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ARTICLE

URBAN AQUEOUS SYSTEM AS A HOTSPOT FOR WATER-ATMOSPHERE EXCHANGE OF CH_4

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Abstract: *Methane (CH_4), a potent greenhouse gas, has negative radiative impacts on the atmosphere, reaching up to 20 times compared with CO_2. However, mitigating CH_4 emissions from its sources, either natural or anthropogenic, will reduce global warming consequences in the short run, given that CH_4 has a short lifetime which takes approximately a decade.*

The urban aqueous system is part of the natural sources of CH_4 in the urban area, which could be influenced by microorganisms or increased human activities and the discharging of sewage water into the system.

Recent studies have stated that high organic matter in river sediments is the primary source for increased production of CH_4 in the river bed. However, the contribution of the urban aqueous system in CH_4 annual fluxes is still uncertain, so a research gap needs to be filled to understand further CH_4 emissions from this system.

This study aims to detect CH_4 content in the urban aqueous system and identify locations with high concentrations to establish further investigation and determine the factors behind these concentrations.

In our study, the aqueous system is classified into three main categories: rivers, lakes, and ponds. Water samples were collected from each of these sub-systems for estimating CH_4 concentration. Samples from Somes River were gathered from three segments: upstream, urban segment within the urban area, and downstream river. Moreover, samples were also taken from lakes and ponds, considering spatial even distribution between sampling points.

Our samples were analyzed via head-space extraction method using Tunable Diode Laser Absorption Spectroscopy (TDLAS), which precisely detects emissions up to 0.1 ppmv.

The results showed that water samples are oversaturated with CH_4 concentration. In other words, the aqueous system in the selected urban area represents an active hotspot for CH_4 exchange from water into the atmosphere.

However, CH_4 fluxes should be quantified to understand better the contribution of the urban aqueous system to the CH_4 budget and to take appropriate abatement measures for reducing these emissions.

Keywords: greenhouse gas, methane (CH_4), urban area, headspace method, aqueous system, gas concentration.

INTRODUCTION

Methane (CH_4) is a potent greenhouse gas (GHG) with radiative forces of more than 86 times CO_2 in terms of global warming potential (GWP) over 20 years (Kang et al. 2016). Eliminating methane emissions from the atmosphere will be significantly noticed in the short term which will also accelerate the process of reversing the climate's negative effects due to the short lifetime of approximately 10 years (Fernandez et al. 2022; Kang et al. 2016; Lamb et al. 2016; UNEP 2021).

Methane emissions have gradually increased since the industrial era due to growing anthropogenic activities of agriculture, fossil fuel combustion, and waste disposal.

The methane budget is attributed to high uncertainty; therefore, precisely quantifying its sources and sinks will be the first toward its abatement (Saunois et al., 2020). Wetlands, permafrost, oceans, lakes, rivers, termites, and wild animals are examples of natural sources. While rice paddies, ruminants, waste management activities, fossil fuel, and biomass burning are among the anthropogenic ones responsible for more than half of CH_4 total emissions (IPCC 2013).

However, the oxidation of CH_4 in the stratosphere and the troposphere is considered an essential sink of CH_4 (Cuna et al. 2008; Isaksen et al. 2014). Moreover, methanotrophic bacteria is another CH_4 sink that oxidizes about 45 – 85 % of soil CH_4, depending on soil humidity and texture (Tate 2015). Although sediments in anoxic environments are potential sources for methane production, the upper water columns with high dissolved oxygen represent an oxidation layer for these emissions (Magen et al. 2014).

Gas exchange between air and water is a biochemical cycle controlled by environmental factors. These factors can be determined through air-water exchange characterization. Some of these factors are: rain, wind speed, bubble entrainment, stability of the boundary layer, and surface films (Wanninkhof et al., 2009). Wind speed affects gas transfer and fluxes from water by causing surface turbulence and bubble hatch push out the gas, but it's not the only factor influencing water-air gas transfer (Wanninkhof 1992).

The wide range of CH_4 sources and sinks increases the confusion in effectively estimating its actual annual budget (Saunois et al., 2020; Ito et Inatomi 2012). In other words, it isn't very easy to determine all the environmental and biological implicated factors in the CH_4 budget. Using one approach in this estimation doesn't guarantee the inclusion of all CH_4 sources and sinks. As a sequence, missing the understanding of the actual budget of CH_4 results in inefficiently applying appropriate measures for reducing its emissions (Ito et Inatomi 2012).

Urbanization and land use policies have shifted lands from being a CH_4 sink into representing a potential source (De Bernardi et al. 2022; Harris et al. 2018). In contrast, land use management policies like afforestation can reverse these effects and promote CH_4 soil consumption (Benanti et al. 2014). In the urban area, traffic and high energy demand are main sources of anthropogenic CH_4 in addition to the leak from natural gas distribution networks (Isaksen et al. 2014).

Recent studies of methane emissions in Urban Areas characterized sources of natural gas leaks (Chamberlain, Ingraffea, et Sparks 2016; McKain et al. 2015; Phillips et al. 2013) and to sewer systems (Beelen et Parker 2022; Guisasola et al. 2008; Kyung et al. 2017).

Water systems contribute to CH_4 annual budget of approximately 103 $TgCH_4$ (Bastviken et al. 2011), but the actual contribution of the aqueous system in the urban area needs to be more studied in the literature. Many studies were interested in understanding CH_4 concentration in water resources for health evaluation and inflammable potential estimation of CH_4 from these resources (Magen et al. 2014)

Water bodies like rivers and streams are considered important sources of CH_4 emissions in the urban area, mainly due to land use changes after the industrial era leading to water pollution from wastewater. The contribution of polluted urban rivers to CH_4 annual fluxes is uncertain (Wang et al. 2020).

However, recent studies estimated methane emissions from urban rivers (Hu et al. 2018; Yang et al. 2015; Zhao et al. 2022) and have determined that high concentrations of dissolved CH_4 are influenced by urban pollution and by the discharging of wastewater into rivers in addition to the land-use changing policies. Nevertheless, other studies have not covered the entire aqueous system in the urban area, particularly in Romania.

While the urban aqueous system is poorly covered by the literature and almost neglected, it is important to determine the actual position of the urban aqueous system from CH4 emissions as either a potential contributor or an essential sink.

This study is a preliminary exploration to determine CH_4 concentration in urban aqueous systems. It also represents a reference for further investigation to quantify the actual contribution of these systems in the CH_4 budget to apply specific abatement strategies.

MATERIALS AND METHODS

The sampling locations were selected based on spatial distribution by considering the variety of the surrounding activities.

About 49 water samples were collected, in the period between December 2022 and January 2023, from 49 different locations; in addition to the recording of their geographic coordinates and site description to establish a clear image of the actual locations and for relatively analyzing them spatially, **Figure 1**.

A liter of water was collected from each sampling point, shown in **Figure 1,** using a one-liter sampling bottle. The bottles were entirely filled with water slowly down the side to avoid bubbles production or entrapping air inside the sample, and to keep the dissolved gas in the samples not affected until the analysis process.

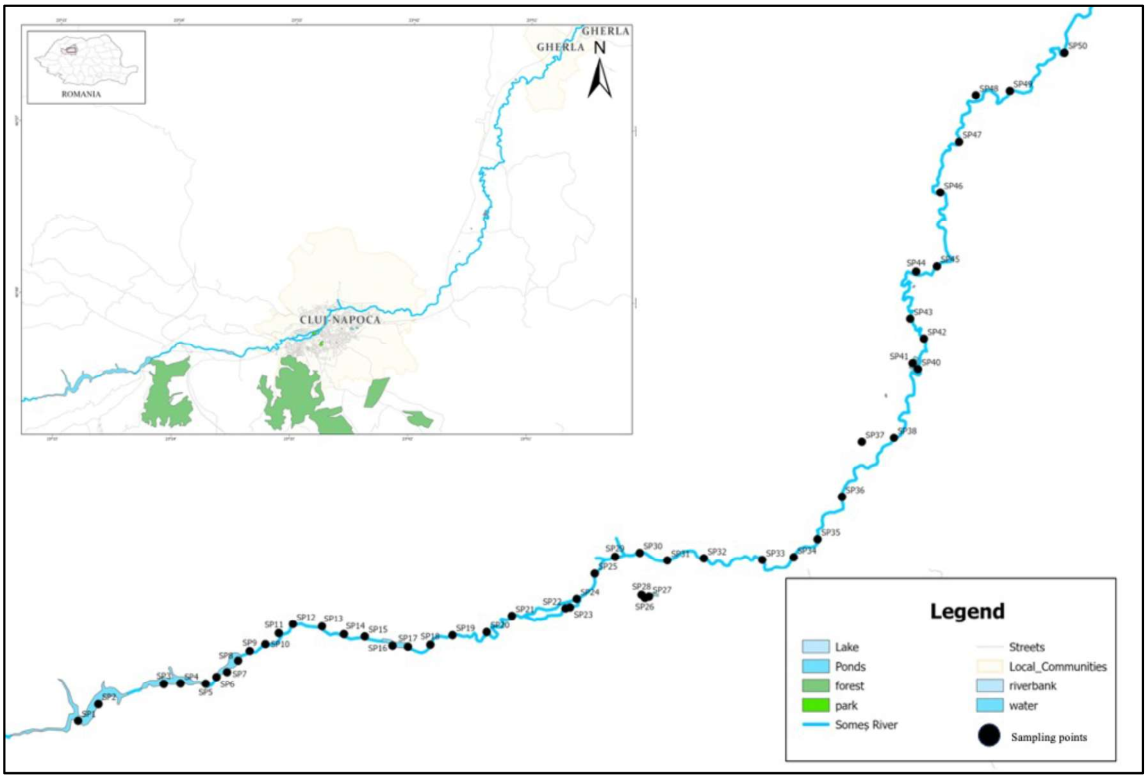

Figure 1. Water sampling locations were distributed evenly over the urban aqueous system from which one liter of water was collected from each site.

The samples' temperature was raised to room temperature of approximately 18˚C. A subsample of 700 mL water was transferred from the sampling bottles into a 1000 mL sample vial, while a volume of 300 mL was filled up with the ambient atmospheric air to function as a headspace.

However, ambient air inside the lab was circulating in order to keep on the actual atmospheric background and to avoid any influence from precedent CH_4 measurements to affect the measuring of the next ones, and also to effectively recalibrate the Tunable Diode Laser Absorption Spectroscopy (TDLAS) to be prepared for the following CH_4 estimation.

Samples were analyzed on the same day of collection to reduce the chance of any potential influence from biological activities or any chemical reaction inside the samples and to skip the step of adding preservatives to the samples.

Each sample is described in a table indicating the sample number, coordinate, spatial description, and its order in the process CH_4 measurement. Sampling bottles were arranged according to their field description to effectively conduct laboratory analysis.

After filling up the sample vial with 700 mL of water sample X and letting the 300 mL of the headspace be filled up with atmospheric air, the vial septum sample was shaken for 2-3 minutes in order to equilibrate the gas concentration between the headspace and the water, **Figure 2**.

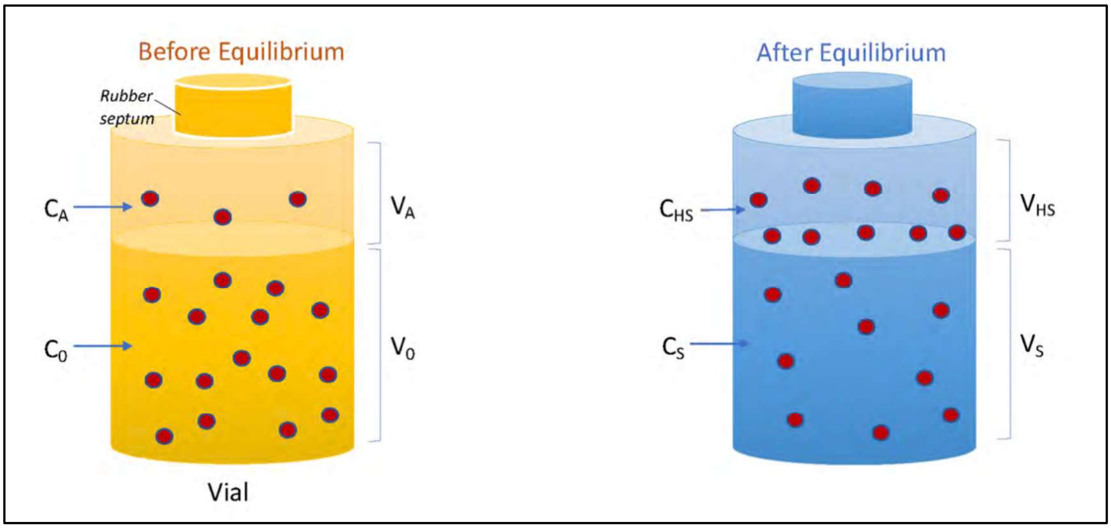

Figure 2. Headspace conceptual method before and after equilibrium, where C_A is the concentration of CH_4 before the equilibrium, C_0 is the initial concentration, and V_A and V_0 are the initial volumes of the headspace and the sample, respectively. After the equilibrium, C_{HS} is the headspace concentration, C_S is the sample concentration, V_{HS} is the headspace volume, and V_S is the sample volume.

After the equilibrium, two needles connected to the TDLAS were inserted simultaneously inside the vial through its rubber septum cap. At the same time, the TDLAS was just calibrated to the background of the ambient atmosphere, **Figure 3**.

The measurements lasted 1 minute for each sample, and the readings of CH_4 concentration in the headspace were recorded up to 0.1 ppmv s^{-1}. The delay time was estimated to be between 3-4 seconds.

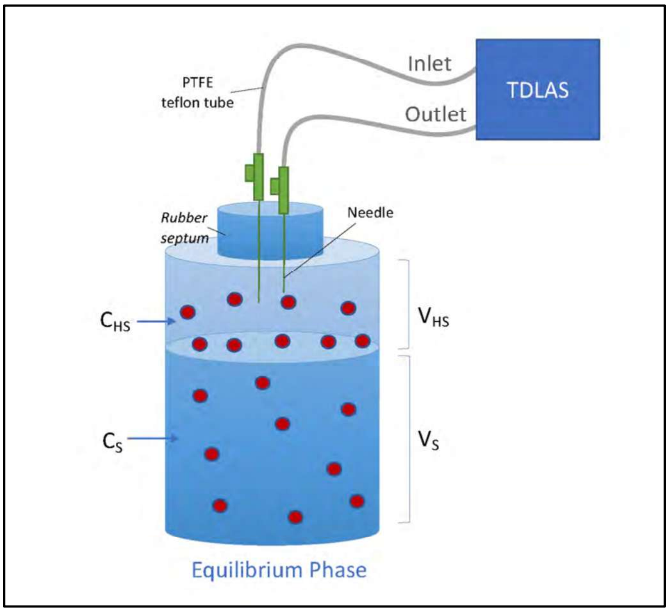

Figure 3. Methane headspace concentration measurement technique after the equilibrium by simultaneously inserting inlet and outlet needles connected with TDLAS. Given that the vial headspace is 300mL, it's recommended to take caution for keeping the hands away

Calculation

In our headspace theory, no gas chromatography was used; the headspace CH_4 concentration was determined via TDLAS, as described in **Figure 3**. In the headspace method and at a specific temperature, an equilibrium is reached between the volatile in the sample and the headspace.

The volume of the sample would almost remain the same after the transfer of the analyte into the headspace. Therefore, the sample volume will be considered the same, $\mathbf{V_0}$ equals $\mathbf{V_s}$, and $\mathbf{V_{HS}}$ remains unchanged by default.

The ratio phase (**ß**) is the ratio of the headspace volume $\mathbf{V_{HS}}$ to the sample volume $\mathbf{V_S}$ (Kolb et Ettre 2006), **Equation 1**.

$$\mathbf{ß = V_{HS}/V_S} \qquad (1)$$

Also, the concentration in the original sample $\mathbf{C_0}$ equals the initial amount of the analyte $\mathbf{Q_0}$ (CH_4 in our case) divided by the sample initial volume $\mathbf{V_0}$ in the vial (Kolb et Ettre 2006), **Equation 2**.

$$\mathbf{C_0 = Q_0/V_0} \qquad (2)$$

The sum of the amount of CH_4 in the headspace and the sample after the equilibrium equals its initial amount in the sample before the equilibrium.

The concentration equilibrium is influenced by the thermodynamic force that can be expressed by the coefficient (K), which is the ratio between concentration in the sample $\mathbf{C_S}$ and the concentration in the headspace $\mathbf{C_{HS}}$ (Kolb et Ettre 2006; Snow et Bullock 2010), subtracting the amount in the initial headspace (which equals the ambient amount in the atmosphere), because the TDLAS reading doesn't represent the added amount of CH_4 from the initial sample, **Equation 3**.

$$\mathbf{K = C_S/C_{HS}} \qquad (3)$$

After considering all the relations mentioned above, the amount of CH_4 in the initial sample, **Equation (2)** becomes:

$$\mathbf{Q_0 = C_0.V_{0\ Before}}$$
$$\mathbf{Q_0 = (C_S.V_S + C_{H.S}.V_{HS})_{After} - C_A.V_{HS\ Before}} \qquad \mathbf{(4)}$$

By substituting **Equation 3** in **Equation 4**:

$$\mathbf{Q_0 = K.C_{HS}.V_S + C_{HS}.V_{HS} - C_A.V_{HS}} \qquad \mathbf{(5)}$$

For determining CH_4 concentration in the initial sample, **Equation 5** is rearranged in terms of $\mathbf{C_0}$ by combining **Equations 2** and **5** in **Equation 6.**

$$\mathbf{C_0 = K.C_{HS} + \frac{V_{HS}}{V_S}\,(C_{HS} - C_A)} \qquad \mathbf{(6)}$$

The solubility coefficient K will be considered as the Bunsen coefficient 0.035 L_g/L_s at atmospheric pressure 1 atm and at room temperature 18°C with 0 - 10 salinity (Donval et Guyader 2017). Bunsen coefficient is expressed by temperature and salinity. It is a volume of gas dissolved in water at specific temperature and salinity values (Wiesenburg et Guinasso 1979).

According to Weiss (1970), the atmospheric equilibrium of the gas depends on its solubility in a relationship that integrates the temperature and the salinity.

RESULTS AND DISCUSSIONS

After analyzing all the collected 49 samples and obtaining the water concentration of CH_4 in the original sample by headspace method applying **Equation (6)**, the results can be illustrated in **Figure 4.**

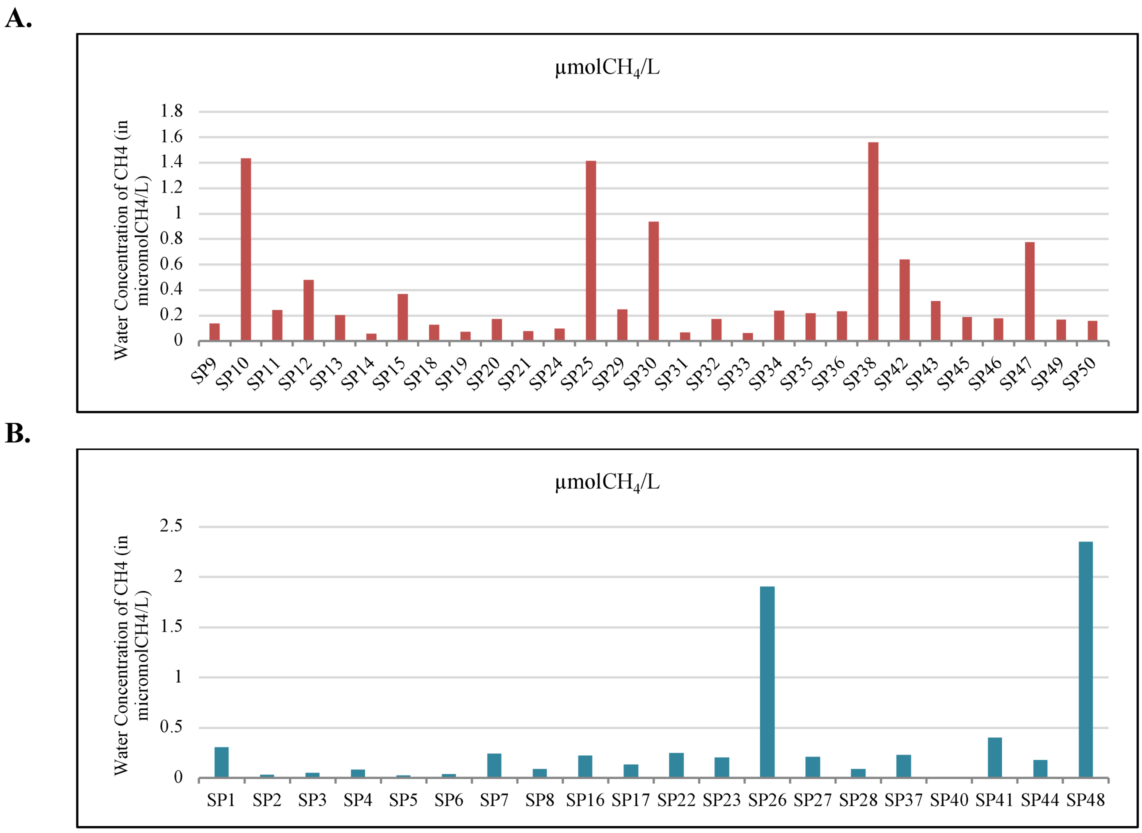

Figure 4. Methane concentration in water samples (in µmol/L) for samples collected from **A**. Someș River; **B.** Lakes and ponds.

It's important to mention that the reason for the separation of the results for river samples and lakes and ponds is related to the differences between these systems and to observe the influence of any changes over river segments before and after the urban area to estimate the impact of the urban activities down-stream. Moreover, the total number of samples is 49, which is less than the real number in the initially prepared plan for fieldwork. This change is due to the inaccessibility of some sampling locations.

The concentration of CH_4 in the samples varied between a minimum value of 0.03 µmol/L to a maximum value of 2.35 µmol/L, with a mean value of 0.38 µmol/L, **Table 1**.

Table 1. Statistics of CH_4 street-level concentration in (ppm) of the four land-use types, including mean, maximum, and minimum values in addition to the standard deviation (SD)

Min	Max	Mean	SD
0.03	2.35	0.38	0.51

Approximately 90% of the samples have values over 1 µmol/L, and 10% have values between 1 – 2.35 µmol/L. However, given that the estimated background level is 2.0 ppm, the saturation value of CH_4 concentration in the water sample is calculated as 0.01 µmol/L.

By comparing all CH_4 water concentration values with this saturation level, the result is that water samples from all sampling locations are oversaturated with methane, as it's evident by also that the minimum value is higher than the saturation estimate.

It's clear that sampling locations within and after the urban area have high dissolved CH_4, similar to sampling locations in lakes and ponds.

The results obtained by (Wang et al. 2020) stated that the dissolved CH_4 varied between 0.4 – 27 µmol/L with an average of 5.4 µmol/L which seems to be significantly similar to our results of high CH_4 concentrations.

In other words, our survey of dissolved methane in the urban aqueous system shows an oversaturated system of dissolved CH_4.

Therefore, this system is a potential source of CH_4 in the urban area.

The study of Hu et al., (2018) stated that CH_4 concentrations in water were positively correlated to temperature but negatively to dissolved oxygen DO. It also estimated high CH_4 concentrations at polluted water sampling locations.

Large quantities of organic matter in river sediments are the main sources of high river CH_4 concentrations. Nevertheless, we still can't specify the natural source behind these high concentrations in our results if they either come from microbial activities of the methanogenesis in the sediments or being influenced by urban pollution, or even from discharging of wastewater into this system.

Here, we can confirm that our urban aqueous system is a hotspot for CH_4 exchange from water into the urban atmosphere. Therefore, the degree of this contribution and the actual fluxes should be investigated in future studies, beside the reason behind them.

CONCLUSIONS

Methane is one of our main approaches toward gaining short-term climate benefits. With its diverse sources and sinks, determining its actual budget has become a challenging mission.

Urban Areas are responsible for more than 50% of CH_4 emissions following its growing anthropogenic activities (IPCC 2013).

Previous studies have not explored the aqueous system in the urban area for determining its contribution to CH_4 annual budget. And the degree of uncertainty is relatively high which puts more challenges and stresses the need for effectively understanding the system's behavior.

In this study, CH_4 concentration in each water sample was determined by applying the headspace method combined with the TDLAS estimation.

The results of this study revealed a high concentration of CH_4 in the urban aqueous system and even a case of oversaturation in all water samples concluding that this system is an active hotspot for CH_4 exchange from water to the urban atmosphere.

This study represents a reference for future urban CH_4-relevant studies. Nevertheless, it is a preliminary study that points out the significance of the aqueous system in atmospheric methane, which in turn influences the total methane coming from urban sources. Still, it failed to associate environmental factors and seasons with these concentrations.

This observation of oversaturation points can result from water pollution due to urbanization and industrialization, or simply from active microorganisms in the sediments.

However, the sources behind these concentrations as well as the actual contribution of the aqueous system to the CH_4 budget, should be determined in future studies.

Also, methane fluxes from this system should be quantified spatially and temporarily in this system in a long-term study.

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