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Technical note: Resolving Vertical CO_2 Concentration Gradients at the Air-Water Interface Using a Novel Membrane Equilibration Technique

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Abstract. Quantifying diffusive greenhouse gas emissions from inland waters often relies on the concentration gradient between dissolved gases in the water and their equilibrium concentration in the atmosphere. However, while gas exchange occurs directly at the surface, dissolved CO_2 is mostly measured further below the surface, potentially introducing bias. Recent studies have shown the presence of vertical CO_2 gradients during calm conditions, which can lead to systematic errors in flux estimations when measurements are not taken at the correct depth. To address this issue, we developed a novel Floating Membrane Equilibrator (FME)—a thin (0.6 cm), flexible device equipped with gas-permeable silicone tubing arranged in a flat plane with a theoretical vertical resolution of 1 cm. By directing airflow through the tubes, for instance using an infrared gas analyzer, CO_2 in the water equilibrates with the gas phase inside the tubing. The resulting CO_2 concentration reflects the local dissolved CO_2 in the adjacent water layer. We tested the FME in a controlled pool experiment using CO_2-supersaturated water. Two FMEs were deployed at 1 cm and 25 cm depth. Results show that the FME provides reliable equilibration with the surrounding water and delivers accurate CO_2 measurements. Compared to conventional CO_2 probes, the FME shows faster response times and higher temporal resolution, enabling detection of short-term fluctuations that are typically missed by standard sensors. Moreover, our measurements revealed a distinct vertical CO_2 gradient in the pool, with higher concentrations at 25 cm depth compared to 1 cm, which is consistent with surface outgassing. This highlights the risk of misestimating fluxes when relying on deeper CO_2 measurements. The FME is a valuable tool for resolving near-surface CO_2 profiles with fine vertical resolution, thereby improving our understanding of the dynamics and drivers of lake-atmosphere gas exchange. Ultimately, the FME helps reduce uncertainty in CO_2 flux estimates and supports the development of more accurate models for greenhouse gas emissions from aquatic systems.

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1 Introduction

Lakes and reservoirs play a relevant role in the global carbon cycle by acting as sources of carbon dioxide (CO_2) and methane (CH_4). Despite their relatively small area compared to the oceans, lakes emit disproportionally large amounts of greenhouse gases (GHGs) to the atmosphere (Raymond et al., 2013). Diffusive emissions of GHGs from the water to the atmosphere can be calculated using the Thin-Boundary-Layer approach (TBL; (Cole and Caraco, 1998)). This method calculates the gas flux (F) from the product of the gas transfer coefficient (k) and the difference between the gas's concentration in the water ($c_{CO_2\ water}$) and at equilibration with the atmosphere ($c_{CO_2\ equilibrium}$):

$$F_{CO_2} = k * (c_{CO_2\ water} - c_{CO_2\ equilibrium})$$

Compared to other flux measurement methods, the TBL approach is simple and cost-efficient. It requires only gas concentration measurements in the water, while atmospheric gas concentrations and gas transfer coefficients can be obtained from databases and modelled using wind speed data, respectively. However, fluxes calculated from the TBL approach are error prone in conditions of low windspeeds. This is mainly due to poor performance of k models at low wind speeds (Crusius and Wanninkhof, 2003; Yang et al., 2022). Therefore, over the past three decades, research efforts have focused on improving the parameterization of k, which remains one of the largest sources of uncertainty in estimating gas fluxes across the air–water interface (e.g., Wanninkhof, 1992; Crusius and Wanninkhof, 2003; MacIntyre et al., 2010; Vachon and Prairie, 2013). These studies have aimed to optimize the parametrization of k based on parameters like wind speed, water-side turbulence, or temperature. However, parametrization of k is not the only source of uncertainty of the TBL approach. Recent studies have shown that unresolved vertical gradients near the water surface can systematically bias CO_2 flux calculations in marine and freshwater systems (Ford et al., 2024; Aurich et al., 2025). Thus, while improving k parametrizations remains one way to improve flux calculations, it is also important to obtain more accurate data of the actual CO_2 gradient by having more reliable measurements at the water-air interface, which is especially relevant under calm conditions.

Most studies do not explicitly address the assumption of homogeneous CO_2 concentrations near the water surface. Measurements or samples are typically taken within the upper meter of the water column, often without discussing vertical CO_2 gradients or explaining the choice of sampling depth. However, in a recent study we revealed significant differences in CO_2 concentration between 5 cm and 25 cm depths (Aurich et al., 2025). We observed higher CO_2 concentrations at 5 cm depth compared to 25 cm depth, which challenges the representativeness of probe measurements typically conducted at depths between 25 cm and 1 m. Modelled CO_2 fluxes based on concentration measurements at 5 cm aligned more closely than those derived from 25 cm depth with CO_2 fluxes measured in the atmosphere above the water by eddy covariance measurements. This was explained by an accumulation of plankton at the water surface, which led to high CO_2 production at night, resulting in a steep concentration gradient within the uppermost 25 cm of the water column.

Discrepancies between concentration-gradient-based flux estimates and direct measurements such as eddy covariance have also been reported elsewhere, particularly under calm night-time conditions. For instance, Mammarella et al. (2015), found that CO_2 fluxes were often underestimated by standard models relying on wind-based gas transfer coefficients and bulk CO_2

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measurements. While they attribute the mismatch primarily to buoyancy-driven turbulence enhancing gas exchange, such discrepancies may also reflect vertical CO_2 gradients that are not resolved when concentrations are measured too deep in the water column. These findings suggest that the commonly used method to measure CO_2 at a presumed safe depth, can introduce uncertainties in emission estimates.

Most of the currently available methods to measure aquatic CO_2 concentrations are limited in their ability to capture fine vertical CO_2 concentration gradients close to the surface. For example, CO_2 probes have relatively large diffusive membranes, reducing the vertical resolution to the probe's diameter. Manual water samples for headspace analysis are more precise in depth targeting, but may disturb the water column, especially when conducted from a boat. Moreover, manual samples cannot resolve temporal fluctuations needed to track short term variability in at the water-air interface. Other studies have used floating chambers that equilibrate with the surface water concentration, enabling precise measurements directly at the surface but at the expense of temporal resolution (Pajala et al., 2023; Rudberg et al., 2024).

To address these challenges, there is a need for a method that can reliably measure CO_2 concentrations in the upper centimeters of the water column. A previous study (Hari et al., 2008) demonstrated that silicone tubes can be used to measure CO_2 in the water. The authors connected a simple CO_2 analyzer to silicone tubes, which were mounted to a submergible structure, allowing for concentrations measurements at 0.1 m and 0.5 m depth. The method was also used in other studies (Heiskanen et al., 2014; Mammarella et al., 2015; Erkkilä et al., 2018) to measure CO_2 concentrations in multiple depths, but previous implementations were not designed for measurements in the upper centimeters of the water column, where fine-scale gradients regulate diffusive fluxes.

Here, we describe a novel design that allows in situ high frequency measurements of dissolved CO_2 with a vertical resolution below 0.03 m. We describe the assembly of the structure, performance tests, and examples of in situ applications. Our aim is to provide a complementary method that improves the vertical resolution for near surface CO_2 measurements, thereby supporting a better quantification of water-air gas exchange under wind still conditions.

2 Materials and methods

To measure dissolved CO_2 concentrations at high vertical resolution, we designed a thin, flexible, and floating structure, which we call Floating Membrane Equilibrator (FME). The primary goal of the design was to create a widely accessible and affordable CO_2 measurement solution. We cut twin-sheet plates (polycarbonate) into 1 m x 1 m panels and created a window-like structure by cutting four rectangular openings. The outer frame of the structure was 4-4.5 cm in width, and the cross frames within the windows were also 4 cm in width (Figure 1, created using Autodesk Inventor 2017 (Autodesk Inc., 2017)). Polytetrafluoroethylene tubes (PTFE, 4 mm and 6 mm inner and outer diameter, respectively) were cut into 25 cm long pieces, which were used for the bend parts of the serpentine pattern to prevent kinks (Figure 1). The PTFE tubes would not fit naturally in the frame due to the outer diameter of 6 mm interfering with the 6 mm thickness of the twin-sheet plate. However, applying a small amount of water or soap the outer surface of the PTFE tubes can facilitate its insertion to the plate. Silicone tubes (4

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mm inner and 5 mm outer diameter) were attached to the PTFE tubes to span through the openings. The silicone tube was elastic enough to pull over the PFTE tubes to connect them. In total, the of silicone made up 12.4 m with a contact area of 0.234 m².

The idea of using semipermeable silicone tubes with an in-line flow through analyzer has previously been shown to provide accurate and reliable CO_2 measurements in aquatic systems (Hari et al., 2008; Heiskanen et al., 2014; Erkkilä et al., 2018; Provenzale et al., 2018). In preliminary, unpublished, results, we found that using two CO_2 analyzers for two FMEs introduced systematic differences due to calibration offsets and different measurement ranges of the analyzers. These differences made it difficult to distinguish real CO_2 gradients from instrument-driven variability. To avoid this issue, the lab tests were performed using a single analyzer connected to both FMEs.

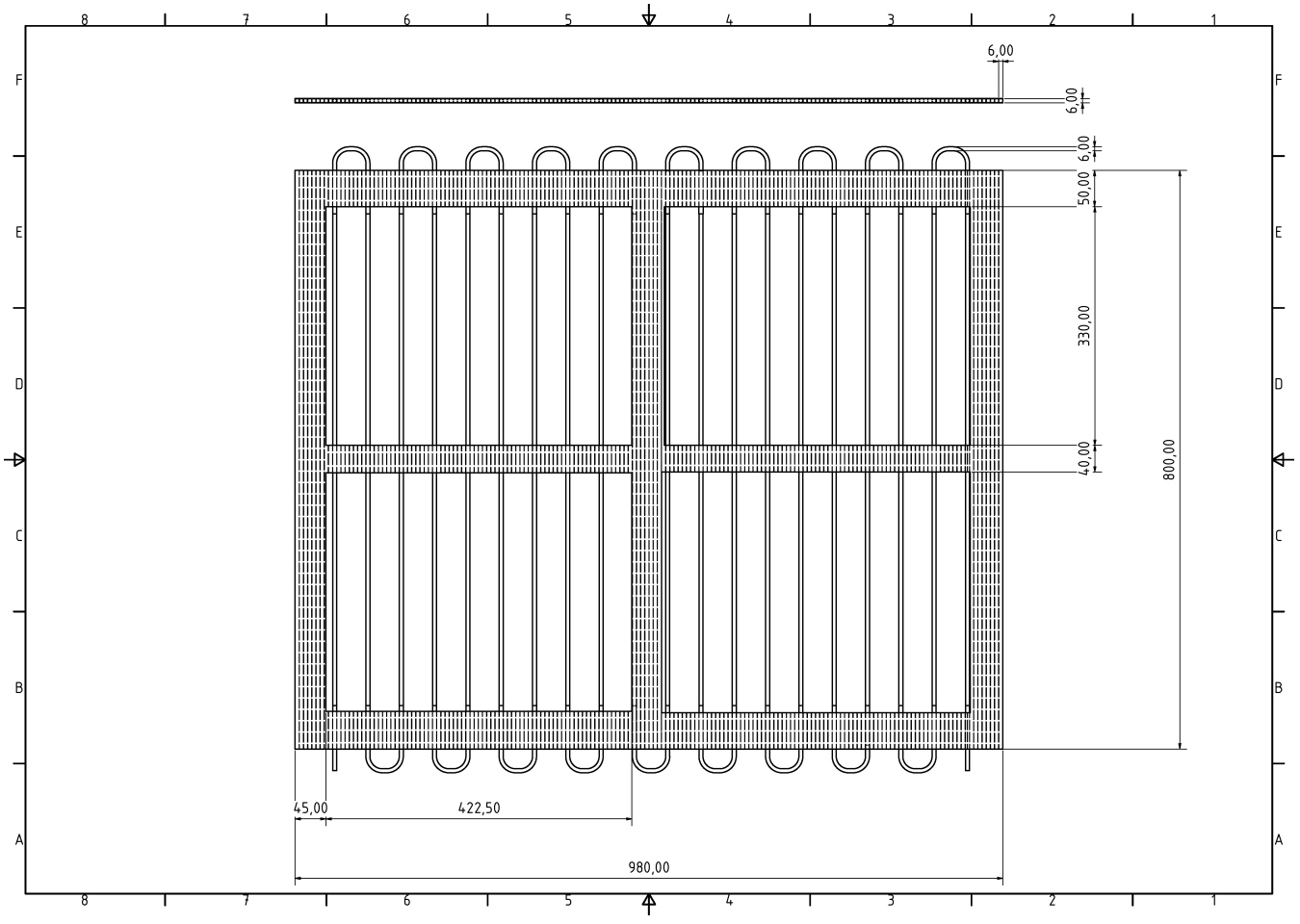

Figure 1: Technical drawing of the Floating Membrane Equilibrator (FME). Measurements are given in mm. The number of silicone tubes fit to the frame may vary according to practicality. In this case, 20 tube pieces of 700 mm each are fit to the frame. 19 pieces of PTFE tube are used for stiff bends between the silicone tubes.

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The FME was connected to a nondispersive infrared analyzer (NDIR, EGM-5 Portable CO_2 Gas Analyzer, PP Systems, Amesbury, Massachusetts, USA). The NDIR analyzer was set to 300 m L min^{-1} flow rate, and the instrument offset corrected before deployment using the internal zero calibration.

In order to confirm the FME's ability to measure vertical CO_2 gradients with a high resolution, a performance test was conducted. For this, an inflatable pool (2.62 x 1.75 x 0.51 m) was set up indoors and filled with tap water to an initial water depth of just over 30 cm (ca. 850 L, Figure 2). The indoor setup was chosen to exclude any environmental factors that could affect the dissolved CO_2 concentration in the water, such as weather (esp. wind) or microbial activity (esp. phytoplankton photosynthesis). In this setup, the pool was filled with CO_2-supersaturated tap water (typically containing approximately 3000 ppm CO_2), which could only degas via diffusion across the water surface. Tap water was added after each experiment to compensate for evaporative water loss and to restore CO_2 concentrations reduced by degassing. Water temperature was monitored to avoid fluctuations during the experiments.

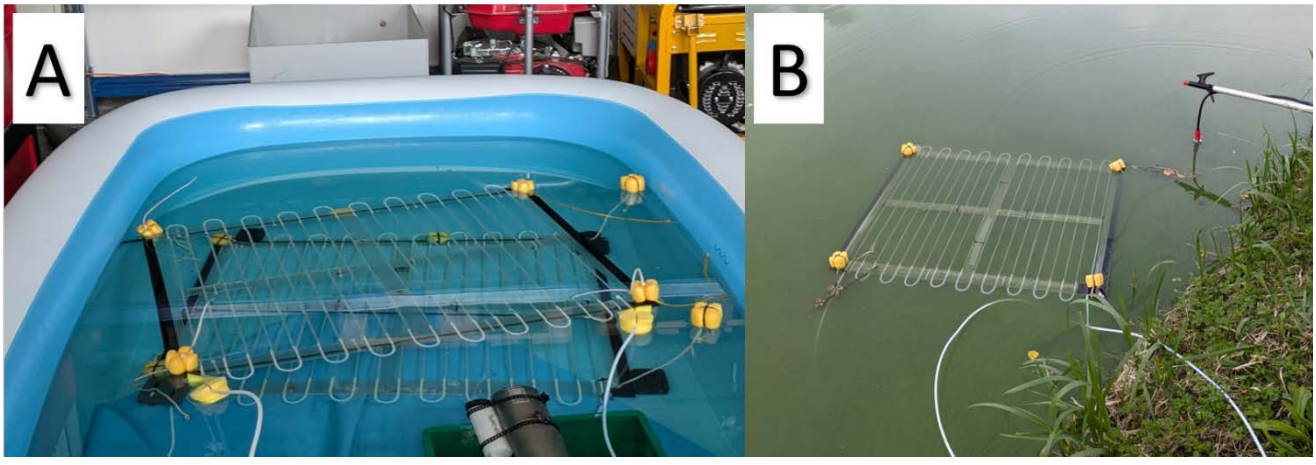

Figure 2: Experimental setups. A: the inflatable pool was filled with approximately 850 L tap water, which was oversaturated with CO_2. Two FMEs were placed at 1 cm below the surface and just above the ground, respectively. Both FMEs were connected to the same CO_2 gas analyzer using two three-way valves on the in- and outlet. B: The FME was placed at the water surface of a pond with a submerged CO_2 probe at 25 cm depth.

Two FMEs were placed in the pool and connected to the NDIR analyzer (1 second sampling interval) with three-way valves. One FME was placed on small weights 1 cm above the bottom of the pool (25 – 29 cm depth, depending on the water depth). The other FME was placed above it, either laying on top (24 – 28 cm depth, depending on the water depth) or floating just below the surface (ca. 1 cm depth, to ensure it was fully submerged). Halfway through the experiments the two FMEs were swapped. As the NDIR analyzer is only able to measure one FME at a time, the three-way valves were used to switch the measurement between the two FMEs at regular intervals. In the same-depth setup, the two FMEs were compared to ensure that they measure the same CO_2 values. In the two-depths setup, the CO_2 gradient between the surface and the pool bottom was observed. At the end, two small aquarium pumps (Eheim compact 300, Eheim GmbH & Co. KG, Deizisau, Germany;

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flow rate 300 L h-1) were added to the pool to break the CO_2 gradient and circulate the water throughout the entire water column.

The FME was also tested in the field during measurement campaign in late September 2024 in a fish pond at the experimental study site of the Department of Fisheries of Saxony in Königswartha, Germany. The fishpond we chose for the field test had a mean depth of 1 m and was 1 ha in area. The pond was stocked with carp and received no additional food. As the carp fed primarily on zooplankton, the pond experienced a cyanobacterial bloom as a result. The FME was placed floating just below the water surface and connected to the NDIR analyzer (1-minute sampling interval). A CO_2 probe (AMT Analysemesstechnick GmbH, Rostock, Germany) was installed at 25 cm depth and measured the dissolved CO_2 concentrations at a measurement interval of 1 second (Figure 2). Both the FME and AMT probe measured continuously for about 30 hours.

Data analysis and plotting were done in R (version 4.4.1, R Core Team, 2023). For each experiment, the first five minutes of measurements after switching the gas circulation to the other FME were discarded due to pressure-induced artefacts caused by flow interruptions during the switching process. Note that this was not necessary when measuring with one FME only. To quantify relative errors (%) between two FMEs installed at the same depth, subsequent measurements were paired across switching events to assess whether both devices yielded comparable results under identical conditions:

$$\text{Relative error (\%)} = \frac{|\overline{M_1} - \overline{M_2}|}{\overline{M_1}} * 100$$

3 Results

In the first experiment, the two FMEs were placed at the same depth, with one positioned directly above the other, to assess whether they measured consistent CO_2 concentrations. In close proximity, measurements between the two FMEs were nearly identical (Figure 3). The average relative error between the measurements was 0.18 % at concentrations around 950 ppm (Figure 3 A) and 0.15 % at concentrations near 2800 ppm (Figure 3 B).

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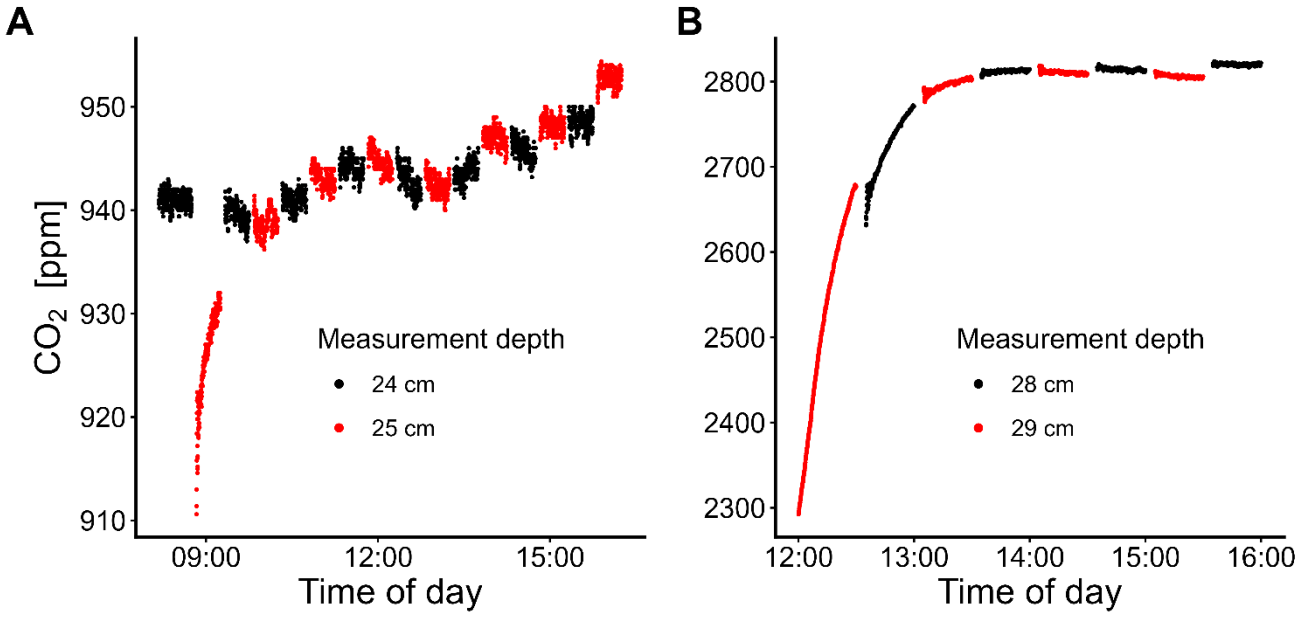

Figure 3: CO_2 measurements with two FMEs placed 1 cm vertically apart from each other in a pool filled with tap water. Displayed are the mixing ratios of CO_2 of the upper (black dots) and lower (red dots) FME. A valve in the gas stream of the CO_2 analyzer was switched between the FMEs every 30 minutes in intermediate (A, left) and high concentrations (B, right). Switching between the two FMEs did not change the pattern of the CO_2 mixing ratio in both cases.

In the second experiment, the FMEs were positioned at different depths: one remained at the bottom of the pool, while the other one floated just below the water surface. The idea was to observe stable concentrations at the bottom and decreasing concentrations near the surface due to diffusive outgassing to the atmosphere. In both tests, CO_2 concentrations at the bottom remained stable (ca. 3000 ppm and 2740 ppm, red points in Figure 4 A and B, respectively), while surface concentrations declined rapidly (2850 ppm to 2400 ppm in 9 hours, and 2750 to 2600 in 3 hours, see black points in Figure 4 A and B, respectively).

The response time (T90) of the FME was calculated for each deployment of the FME, where it ranged from 21 minutes to 54 minutes (average 37 minutes; Table S1 in the supplement).

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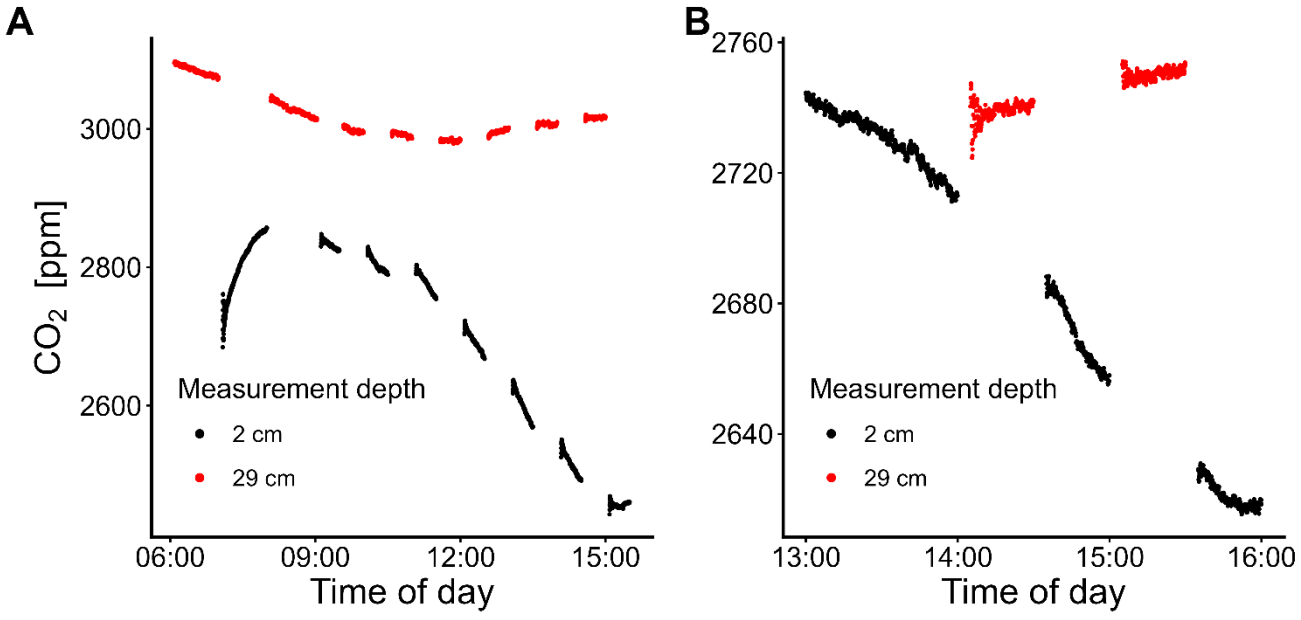

Figure 4: Demonstration of CO_2 gradients in the pool setup using Floating Membrane Equilibrators at both the surface (2 cm depth, blue dots) and at the bottom of the pool (29 cm depth, red dots).

After adding pumps to the pool to mix the water column (October 30th, 11:16), the CO_2 gradient was destroyed and the CO_2 concentrations at both depths immediately became similar and continued to become more similar the longer the mixing went on, while decreasing overall (Figure 5).

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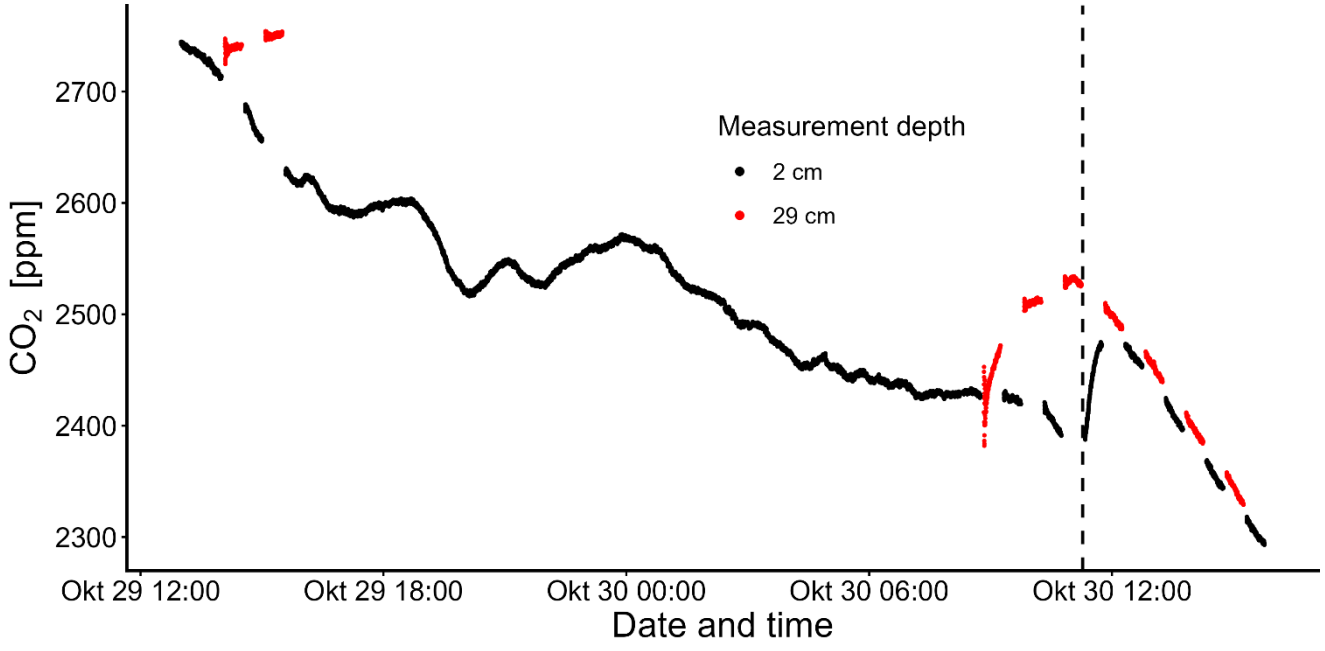

Figure 5: Impact of a mixing event after CO_2 gradient established. FMEs were kept at two different depths (2 cm, black dots; 29 cm, red dots). From October 29th 15:00 to October 29th 16:00, both depths were recorded with the CO_2 analyzer. After that, only the surface FME was measuring, while developing a gradient in the pool. From October 30th 09:00, we linked the FME in 29 cm to the analyzer again, which adapted to the higher concentration in that depth afterwards. After validating that the CO2 concentration was lower in 2 cm than in 29 cm, we turned on two aquarium pumps (dashed vertical line) to mix the water column, resulting in the concentrations to converge.

After we validated that the FME is able to measure different CO_2 concentrations at small vertical scales under lab conditions, we deployed an FME under real field conditions. A single FME was placed directly below the water surface of a shallow pond together with a standard CO_2 probe in 25 cm depth (Figure 2 B). The FME and the CO_2 probe measured undersaturation of CO_2 in the pond during the entire period. After deploying the two devices in the evening of September 25th, both of them equilibrated to the *in-situ* concentrations. However, the FME equilibrated faster to the in-situ concentration (T_{90} = 36 min) so that we observed the night-time increase of CO_2 already after sunset. The CO_2 probe needed until midnight to adapt to the local CO_2 concentration (T_{90} = 72 min). In general, the FME measured higher concentrations than the CO_2 probe, which was likely due to the fact that the actual concentration of CO_2 at the surface was higher because of diffusive CO_2 uptake from the atmosphere. During two rain events on September 26th, the FME recorded a sudden increase in CO_2. The increase of CO_2 measured by the CO_2 probe was always delayed and slower when compared to the measurements of the FME (Figure 6).

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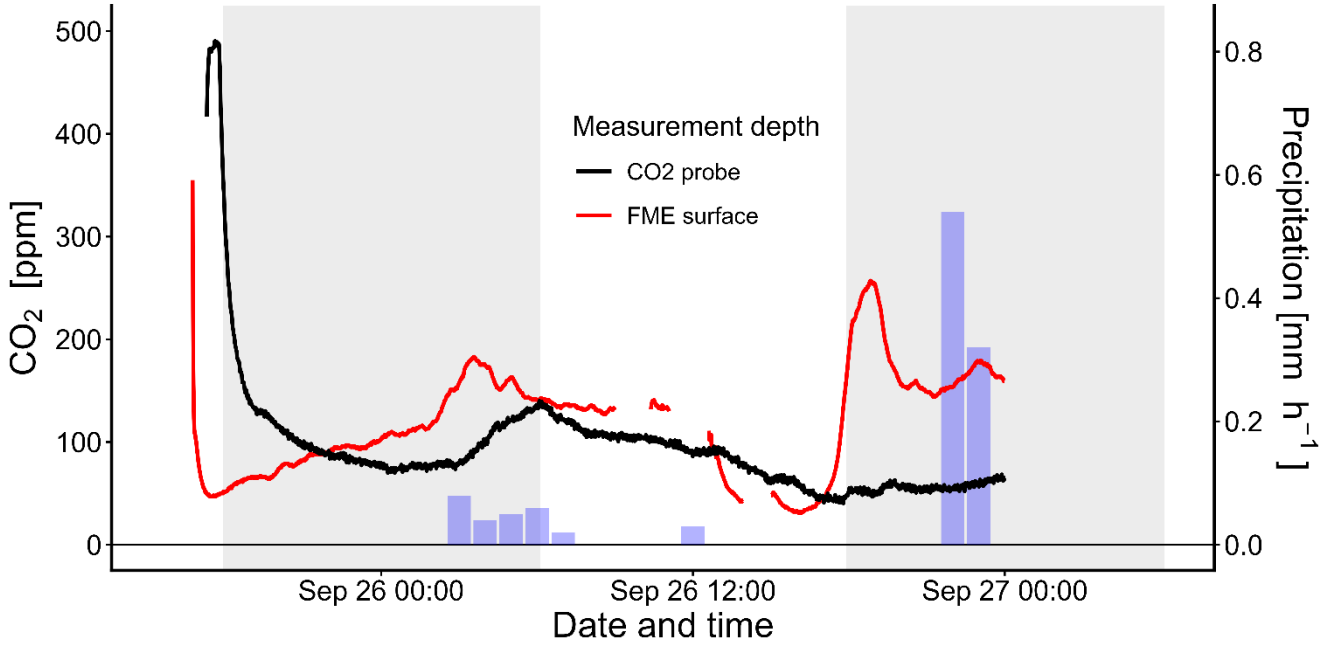

Figure 6: Time series of CO_2 measurements with Floating Membrane Equilibrator alongside a conventional CO_2 probe in a field test. Measured CO2 mixing ratios are displayed on the left y-axis. The FME (red line) and the CO2 probe (black line) were placed at 2 cm depth and 25 cm depth, respectively. Hourly precipitation (mm h^{-1}; right y-axis) is shown as blue columns.

4 Discussion

Vertical resolution

The Floating Membrane Equilibrator successfully measured CO_2 concentrations at both high vertical and temporal resolutions. The design allowed us to measure CO_2 concentrations at centimeter-scale vertical resolution at the surface. While we clearly saw differences between measurements at the surface and 25 cm depth, the exact depth of measurement and vertical resolution of the FME is difficult to quantify. It is determined by the thickness of the silicon tubes (6 mm) and the precision of the vertical positioning of the FME. According to our experience it is possible to adjust the depth of each corner relative to the water surface with a precision of about 5 mm. If we assume that the FME measures the CO_2 concentration in the water layer 1 mm around the tubes the vertical resolution of the measurement is between 6 and 13 mm.

During the development of the FME we feared that contamination of the measurement by atmospheric air might be an issue. That's why in a first version of the FME the silicon tube was fixed to a wooden plate which was floating at the surface. That design prevented access of atmospheric air but led to gas bubble trapping under the plate. Changing to the final open design solved the gas bubble problem. To be sure that the silicon tube does not come into contact with the atmosphere we decided to lower the FME about 5 mm below the surface. Optical observations of the FME during deployment confirmed that the silicon tubes were permanently submerged – even under wavy conditions (Video S1, see Sect. "Video supplements"). This was

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facilitated by the flexible construction which partly buffered wave movement. This means that under the conditions of our field measurements the FME measured the CO_2 concentration 8 – 12 mm below the water surface. Thus, we were able to measure nearer to the surface than any existing method, but did not measure directly at the very surface like equilibration chambers would (Rudberg et al., 2024).

Temporal resolution

The equilibration times in our tests were comparatively high. However, they were obtained under calm, wind-still conditions and with fixed gas flows in the analyzer. Faster equilibration times are usually achieved by increasing the water side turbulence, for example with pumps installed near the probes. However, the FME is specifically designed to avoid disturbances, as this could destroy natural heterogeneity in the water. Despite the longer equilibration times under still conditions, our results show that the FME can nevertheless resolve rapid changes when turbulence or sudden shifts in CO_2 concentration occur. In the pool experiment, the formation of a gradient disappeared within minutes once we activated the pumps, and the FME measurement immediately reflected the homogenization of the water column. Similarly, in the field campaign, the FME tracked a sharp increase in surface CO_2 following a rain event, while the conventional probe reacted much slower. Conventional methods, such like probes at depths free of atmospheric contamination or manual headspace samples often miss short-lived disturbances such as wind gusts or rain, which affect gas exchange at the surface. The CO_2 probe did not measure a notable signal of that event, while the FME detected it directly. Two features of the FME contribute to this advantage: proximity to the water surface is much lower and the constant air flow in the tubing, which enhances diffusive exchange on the gas side compared to passive probe membranes (Colson and Michel, 2025). Existing fast responding CO_2 measuring systems (DeGrandpre et al., 1995; Frankignoulle et al., 2001; Xiao et al., 2020) may be able to trace such fast events but lack spatial resolution. The FME allows to study CO_2 dynamics directly at the water surface with sufficient temporal resolution.

Application of the FME

Applying the FME, we detected rapid changes in CO_2 concentration at the water surface, which conventional in-situ probes would have missed. First, in a controlled pool experiment we observed that the near-surface concentration gradient, which formed due to diffusive CO_2 loss to the air, was quickly destroyed when turning on water pumps in the pool, hence mixing the water column to homogeneous concentrations. This showed that the FME can track fast shifts in surface conditions within minutes. During our field measurement in the fish pond, we showed a clear effect of a short rain event on the CO_2 concentration at the water surface. In contrast, the conventional CO_2 probe did not capture a clear signal of this event, likely due to a combination of its slower response time and its greater distance from the water surface. Such short-term events are of particular importance because they can disproportionally contribute to total CO_2 fluxes (Podgrajsek et al., 2014; Erkkilä et al., 2018), but often remain undetected due to lack of proper temporal resolutions and response times.

The FME also revealed increasing CO_2 concentrations near to the water surface at night. Such diurnal fluctuations of CO_2 directly at the water surface are probably a widespread phenomenon which cannot be detected by standard methods. In the

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24 hours from September 26th 00:00 to September 27th 00:00, the submerged probe would have underestimated the surface concentration – and thus CO_2 flux – by ~30-35 % on average, with nighttime peaks reaching ~2.7-fold underestimation. The ability of the FME to measure not only gradual day to day changes, but also fast fluctuations, provides an improved basis for water-atmosphere flux calculations.

The thin boundary layer model requires accurate surface CO_2 concentration measurements to calculate fluxes. Our FME provides more representative surface CO_2 concentrations than traditional methods. This may be of particular importance when calculating the gas transfer coefficient k from in situ fluxes and concentrations. However, these comparisons often go along with discrepancies between EC measurements and TBL predictions, which have been partly attributed to unresolved near-surface heterogeneity (Heiskanen et al., 2014; Erkkilä et al., 2018). Therefore, the FME is a new method to resolve fine scale gradients and short-term variability to reduce uncertainty in k and improve method comparisons.

Limitations

Despite its strengths, the FME has some limitations. One of the most critical limitations is condensation inside the tubing due to diffusion of water vapor. This challenges the airflow and functionality of the connected CO_2 analyzers. It can be controlled by installing water traps or desiccant in front of the air intake of the analyzer. We observed light growth of biofilm on the outside of the tubing after about 48 hours. Any organisms settling on the device may influence CO_2 measurements. These limitations can be managed with periodic maintenance. However, they also prevent the FME from being used unattended for long-term monitoring. Further, strong wind or high waves may cause currents that can pull the FME under water (Video S2, see Sect. “Video supplements”).

Conclusion

The Floating Membrane Equilibrator (FME) provides new opportunities to measure CO_2 at an unprecedented proximity to the water-atmosphere interface, while maintaining high temporal resolution. This will help to resolve differences between established methods of aquatic CO_2 flux determination and advance our capacity to quantify those fluxes with greater accuracy. Beyond CO_2, the design could be transferred to other gases to refine greenhouse gas budgets. The simple and robust design of the FME offers substantial potential for technological refinement, further improving our ability to measure CO_2 dynamics at the water-air interface. With further optimization, FMEs may help to better quantify gas transfer velocities from simultaneous concentration and flux measurements and contribute to closing the gap between studying small scale processes at the water surface and ecosystem fluxes.

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Appendix A:

Table A 1: T90 equilibration times of the Floating membrane equilibrator in different experiments, including starting mixing ratio and equilibrium mixing ratio.

Site description	CO_2 Start [ppm]	CO_2 Equilibrium [ppm]	T90 [min]
Rappbode Reservoir	454	1566	45
Möhne Reservoir	438	608	26
Magdeburg pond	411	6343	42
Lab pool	910	3358	54
Rappbode Reservoir	412	118	21
Königswartha pond	430	48	36

Code availability

Code is available upon request to the corresponding author.

Data availability

Data are available upon request to the corresponding author.

Video supplement

The video supplement is available online via the TIB AV-Portal under the DOI https://doi.org/10.5446/s_1995.

Author contribution

All authors contributed to the study's conception and design. Field measurements were carried out by PA and VB. PA and VB performed the data analysis. PA wrote the first draft of the manuscript, and all authors contributed to the final version. All authors read and approved the final manuscript.

Competing interests

The authors declare that they have no conflict of interest.

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