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Correction factors for large-scale greenhouse gas assessment from pulp and paper mill sludge landfill sites

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ABSTRACT

Assessments of greenhouse gas (GHG) emissions in managed areas are facing various challenges. A non-flow-through, non-steady-state (NFT-NSS) chamber coupled to a frame permanently inserted into the landfilled substrates is a standard method for quantifying GHG emissions in managed areas, such as pulp and paper mill sludge (PPMS) landfill sites. Frequent measurements are needed to minimize uncertainties on GHG emission factors at the landfill site scale. However, maintaining a frame inserted into the substrates for a long time period is often impossible due to landfilling management operations. Therefore, GHG measurements using NFT-NSS chambers placed directly on substrates' surface could be an interesting option. Our objectives were to determine the relationships between CO_2, CH_4, and N_2O fluxes measured with (F +) and without (F-) a frame inserted in the substrates' surface and to develop correction factors for fluxes measured without a frame. Measurements were made at different PPMS landfill sites in the province of Québec, Canada. Stronger GHG flux relationships were observed at the provincial (across sites) than the specific site scale: the variance in GHG fluxes from F- chambers explained up to 80 % of variance in fluxes from F + chambers. The measured CO_2, CH_4, and N_2O fluxes in F- chambers were on average 53, 78, and 63 % lower, respectively, than those estimated by the models at provincial scale. The correction factors developed with this approach could greatly extend the number of sites where *in situ* GHG measurements can be done and would help refining GHG inventories at the provincial and national levels.

1. Introduction

Pulp and paper mill sludge (PPMS) is the main organic by-product generated by wastewater treatments in the pulp and paper industry. PPMS are mainly disposed of by landfilling, application to agricultural land, and combustion for energy recovery (Camberato et al., 2006; Faubert et al., 2016; Simão et al., 2018; Turner et al., 2022; Vilarinho et al., 2022; CANMET, 2005). Global efforts and policies towards the development of circular economy and industrial ecology put forward a reduction in PPMS landfilling to lessen greenhouse gas (GHG) emissions and mitigate climate change (Amândio et al., 2022; Faubert et al., 2016; Faubert et al., 2019; Vilarinho et al., 2022). For instance, in the province of Québec (Canada), where the annual PPMS production is 0.9 million tons, proportions of PPMS landfilled have recently decreased from 25 to 14 % while application to agricultural land increased from 33 to 48 % (RECYC-QUÉBEC, 2019; RECYC-QUÉBEC, 2023). GHG emissions from PPMS landfill sites represent less than 4 % in the waste sector in Québec and Canada (Environment and Climate Change Canada, 2023b; MELCCFP, 2022), but they can be a hotspot in the GHG emission budget at the pulp and paper mill scale (Heath et al., 2010), thus promoting

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reduction. In this context, it becomes essential to accurately quantify GHG emissions from the different PPMS management options to clarify the impact of the industry on climate and refine national GHG inventories.

Large uncertainties are associated with current GHG emission factors in PPMS landfill sites. Better estimates are required to reliably assess changes in GHG emissions as a function of PPMS management at the jurisdiction scale (Faubert et al., 2016; Faubert et al., 2019; IPCC, 2019). Some studies quantified GHG emissions from PPMS landfill sites via modeling or *in situ* measurements at the pilot scale (Faubert et al., 2016; Faubert et al., 2019; Heath et al., 2010), and suggested that, in the long term, PPMS landfilling emits up to three times more GHGs than land application (Faubert et al., 2019). However, these estimates accounted only for CH_4 emissions while N_2O emissions, with its 100-year global warming potential (GWP) of 298 that of CO_2 (IPCC, 2007; UNFCCC, 2014), are not accounted for in current inventories (Faubert et al., 2019; IPCC, 2019).

As for landfill sites of municipal solid waste (MSW), CH_4 emissions from PPMS landfill sites are currently estimated by the first order decay (FOD) method (Environment and Climate Change Canada, 2023c; Faubert et al., 2019; IPCC, 2019; NCASI, 2005). The FOD principle assumes that the degradable organic carbon (DOC) in waste decomposes throughout time, resulting in CH_4 and CO_2 emissions (IPCC, 2006; 2019). One important parameter of the FOD model is the CH_4 generation potential (L_0, which can be reported in m^3 CH_4 per mass of waste), which is related to the DOC (IPCC, 2019). In Canada, the national GHG inventory report uses a Tier 1 approach based on the default DOC values from IPCC (2019) to report GHG emissions from the pulp and paper industry (Environment and Climate Change Canada, 2023c). The latter uses a Tier 2 approach in which the FOD method has a default L_0 integrated in the model of the National Council for Air and Stream Improvement (Faubert et al., 2019; NCASI, 2005; 2022). In MSW landfill sites, discrepancies are reported in studies comparing CH_4 emissions assessed by the FOD method and direct field measurements (Chakraborty et al., 2011; Di Bella et al., 2011; Mou et al., 2015; Scheutz et al., 2022), raising doubts in the uses of default L_0 values with Tier 1 or Tier 2 approaches (Vu et al., 2017). The same uncertainties apply in PPMS landfill sites (Faubert et al., 2016; Faubert et al., 2019). Moreover, N_2O emissions from MSW landfill sites are currently discussed by the Intergovernmental Panel on Climate Change (IPCC) guidelines but concerns remain (IPCC, 2019). For PPMS, N_2O emissions may represent 73 % of CO_2-equivalent emissions from landfilled mixed PPMS (measured at pilot scale) (Faubert et al., 2019). Addressing current uncertainties in Tier 1 and 2 approaches and building a site-specific Tier 3 method for the quantification of GHG emissions from PPMS landfill sites requires field measurements at the industrial scale to adjust parameters of the current model (Faubert et al., 2019). Therefore, the very first step to calibrate the FOD method by adjusting the L_0 factor values and investigate the N_2O emissions from PPMS landfill sites is to measure GHG fluxes directly on site (IPCC, 2006; 2019). Chamber measurements can be used for such purposes (Faubert et al., 2019; IPCC, 2006; 2019).

In managed and natural areas, CO_2, CH_4, and N_2O fluxes are often measured at the soil-air interface using non-flow-through, non-steady-state chambers (NFT-NSS) (Maier et al., 2022; Rochette, 2011; Rochette and Bertrand, 2008). The GHG fluxes are calculated using the concentration change rate at different measurement intervals (*e.g.*, daily, weekly, monthly) (Rochette and Bertrand, 2008). The NFT-NSS method for *in situ* GHG measurements consists of a chamber tightly fitted onto a frame permanently inserted in the top soil (Rochette and Bertrand, 2008). Maintaining a frame in place may be difficult in PPMS landfill sites though, due to frequent landfilling operations and intense machinery traffic. The GHG measurements using NFT-NSS chambers placed directly on the PPMS surface could be a practical alternative. However, chamber deployment without a frame may lead to an underestimation of GHG fluxes compared with a chamber deployed on a frame (Rochette and Eriksen-Hamel, 2008). Therefore, correction factors may be required to report actual GHG fluxes measured with the NFT-NSS method without the frame.

The objectives of this study were (i) to determine the relationships between CO_2, CH_4, and N_2O fluxes measured using the NFT-NSS method with (F +) and without the frame (F-) in different PPMS landfill sites in the province of Québec, Canada; (ii) to use these relationships to develop correction factors for CO_2, CH_4 and N_2O fluxes measured without a frame.

2. Material and methods

2.1. Study sites

The GHG flux measurements were carried out in three industrial PPMS landfill sites in the province of Québec, Canada (hereafter called sites 1, 2, and 3 for confidentiality). Site 1 has mean annual air temperature of 2.4 °C and mean annual precipitations of 1180 mm, whereas sites 2 and 3 have mean annual air temperature of 2.8 °C and mean annual precipitation of 931 mm. These data were compiled for the climate normal period of 1981–2010 (Environment and Climate Change Canada, 2023a). The three mills use thermomechanical methods for pulp and paper production. The management practices used in the studied landfill sites are representative of the pulp and paper industry at the provincial scale. The material disposed of in each site is a mixture of bark and mixed PPMS (primary and secondary) (Faubert et al., 2016), with more than 80 % of PPMS. Bark and PPMS contents were homogeneously mixed in all three sites. The three landfill sites had accumulated more than 2 m of PPMS materials when measurements were made.

2.2. Experimental design

In each site, CO_2, CH_4, and N_2O fluxes were measured in 12 pairs of NFT-NSS chambers distributed equally in four zones, each minimally of 100 m^2, and were systematically distributed to account for spatial variability caused by differences in the time course of PPMS application. Each zone contained PPMS of different ages, ranging from freshly applied up to five years since the last application. Each chamber pair was composed of NFT-NSS chambers, one with (F +) and one without (F-) a frame inserted in the PPMS surface with 50 cm between chambers (Fig. 1).

2.3. Gas sampling and analyses

Surface CO_2, CH_4 and N_2O fluxes were monitored once a month from May to October 2017 following the principles of the NFT-NSS method (Rochette and Bertrand, 2008). The chambers were in acrylic (Polyalto Group, Québec), vented, and insulated according to quality control criteria for GHG flux measurements (de Klein and Harvey, 2015; Rochette, 2011; Rochette and Eriksen-Hamel, 2008). On each sampling day, the F + chamber (0.55 m × 0.55 m × 0.15 m height) was tightly fitted onto a wooden frame (0.55 m × 0.55 m × 0.20 m height) inserted at 0.17-m depth into the PPMS materials; bricks were placed on top of the chamber to ensure tightness at time of deployment (Fig. 1). The frames were left undisturbed during the whole measurement campaign. The F- chamber (0.55 m × 0.55 m × 0.14 m height) was gently pushed into the PPMS surface at a maximum of 1-cm depth. A flexible rubber gasket (10-cm width) was fitted around the base of each F- chamber, and a metal chain and surrounding PPMS materials were placed on the rubber gasket at time of deployment to avoid gas leakage (Fig. 1). Although the business-as-usual scenario in PPMS landfill sites involves frequent disturbances by machinery, the study areas on each site were left undisturbed over the course of the experiment. Absence of disturbance was also necessary to respect the basic principles of the NFT-NSS method, especially for F + chambers where frames are fixed during the measurement campaigns (Rochette and Bertrand, 2008).

GHG sampling was carried out according to Rochette and Bertrand

Fig. 1. Photo of a chamber pair designed to measure greenhouse gas fluxes (CO_2, CH_4 and N_2O) in landfill sites of pulp and paper mill sludge (PPMS). Chamber on the right is deployed directly on the PPMS materials without frame (F-) and gently pushed into the surface; PPMS materials are covering a heavy metal chain placed over a flexible rubber gasket (10-cm width) fitted around the F- chamber base to isolate the headspace (blue arrow). Chamber on the left is deployed on a frame (F +) following the principles of the standard method of the non-flow-through, non-steady-state (NFT-NSS) chambers.

(2008). In each chamber, 20-mL air samples were collected from the chamber headspace 0, 4 and 8 min after chamber deployment using a syringe through a rubber septum. The collected air was transferred to 12-mL pre-evacuated glass vials (Exetainer, Labco, High Wycombe, UK). Chamber deployment was done between 9:00 h and 14:00 h EDT on all sampling days. The air samples, along with standards and quality control samples, were analyzed within 15 days using a gas chromatograph (Bruker, model 450) with Ar/CH_4 (95/5) carrier gas through a 1.75-m-long Porapak Q 80/100 column (60 °C) equipped with an electron capture detector for N_2O, and He carrier gas through a 3.6-m-long Hayesep A 80/100 column equipped with a flame ionization detector for CH_4, and for CO_2 after passing through a methanizer (catalyst column, Ni Nitrate 10 %, 400 °C). PPMS CO_2, CH_4 and N_2O fluxes were calculated using equations proposed by Rochette and Bertrand (2008) and were considered greater than 0 when changes in gas concentrations during deployment were above the analytical variability levels (CO_2: 1 ppm; CH_4: 0.05 ppm; N_2O: 0.005 ppm). The fluxes are reported as a CO_2-C, CH_4-C, and N_2O-N basis, but are referred herein to as CO_2, CH_4, and N_2O fluxes for simplicity (Faubert et al., 2019).

2.4. Statistical analyses

The relationships of CO_2, CH_4, and N_2O fluxes between F- and F + chambers were evaluated using linear regressions at the site and provincial (*i.e.*, all three sites together) scales with the JMP Pro software, version 16.0.0 (SAS Institute Inc.). F + was the dependent variable and F- was the independent variable. The normality assumptions was checked by graphical examination of the residuals (Montgomery, 2012; Quinn and Keough, 2002) and the data were all transformed using the Napierian logarithm in the BoxCox procedure before performing the regression analyses. Chamber pairs were excluded from regression analyses when the GHG flux value in F + chamber was lower than in F- chamber. This criterion was used because the F + chamber remains the standard method for GHG flux measurements (Rochette and Bertrand, 2008). A larger GHG flux in F- than in F + chambers would indicate a handling error in the field (*e.g.*, an unintentional manual pressure when gently inserting the F- chamber into the PPMS may cause a gas burst leading to overestimating the flux).

3. Results and discussion

The CO_2, CH_4, and N_2O fluxes in the F- and F + chambers were generally positively and strongly related at all sites (Figure S1). It was thus possible to establish the relationships at the provincial scale by pooling results from all sites (Fig. 2). Indeed, CO_2, CH_4, and N_2O fluxes in the F- chambers explained up to 80 % (regression parameters: r^2 values between 0.78 and 0.80, p-values < 0.0001) of the variance of the fluxes in the F + chambers. Moreover, the values were well dispersed along the regression line for each gas. Therefore, these results indicate that the number of samples was sufficient for the exercise. Most importantly, these prove that using F- NFT-NSS chambers is a valid approach for measuring GHG fluxes and making inventories in PPMS landfill sites where it is not possible to use a frame due to frequent perturbations caused by an intense machinery traffic. Moreover, the GHG fluxes between the F- and F + chambers at the site scale showed some strong positive relationships, except for CH_4 at site 1 (Table S1, Figure S1). Generally, the r^2 values were smaller at the site than at the provincial scale. In addition, differences in the intercept and slope values up to almost five times between sites suggest large inter-site variability that may be caused by differences in local management practices and time since PPMS application. Thus, the site effects on GHG fluxes should be thoroughly investigated in the future.

At the provincial scale, measured CO_2, CH_4, and N_2O fluxes from F- chambers were on average 53, 78, and 63 % lower than the estimated fluxes from F + chambers, respectively. The percentages show the order of discrepancy between the measured F- and modeled F + for CO_2, CH_4 and N_2O fluxes. The magnitude of these discrepancies demonstrates the importance of correcting GHG flux data (see the correction factor for each gas, *i.e.*, regression equation, in Fig. 2) to avoid underestimation of fluxes when using the more practical F- chambers in PPMS landfill sites. Moreover, the use of the provincial scale correction factors appears as a robust approach as (i) r^2 values were generally greater than those at the

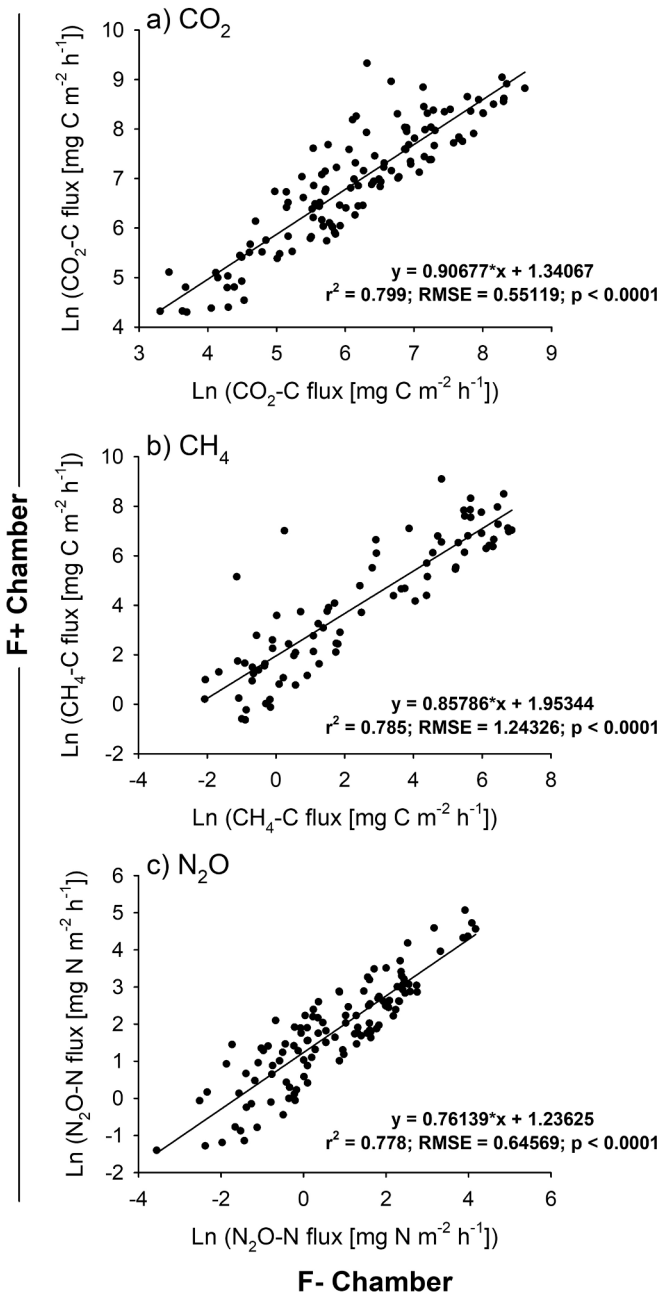

Fig. 2. Biplots of greenhouse gas fluxes of a) CO_2, b) CH_4 and c) N_2O measured on landfill sites of pulp and paper mill sludge (PPMS) in chambers with frame (F+, on Y axes) and without frame (F-, on X axes) inserted in the PPMS surface at the provincial scale (data pooled from three industrial sites), Québec, Canada. Parameters obtained from the linear regressions are shown where the equations represent the correction factors. Number of data points included: a) n = 123, b) n = 84, c) n = 118. Regression analyses were performed on the flux values on a CO_2-C, CH_4-C, and N_2O-N basis and Ln-transformed.

site level and (ii) the correction factors are based on a wider range of GHG flux values that integrate different management practices and environmental conditions. Grouping the sites at a provincial scale is also consistent with the good practice principle of IPCC (IPCC, 2006; 2019) as long as the sites share similar characteristics as observed here (*e.g.*, similar characteristics of the three sites: PPMS type and age of last application of < 5 years, proportion of PPMS in the landfilled material, landfill site management without gas collection system, thermo-mechanical pulp and paper production process and climate). The minimal values observed for CO_2, CH_4, and N_2O fluxes in the F- chambers were 27.3, 0.1, and 0.03 mg $m^{-2}h^{-1}$, while the maximum values were 5503.9, 955.3, and 64.9 mg $m^{-2}h^{-1}$, respectively. In accordance with good practices (WBCSD/WRI, 2004), conservatively, this represents the ranges within which the provincial correction factors can be used for F- chamber measurements, as we have not demonstrated that the relationship still applies outside of these measured ranges. It is also recommended to use enough replicates when measuring with F- chambers as some samples may be excluded due to handling or other errors (as explained in section 2.4). It is challenging to determine a minimum number of replicates when considering possible exclusions as PPMS management and material ages differ within and between landfill sites. In this context, it is recommended to run trial flux measurements with F- chambers on the landfill sites to determine a sufficient number of usable and appropriate flux replicates per PPMS management practice and measurement day to perform reliable statistical analyses and build a GHG inventory.

4. Conclusion

To our knowledge, this is the first time that GHG fluxes are compared in chamber pairs with and without frame inserted in the PPMS surface in large-scale experiments on landfill sites at the industrial mill scale. The CO_2, CH_4, and N_2O fluxes measured at different PPMS landfill sites with the NFT-NSS chamber placed directly on the PPMS surface were closely related to measurements using a frame inserted at 17 cm into PPMS materials. Data relationship was better at the provincial scale, with r^2 values of 78–80 %. For PPMS landfill sites where it is impossible to use frames to monitor the CO_2, CH_4, and N_2O fluxes, the linear regression models developed at the provincial scale could be used to correct fluxes measured with NFT-NSS chambers placed directly on PPMS surface. This approach consists of one of the first steps that could allow the pulp and paper industry to calibrate a FOD model at the site-specific scale. This approach could also greatly extend the number of sites where *in situ* GHG measurements can be done and would help refining GHG inventories at the provincial and national levels in different jurisdictions worldwide.

CRediT authorship contribution statement

Ranieri Ribeiro Paula: Data curation, Formal analysis, Methodology, Validation, Visualization, Writing – original draft, Writing – review & editing. **Mathieu Cusson:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Methodology, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing. **Normand Bertrand:** Conceptualization, Data curation, Formal analysis, Methodology, Validation, Writing – original draft, Writing – review & editing, Visualization. **Sylvie Bouchard:** Validation, Visualization, Writing – original draft, Writing – review & editing. **Martin H. Chantigny:** Conceptualization, Data curation, Formal analysis, Methodology, Validation, Visualization, Writing – original draft, Writing – review & editing. **Julie Lemieux:** Data curation, Methodology, Validation, Visualization, Writing – original draft, Writing – review & editing. **Emna Marouani:** Data curation, Formal analysis, Methodology, Validation, Visualization, Writing – original draft, Writing – review & editing. **Claude Villeneuve:** Conceptualization, Funding acquisition, Methodology, Project administration, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing. **Patrick Faubert:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Methodology, Project administration, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

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