (ASTM, 2012). The main issue with these regulations is the necessity of periodic calibration and validation studies to ensure that floating chamber measurements remain accurate and reliable.

One of the main concerns of using floating chambers to collect gases at the air-water interface comes from the uncertainties related to disturbances caused at the interface by the restriction of the atmospheric boundary layer, which changes the gas diffusive transfer rate. Dumestre et al. (1999) consider that measurements using floating chambers are realistic as long as the aquatic boundary layer is not affected by the chamber itself, decreasing the renewal rate of the air-water interface in comparison to the atmospheric boundary layer. Other factors related to sampling uncertainties are the ratio between chamber volume and area, the geometry (Eklund, 1992), the device material (Hutchinson & Livingston, 2001), and chamber leakage. Moreover, the temperature effect on sampling accuracy is still controversial (Christiansen et al., 2011; Minke et al., 2016).

Considering the importance of the quantification of gas flows in air-water interface, such as greenhouse

gases (GHG) (Deemer et al., 2016; Prairie et al., 2018), this study is based on the premise that floating static chambers (SC) can be more robust than flushed chambers (FC) with inert gas since air movement into the chamber causes diffusive resistance reduction, which decreases the boundary layer thickness and might increase the sampling uncertainty.

The present study aimed to develop and test a low-cost device for sampling greenhouse gas flows at the air-water interface. Our goal is to evaluate these different methods and provide recommendations for future measurements based on our findings at a lentic aquatic ecosystem, the Rio Grande Reservoir, in the Metropolitan Area of São Paulo (MASP), Brazil. Within these results, we can assess the uncertainty associated with quantifications of gas emissions and implement improvements based on performance assessments and independent verifications.

2. Material and Methods

2.1. Construction of the floating sampling device

The device was a built-in high-density polyethylene pipe (15 cm internal diameter, i. d.) with a cap for which sealing with silicone rubber or epoxy resin was evaluated. The chamber had a circular transversal section with a 15 cm diameter (surface area of 0.017 m²). Two different heights were tested: 25 and 35 cm, resulting in chamber volumes of 0.018 m³ and 0.025 m³, respectively. A polytetrafluoroethylene (PTFE) valve was installed at the top of the chamber to allow the gas collection. Yet, to facilitate the sampling, a plastic tube (0.5 cm length; 1 mm i.d.) was connected to the valve. An ethylene vinyl acetate (EVA) square plate of 1 m² area was used to float the system (Figure 1).

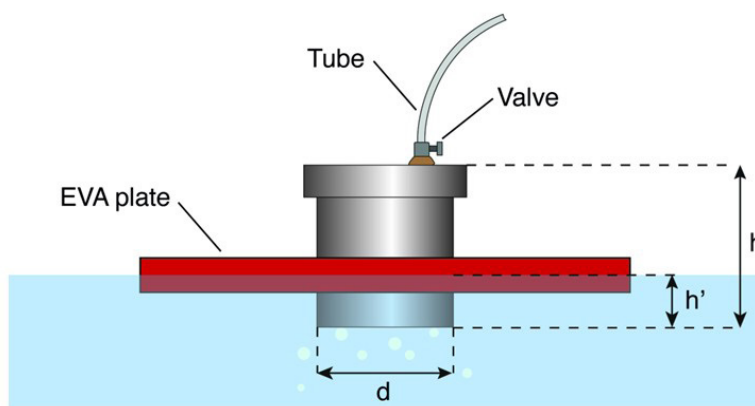


Figure 1. Floating static chamber. Ethylene vinyl acetate (EVA) square plate; h: total chamber length; h': submerged chamber height; d: internal chamber diameter.

2.2. Evaluation of chambers performance in laboratory

The experiments were carried out under monitored environmental conditions in the Environmental Analysis Laboratory at the Federal University of ABC, Santo André, Brazil, and the Automation and Analytical Instrumentation Laboratory at São Paulo University, São Paulo, Brazil. The background concentration inside the laboratory was (420 ± 60) ppmv for CO₂ and (1.6 ± 0.8) ppmv for CH₄ during the assays.

The sampling device was evaluated with and without the introduction of an auxiliary airflow, that is: flushed chamber (FC) or static chamber (SC), respectively. The procedure defined for FC mode used a synthetic air flow rate of 200 L h⁻¹ during the sampling period by connecting an aquarium pump with a rotameter to the plastic tube (Lucernoni et al., 2017). Sampling device facilities (with silicone rubber and epoxy resin) were also evaluated for leakage with the lowest and highest gas concentrations (100 and 1000 ppmv to CO₂ and 10 and 100 ppmv to CH₄, respectively).

To evaluate the chamber performance, CO₂ and CH₄ gas standards with $\geq 99,995\%$ purity (Linde, Brazil) were injected into the chambers through the valve. The gas amounts were intended to represent a concentration headspace of approximately 100, 200, 500, and 1000 ppmv above the background levels for CO₂ and 10, 20, 50, and 100 ppmv above for CH₄. Each gas concentration was measured 15 times per chamber type during the experiment (n = 15). Within a multi-factorial approach, modifications of both non-steady-state closed chambers were tested (Table 1). The relationship between the surface area and the volume (A/V) of each chamber was calculated to evaluate the mass and energy transfer within the sampling device and between the chamber and its environment.

Within these facilities characteristics, the following comparisons were possible: i) Changes Sealing Material (A with B); ii) Influence of the air vent (B with C, and D with E); and iii) Influence of A/V (B with D, and C with E).

2.3. Gas sampling

Gas collections from inside the chamber were performed for 1 hour at 15-minute time intervals, in times 0, 15, 30, 45, and 60 minutes. The collected gas volumes were transferred to gasometrical vessels (V = 50 mL) provided with a piston with a valve (Construmaq, Brazil). A stainless-steel system consisting of a drawing piston, probe, and transfer tube allowed the gas to transfer to the vessels. To facilitate the gas collection, a plastic tube connecting the inside of the collector with the external environment ensured isobaric conditions during sampling, mitigating errors (Rachor et al., 2013; Lucernoni et al., 2016).

2.4. CO₂ and CH₄ flowrate calculations

For the system operating in FC mode, the mass balance for GHG emissions was estimated by Equation 1.

$$Q_{GHG} = \frac{Q_{in} \cdot C_{GHG}}{C_{GHG,air} \cdot S} \tag{1}$$

where: Q_{GHG} is the measured flow rate of CH₄ ou CO₂ (L m⁻² h⁻¹); Q_{in} is the entrance of air flux (L h⁻¹); C_{GHG} is the concentration of CH₄ ou CO₂ determined by GC-FID (mol L⁻¹); C_{GHG,air} is the concentration of CH₄ ou CO₂ in atmospheric air (mol L⁻¹); and S is the cross-section of the sampling device (m²).

In SC mode, the boundary layer model was adopted, which is used in oceanographic studies (Rajkumar et al., 2008). It predicts the occurrence of two thin layers involving air-water interface and the gas exchange is connected to the transfer resistance between two layers. Considering the mass balance for the GHG in the chamber, it follows Equation 2.

$$V \cdot \frac{dC_{GHG}}{dt} = Q_{GHG} \cdot C_{GHG,air} \tag{2}$$

where: V is the volume of the static chamber, (dC_{GHG})/dt is the average of GHG concentration variation (CO₂ or CH₄) inside the chamber over time (mol L⁻¹), Q is the emitted GHG flow (L h⁻¹), and C_{GHG,air} is the concentration of the gases in external atmosphere (mol L⁻¹).

Table 1. Characteristics of floating chamber facilities and modifications for CO₂ and CH₄ injection pulses (n = 15).

Parameters	Facilities				
	A	B	C	D	E
Volume (m ³)	0.018	0.018	0.018	0.025	0.025
Area/Volume (A/V; m ⁻¹)	21.3	21.3	21.3	18.9	18.9
Air vent	No (SC)	No (SC)	Yes (FC)	No (SC)	Yes (FC)
Sealing material	Silicone rubber	Epoxy resin	Epoxy resin	Epoxy resin	Epoxy resin

The specific GHG flow rates were obtained assuming that GHG gases concentrations inside the chamber have a linear growth over time ($R^2 = 0.98$) and gases concentrations determined by GC-FID are equal to the average gases concentrations inside the device. For all situations, gas concentration was corrected by the temperature and pressure measured at the sampling time.

2.5. Analytical determination of CO_2 and CH_4

Gas chromatography with a flame ionization detector (GC-FID, Varian CP 8400, USA) was used to determine CO_2 and CH_4 concentrations. The equipment was calibrated with a standard containing CH_4 4.5 in synthetic air (purity $\geq 99,995\%$ in volume, 0.671 kg m^{-3} from Linde Gases, Brazil). Nitrogen (N_2) was used as a carrier and purge gas from the stationary phase (Restek Rt-Q-PLOT column, 0.53 mm i. d. ; 15 m length). The FID detector was driven with hydrogen and high-purity synthetic air. The signal related to CH_4 was recorded and correlated with the concentration of species in the sample through the calibration curve. For the determination of CO_2 , a new sample aliquot was taken from the vessel and injected into the GC-FID, allowing it to pass through a reducing column (nickel catalyst), that allows the conversion of CO_2 into CH_4 . Thus, the joint determination of the two species was made in the form of CH_4 . Then, the CO_2 concentration was obtained by the difference in the signal before and after the gas flow passing through the reducing column.

The operational conditions used to quantify the GHG were adapted from those established by Yuesi & Yinghong (2003). Table 2 presents the analytical parameters obtained in the determination of CH_4 and CO_2 by GC-FID. Other parameters used to determine the limits of detection and quantification of the flows emitted for each compound were: pressure 1 atm , temperature $(25 \pm 1) ^\circ\text{C}$, exposure time of 10 min , and diffusion chamber volumes of 0.018 m^3 and 0.025 m^3 .

2.6. Quality control check and performance

Standard concentrations of CH_4 (10 ppmv and 100 ppmv), CO_2 (100 ppmv and 1000 ppmv), and ambient air samples were analyzed as reference samples to assess the quality assurance (QA) and quality control (QC) protocols QA/QC. Before analysis, GC-FID was evaluated for calibration errors, sampling system bias, and interference. System zero check (without injection) and system blank check (without sample, with ultra-high purity N_2) were daily performed. Moreover, calibration curve verification and update were performed on each

analysis day, as well as duplication for repeatability for every 20 samples. A control chart for accuracy and precision was registered for sample replicates.

2.7. Field testing of the sampling device

To evaluate the field accuracy of the sampling device, five sampling campaigns were conducted in three stations in Rio Grande Reservoir in triplicate ($n = 45$) between 2014 and 2015: Station 1 (S1) S $23^\circ 43' 58.3''$, W $46^\circ 27' 25.3''$; Station 2 (S2) S $23^\circ 43' 13.5''$ W $46^\circ 26' 10.0''$ and Station 3 (S3) S $23^\circ 44' 00.8''$ W $46^\circ 25' 38.2''$ (Figure 2). Rio Grande Reservoir is an important water source to the Metropolitan Area of São Paulo (MASP), in São Paulo state, Brazil. It has approximately 7.4 km^2 area and is 9 km long (Nishimura et al., 2008). This water body provides many environmental facilities to both nature and the population, serving as a place for recreation, fishing, and public water supply. However, this ecosystem is impacted by inefficiently treated wastewater discharge (Wengrat & Bicudo 2011; Coelho et al., 2020), which high loads of nutrients and organic matter may contribute to greenhouse gas emissions (Beaulieu et al., 2019; Lopes et al., 2022). The sampling periodicity included sampling campaigns in different seasonal periods to evaluate the influence of climatic, limnological, and hydrological variables on diffusive fluxes of CH_4 and CO_2 at the water-atmosphere interface. Figure 1 illustrates the distribution of sampling stations in the Rio Grande Arm of the Billings Reservoir. Collections were carried out, as shown in Table 2.

During field campaigns, three SC ($A/V = 21.3 \text{ m}^{-1}$) operating in parallel were used simultaneously in each sampling station. Diffusive gas samples from air-water interface were collected at interval times of $0, 5, 10, 15, 20$, and 30 minutes . Samples were transferred to the gasometric vessels ($V = 50 \text{ mL}$) and stored at room temperature protected from light until GC-FID analysis. Results were tabulated and evaluated in terms of mean, standard deviation, minimum, and maximum values of carbon dioxide (CO_2) and methane (CH_4) flow rates at each sampling station.

Table 2. Analytical parameters for determination of methane (CH_4) and carbon dioxide (CO_2) by Gas chromatography with a flame ionization detector (GC-FID).

Compound	CH_4	CO_2
Retention time	2.1 min	2.3 min
Concentration range	$0.95 - 60.0 \text{ mg L}^{-1}$	$195 - 4000 \text{ mg L}^{-1}$
Precision (n = 5)	$\pm 32 \text{ mg L}^{-1}$	$\pm 1.3 \text{ mg L}^{-1}$
Detection limit	$27 \text{ } \mu\text{g m}^{-3}$	$180 \text{ } \mu\text{g m}^{-3}$
Quantification limit	$90 \text{ } \mu\text{g m}^{-3}$	$590 \text{ } \mu\text{g m}^{-3}$

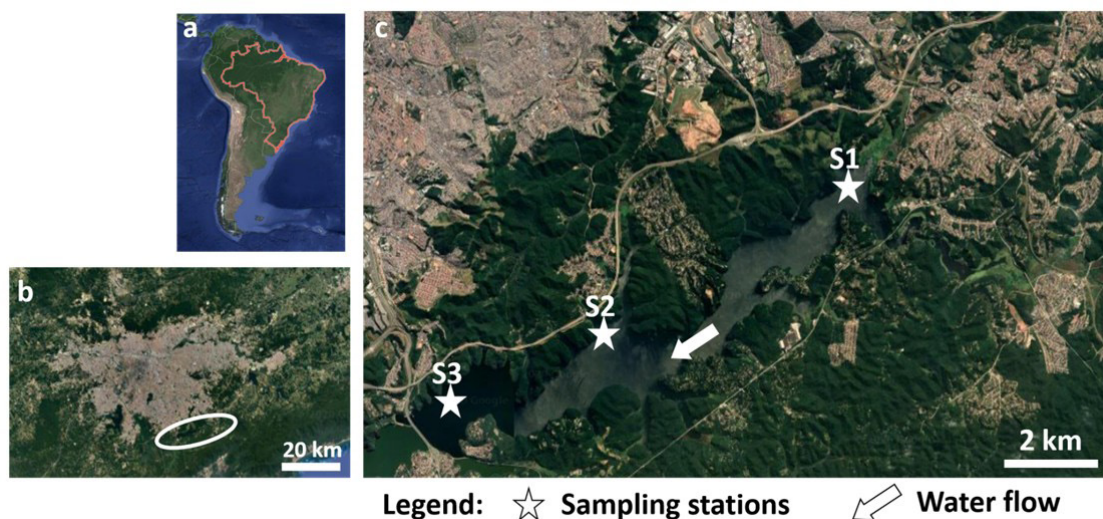


Figure 2. (a) South America with highlight to Brazil; (b) Metropolitan Area of São Paulo and the location of the study site; (c) Sampling stations at Rio Grande Reservoir.

Source: Adapted from Google Maps (2024).

3. Results and Discussion

3.1. Chamber design performance

The construction of chambers for sampling gases in water can present several technical and operational challenges. The sealing and isolation is one of the main concerns in the sampling accuracy since it must ensure that the chambers are hermetically sealed to prevent the entry of external air, which could contaminate the samples. Additionally, chambers must be designed to float stably regardless of water conditions such as waves and currents, and the materials used must be resistant to corrosion and degradation caused by prolonged exposure to water and sunlight (Bastviken et al., 2004).

Considering the characteristics of floating chamber facilities and modifications for CO_2 and CH_4 injection pulses described in the Table 1, silicone rubber eased the assembly of these sampling devices and provided greater malleability (A), which can be quite advantageous in field studies, where the chamber can move due to the water column's natural movement by winds or currents. However, the high flexibility of the silicone rubber provided free volume voids, which allowed gas diffusion resulting in high permeability and, consequently, sample loss. This structural characteristic particularly affects the overall diffusion when silicone is exposed to a solution at the surface and the dissolved gas molecules diffuse into the interior once the permeation depends on both solubility and the diffusion rates.

The decay of CO_2 concentration in the system with silicone sealing (A) was 0.06 min^{-1} , whereas for epoxy resin (B) it was 0.01 min^{-1} . This difference indicates a considerable leakage of CO_2 from inside the silicone sealed chamber (Figure 3a). The permeability of silicone rubber to gases is a direct result of the flexible chains of the material, which create an environment where gas molecules can enter, diffuse through the voids, and exit the material (Andrady, 1994). For CH_4 , in both sealing conditions, the decay was about 0.01 min^{-1} , indicating that for this gas the sealing material does not influence on diffusion hood (Figure 3b). The diffusion coefficient in silicone rubber for CO_2 is $1.1 \times 10^7 \text{ cm}^2 \text{ s}^{-1}$ and for CH_4 $1.3 \times 10^7 \text{ cm}^2 \text{ s}^{-1}$ (Maxwell & Robert 2008). Despite the equivalent values, the greatest loss of CO_2 comes from its solubility in the moisture droplets that may be deposited on the material's surface, as well as the greater internal pressure in the working concentrations that could interfere with the boundary layer enhancing diffusion and leakage.

Atmospheric CO_2 levels, water temperature, and biological activity influence the concentration of dissolved CO_2 . The pH of the water governs the distribution of inorganic carbon species. In typical freshwater systems (pH around 7-8), bicarbonate (HCO_3^-) is the dominant form. Methane dynamics is driven by organic matter availability, water temperature, and sediment characteristics. Gases can exchange between the atmosphere and the water surface, depending on the concentration gradient.

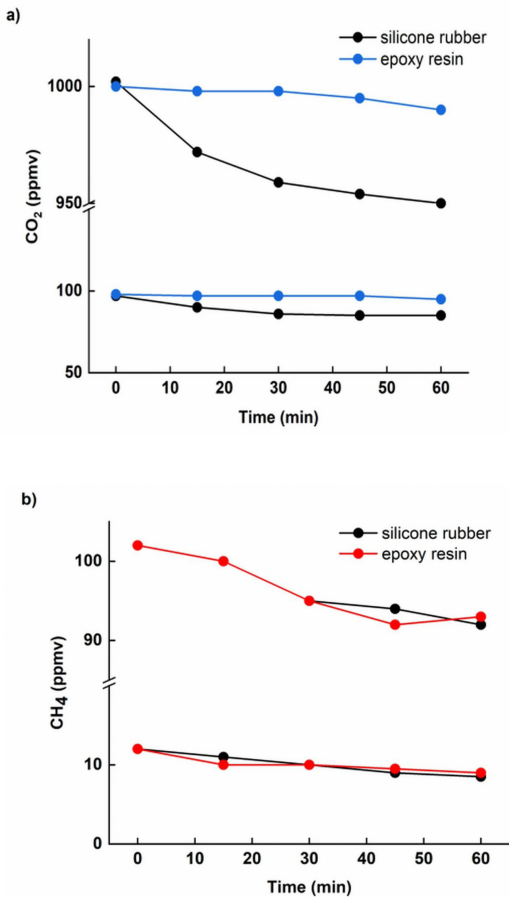


Figure 3. Gases leakage over time for chamber facilities sealed with silicone rubber (Facility A) or epoxy resin (Facility B), both 0.018 m³ of volume: (a) CO₂ at 100 ppmv and 1000 ppmv; (b) CH₄ at 10 ppmv and 100 ppmv (n = 15).

This exchange plays a significant role in the global carbon cycle (Bastviken, 2009). Considering fieldwork applications for air-water interface measurements, it is assumed that immediately above and below the boundary layer, air and water are homogenized and the process of gas exchange is in equilibrium. Thus, only the aqueous boundary layer controls the diffusion process (Jähne & Haußecker, 1998). The diffusion coefficient of CO₂ and CH₄ in the air is about 10⁴ higher than in water (Biondo et al., 2018). CO₂ is very soluble in water (solubility 7.0 × 10⁻⁴ mol L⁻¹ at 20 °C) and diffusion is its major occurrence towards the water-atmosphere. However, CH₄ is relatively insoluble in water (solubility coefficient = 2.8 × 10⁻⁵ mol L⁻¹ at 20 °C) and is often emitted by ebullition from sediments (Bastviken et al., 2004). Thus, for water-soluble gases, such as CO₂, this enhanced chamber out-gassing might introduce several uncertainties in flow rates.

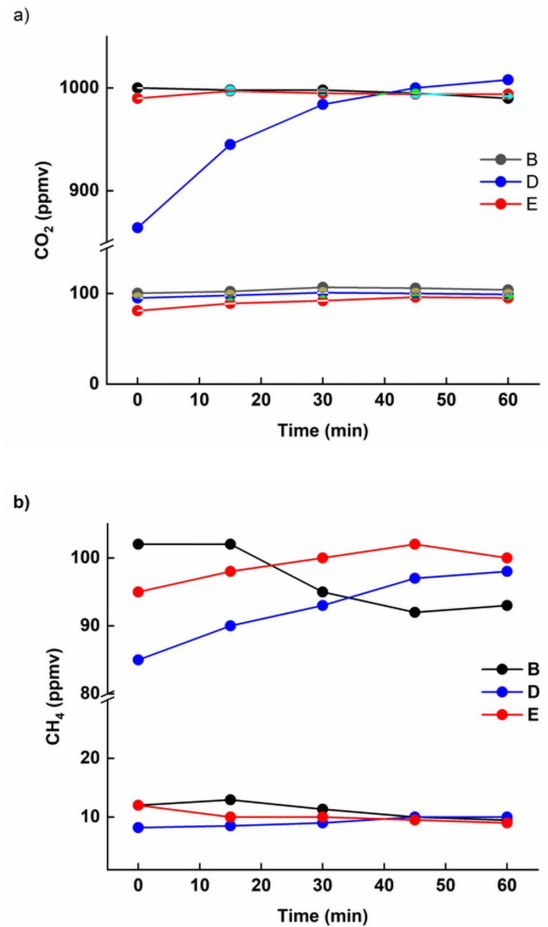


Figure 4. Gases concentrations from the headspace of chamber facilities B and D (both non-vented, A/V= 21.3 m⁻¹ and 18.9 m⁻¹, respectively); and E (vented, A/V = 18.9 m⁻¹): (a) CO₂ at 100 ppmv and 1000 ppmv; (b) CH₄ at 10 ppmv and 100 ppmv (n = 15).

An important factor that changes the effectiveness of the headspace sampling in floating chambers is the chamber design, which affects the area-to-volume ratio (A/V) and the mass transfer inside the floating chambers. Figure 4 shows the gas concentrations over time for devices with an A/V 21.3 m⁻¹ and 18.9 m⁻¹ without a vent (Facilities B and D, respectively) compared to a system with a vent (Facility E). According to Hutchinson & Livingston (2001), ventilation can help homogenize the gas mixture inside the chamber, minimizing overpressure problems, but it also increases the risk of leaks and losses by diffusion. Comparatively, non-ventilated floating chambers, such as those used in your study, offer simplicity but can face similar overpressure challenges if not properly adjusted.

The chamber with greater A/V (B) showed a more homogeneous profile of both CO₂ and CH₄ concentrations in the headspace, potentially capturing gas emissions from water more quickly.

The minor A/V chamber configuration (D) resulted in a delay in reaching equilibrium (30 min) due to a less efficient mixture, which was achieved with the inclusion of ventilation (E). Thus, A/V optimization affects the headspace gas mixture rate and the gas detectability: minor ratios can cause a slow mixing, which results in less sensitivity to changes in concentration, especially for lower concentration measurements, requiring more accurate analytical sensitivity or longer gas hood interval times inside the chamber. However, greater A/V results in a faster concentration change within the headspace. Depending on this established concentration gradient, the greater diffusion of gas at the water/air interface can be boosted by overestimating the flow values (Hutchinson & Livingston, 2001). Davidson et al. (2002) showed that non-steady-state chambers will usually produce uncertainties ranging from negligible to 15% underestimation, depending on the water layer condition. However, Pumpanen et al. (2004) considered that the limiting step in the efficiency of these -steady-state flow chambers is the method of mixing air within the chamber's headspace, which either can underestimate or overestimate gas fluxes from -21 to +33% depending on the type of chamber.

Figure 5 shows the agreement between CO_2 and CH_4 input and measured values for the non-vented (B) and vented (C) modifications of the chamber with 0.018 m^3 of volume sealed with epoxy resin. For the lowest concentrations of CO_2 and CH_4 , both systems performed similarly, with differences between the input and the analyzed concentration below 10%. For the highest concentrations, the uncertainties related to this determination were higher than 20%, especially for CO_2 , whose measured concentration was 40% lower than the input concentration.

This greater uncertainty may be due to a greater mass flow that may cause an increase in internal pressure with ventilation, with greater diffusion losses due to the chamber material or leak through the seal (Lai et al., 2012; Brændholt et al., 2017). For this condition, greater A/V chambers can better dissipate this over-pressure during sampling (Hutchinson & Mosier, 1981), but decreasing the concentration of gas in the headspace, causing longer accumulation times for analytical detectability. For *in situ* studies, the concentrations used in this work are higher than those typically found in aquatic ecosystems, such as in the work done by Xing et al. (2005) in Chinese subtropical lakes and Almeida et al. (2016) in eutrophic reservoir in the semiarid Northeast in Brazil. So the evaluation of

larger chambers is unnecessary since the non-vented facilities worked well for the intended field sampling campaigns.

3.2. Field application of the floating static chamber

In field studies, the sampling period depends on the concentration change over time, which relies on the chamber characteristics and also on the study area characteristics such as substrate concentration for gas production, land use, floods, precipitation, seasonality, time of the day, and temperature. Thus, a specific protocol should be evaluated for each study area.

Wetland areas are transitional regions such as floodplains, reservoirs, swamps, shallow lakes (permanent or temporary), rivers, and other ecosystems. These regions have a high production of CO_2 during net respiration conditions (Mitsch et al., 2008; Nahlik & Mitsch, 2011). However, anoxic conditions in some wetlands zones result in anaerobic microbial processes with lower transference of energy and consequent CH_4 output (Nahlik & Mitsch, 2011). Thus, the release of CO_2 and CH_4 into the atmosphere may occur by diffusion process through water column or boiling.

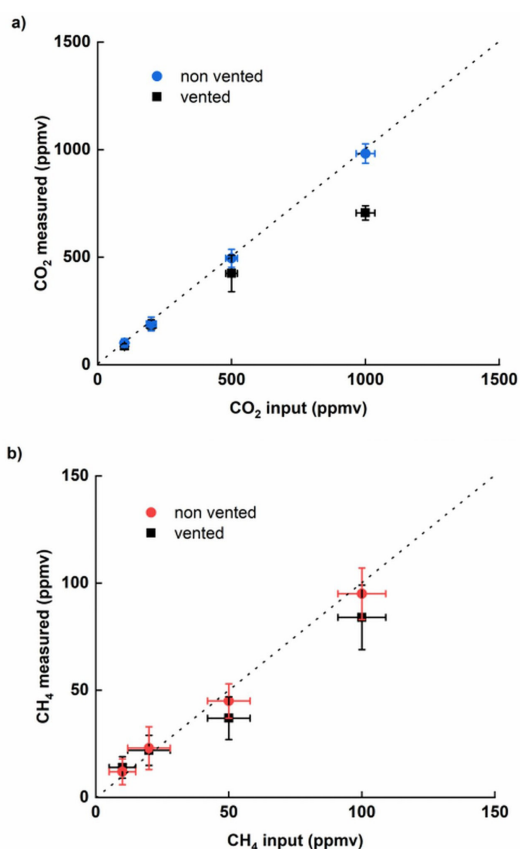


Figure 5. Gases concentrations from chamber facilities B and C (A/V = 18.9 m^{-1} sealed with epoxy resin) to non-vented and vented measurements ($R^2 > 0.99$): (a) CO_2 and (b) CH_4 .

Emission rates of these compounds is controlled and influenced by water temperature, salinity, redox potential, pH, and the availability of organic substrates that are responsible for activating the process and the concentration of nutrients available (Fey & Conrad, 2000). Climatological variables such as air temperature, relative humidity, wind speed, and direction may also affect gas diffusion at the water-air interface (Mitsch et al., 2008; van Bergen et al., 2019). Monitoring methane concentrations in the atmosphere provides insight into the overall abundance and distribution of methane. Identifying the sources of methane emissions is crucial, which can include natural sources such as wetlands, as well as anthropogenic sources like fossil fuel production and agriculture (including livestock digestion and fertilizer management), landfills, wastewater treatment, and inadequate disposal of organic matter into water bodies (Lopes et al., 2022). Concentrations can vary spatially and temporally due to these emission sources, atmospheric transport, and removal processes depending on factors such as human activity levels, management practices, and environmental conditions. Methane has a relatively short atmospheric lifetime compared to carbon dioxide, but its impact on climate depends on various factors, including its rate of removal from the atmosphere through chemical reactions and its interactions with other atmospheric constituents (Hu et al., 2020).

At the Rio Grande Reservoir, the facility B was used to collect samples from the air-water interface at interval times (Figure 6). The sampling interval time has several effects on the gas diffusion flow rates. Initial placement of the floating chamber in the water column might cause an over-pressure artefact in the headspace gas concentration. This disturbance may last about a minute (Davidson et al., 2002). This period is relevant in gas exchange studies in aquatic ecosystems due to the variety of emissions sources. For example, several sampling methods make a combined estimation of air-water gases measurements. This is particularly important in field studies in shallow regions, where the contribution of ebullition cannot be distinguished from diffusion. Thus, shorter sampling intervals can mitigate the uncertainty of the gas flow rate by CH_4 boiling contribution. According to Deemer et al. (2016), the mean sum of ebullition with diffusion fluxes was over double that of diffusion-only fluxes, which can overestimate CH_4 emissions in some regions.

Otherwise, very long sampling intervals can lead to headspace saturation causing leakage (Burrows et al., 2005), leading to a non-linear condition of gas concentration over time. As we assume a linear change in our flow calculations, the time taken for field collection with these chambers was estimated based on the apparent non-linearity observed, usually after a maximum of 15 minutes. No seasonal variation of this sampling time interval was observed in the studied area.

To determine the performance of our sampling device, gas samples from the air-water interface were collected in five field campaigns for the determination of CH_4 and CO_2 flow rates. Table 3 presents the variation of the target gas flow rates, with a minimum below the limit of quantification ($\leq 0.062 \text{ mg m}^{-1} \text{ min}^{-1}$) for CH_4 in some sampling campaigns and a maximum of $42.3 \text{ mg m}^{-2} \text{ min}^{-1}$ for CO_2 in a polluted area during a winter sampling campaign.

The values for GHG flow rates are quite similar to those obtained in hydroelectric reservoirs in Brazil (Marcelino et al., 2015; Vale et al., 2017) and higher than those obtained from reservoirs and natural

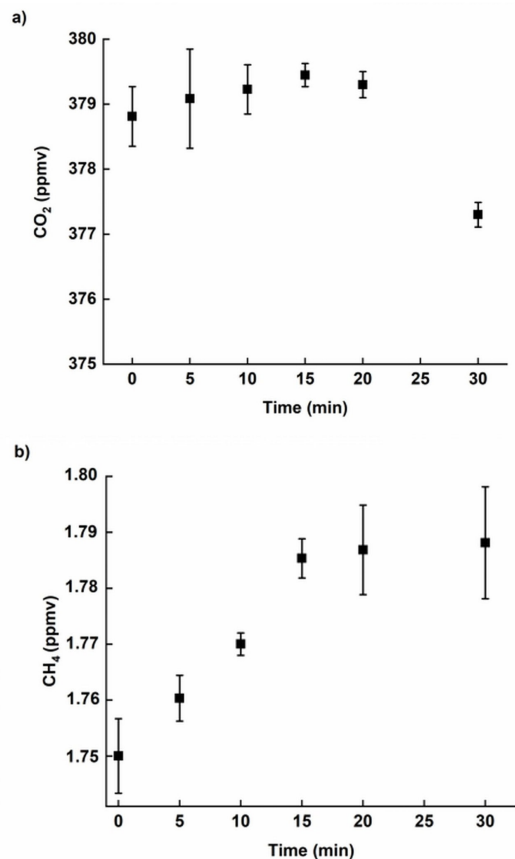


Figure 6. Greenhouse gases concentrations (ppmv) over time using the loathing chamber B at the Rio Grande Reservoir: (a) CO_2 and (b) CH_4 .

Table 3. Mean, standard deviation, minimum, and maximum values of carbon dioxide (CO₂) and methane (CH₄) flow rates at sampling stations (S1, S2, and S3) in the Rio Grande reservoir (Brazil) collected with the floating chamber B. The sampling interval time was 5, 10, and 15 min.

Sampling station	CO ₂ (mg m ⁻² min ⁻¹)	CH ₄ (mg m ⁻² min ⁻¹)
S1	18.89 ± 13.42 (9.14 - 42.27)	0.133 ± 0.060 (0.080 - 0.214)
S2	2.80 ± 0.35 (0.30 - 3.15)	≤ 0.062
S3	13.33 ± 4.58 (8.79 - 20.80)	0.090 ± 0.030 (0.062 - 0.090)

areas of tropical climate in Costa Rica (Nahlik & Mitsch, 2011). Although this was not the case in the present study, seasonality is used to influence the GHG emission in aquatic ecosystems. Several authors found a wide range of GHG values between dry and wet periods in tropical aquatic ecosystems (Rosa et al., 2004; Almeida et al., 2016). This high variability occurs due to several environmental factors already reported in the literature, such as sewage disposal with the entry of organic matter (Yang et al., 2020); water column depth (Borrel et al., 2011); and aquatic plant biomass (Abril et al., 2014).

The use of floating chambers in gas sampling in reservoirs has several significant implications, especially in terms of environmental monitoring and water resources management (Teodoru et al., 2012). Floating chambers allow direct and precise measurement of GHG emissions, especially when equipped with automatic sensors, which can provide real-time and continuous data, allowing a better understanding of diurnal and seasonal variations in gas emissions. As they are autonomous and floating devices, there is less direct human interference, which can lead to more accurate and representative data (Kumar et al., 2019). The ease of mobility of these devices can provide broader and more detailed spatial coverage, which is essential to identify gas emission hotspots. However, wind, currents, and temperature can influence data collection due to mass transfer change. To overcome these drawbacks, adequate sample planning, the optimization of the sampling device, and accurate calibration procedures are required to minimize uncertainties. In regions with extreme weather conditions, such as intense rains or prolonged droughts, data collection may be affected requiring the development of strategies to mitigate these impacts (Bastviken et al., 2008). Pumpanen et al. (2004) compared automatic,

semi-automatic, and manual chambers in measuring soil CO₂ flux, addressing differences in accuracy, ease of use, and cost. Automatic chambers proved to be more accurate but significantly more expensive and complex, while manual chambers, including floating ones, offer greater portability and simplicity, though with potential measurement errors due to the lack of rigorous control over environmental variables.

In summary, floating chambers provides a valuable tool to improve estimates of greenhouse gas emissions from reservoirs, contributing to national and international inventories (Dugan et al., 2024). This is especially sensitive in less developed areas since these devices can provide a relatively affordable way to monitor gas emissions compared to more advanced and high-cost technologies. Furthermore, this information can help fill data gaps in regions in which environmental monitoring is scarce.

4. Conclusions

Our floating static chamber device proved robust for collecting gas flows at the air-water interface. By not requiring electricity, it expands the versatility of using the device for sampling in more remote locations. Thus, our device can help in the study of gas flows, such as GHG, at the air-water interface in inaccessible, poorer, and more vulnerable places since it is cheap and very simple to build and operate. However, it is still worth emphasizing the necessity for a central laboratory structure to carry on the analyses or expensive portable instruments for field measurements.

Diffusive gas measurements in air-water interfaces using floating chambers are affordable, operationally simple, and adaptable for deployment across various locations. By isolating specific areas or sources, flow chambers can help identify and measure GHG emissions from particular sources within a larger environment, aiding in source-specific studies. However, its use must be validated in preliminary fieldwork to define sampling uncertainties and main challenges to be overcome, such as lack of spatial accessibility, absence of electricity, or possible disturbances in the interface with the water column that could cause greater gas emissions. In addition, the high spatial and temporal variability of these emissions in aquatic ecosystems indicates that these collection strategies must be validated for each sampling situation.

Acknowledgements

We thank Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) for the financial assistance (Process n. 475080/2013-0), Instituto de

Química da Universidade de São Paulo (IQ-USP) for the analytical support, Central Experimental Multitusuário da Universidade Federal do ABC (CEM/UFABC) for the use of their facilities, and Guarda Ambiental de São Bernardo do Campo for the field support and the boat used in sampling campaigns. We also thank Vanessa Sontag (Illus Scientia) for Figure 1.

Data availability

The entire dataset supporting the results of this study has been published in the article itself.

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Received: 27 April 2023

Accepted: 09 September 2025

Associate Editor: Irineu Bianchini Júnior.