Pilot Study Comparing Eddy Covariance and Dynamic Closed-Chamber Methods for Measuring CO_2 Fluxes above the Hydromagnesite-Magnesite Playas near Atlin, Northwestern British Columbia (NTS 105N/12)

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Introduction

Carbon dioxide (CO_2) sequestration by carbon mineralization in ultramafic materials has emerged as a promising strategy to reduce net greenhouse-gas emissions (e.g., Seifritz, 1990; Lackner et al., 1995; Wilson et al., 2009; Power et al., 2013). This process incorporates CO_2 gas into inert carbonate minerals ($MgCO_3 \cdot xH_2O$) through the aqueous reaction of bicarbonate anions (HCO_3^-) and magnesium cations leached from ultramafic rocks. Carbon mineralization can occur passively via chemical-weathering processes at the surface, or actively by CO_2 injection into the subsurface (e.g., mine-tailings storage facilities and/or in deep geological formations).

The current method used to estimate passive carbon capture in ultramafic mine tailings is mineral quantification, by which a CO_2 sequestration rate is calculated from the weight of precipitated carbonate minerals in tailings samples over an area and time of deposition. Estimates from a recent study revealed that some mines passively offset over 10% of their CO_2 emissions through carbonate-mineral precipitation in their tailings material (Wilson et al., 2014). Accurately measuring passive rates of CO_2 uptake using mineral quantification has proven challenging due to the intensive field and laboratory labour it requires and low spatial and temporal resolution it provides. Accurate quantification of carbon-uptake rates is necessary to validate this novel form of carbon capture and storage. Developing protocols involving environmental monitoring methods that measure real-time CO_2 fluxes will aid mining companies in quantifying their carbon uptake.

Eddy covariance (EC) and dynamic closed-chamber (DCC) systems, two widely accepted environmental monitoring technologies used in soil science and ecosystem science, can potentially be employed in a geological context to measure CO_2 fluxes between the atmosphere and mine waste. Eddy covariance measures the time-averaged covariance between the fluctuations in vertical wind velocity and CO_2 concentration to compute the gas fluxes between the ground surface and the atmosphere. The dynamic closed-chamber method measures the CO_2 concentration over time in a closed-chamber system and computes the flux from the change in gas concentration occurring over time at the soil–atmosphere interface. In previous studies in ecosystems and agricultural sciences, EC and DCC methods have been paired to cross-validate measured fluxes (Norman et al., 1997; Riederer et al., 2014; Wang et al., 2017; Lucas-Moffat et al., 2018). The EC and DCC methodologies have only once been paired at an operating mine site to measure CO_2 and water-vapour fluxes from a waste-rock pile undergoing pyrite oxidation in the presence of carbonate minerals (Kabwe et al., 2005). No conclusions were made about acid neutralization and CO_2 emissions because the signal was mixed with a biogenic source. Additionally, EC has been implemented at deep geological CO_2 injection sites to quantify, in real-time, surface leakage of CO_2 into the biosphere (Burba et al., 2013). Pairing EC and DCC methods, to target low carbon emissions at mine sites where the ore is hosted in ultramafic rock, may provide the quantification and verification needed to determine CO_2 sequestration potential; nonetheless, the fact remains that there is a clear lack of studies testing the effectiveness of these methods in this geological setting.

In this study, the EC and DCC methods were employed on the hydromagnesite-magnesite playas of Atlin, British Columbia (BC). These playas have been forming at the Earth's surface since the most recent deglaciation and comprise an assemblage of calcium and magnesium carbonate minerals

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(Power et al., 2014). It has been proposed that, due to the high concentration of carbonate in the alkaline groundwater, CO_2 is likely to degas. (Power et al., 2014, 2019). This site is of interest because it provides insights on conditions favourable for ex situ carbon mineralization and presents features similar to deep geological CO_2 injection sites with potential CO_2 leakage.

The objectives of this study are to

- detect and compare fluxes occurring at this site using both EC and DCC methods; and
- identify the source of the CO_2 emitted.

The overarching goal is to establish protocols for validating these methods of measuring in real time CO_2 fluxes associated with both passive and active carbon mineralization in ultramafic mine tailings, leading to the development of carbon capture and storage strategies.

Geological Setting

The study site is located in Atlin, northwestern BC, where unique bedrock geology and postglacial history have led to the formation of hydromagnesite-magnesite alkaline playas. Atlin lies 48 km from the Yukon border (59.5818°N, 133.6885°W, at an elevation of 696 m asl; Figure 1). The bedrock is composed of ultramafic and altered ultramafic materials originating in the upper mantle of the oceanic lithosphere, and contains Mg-rich harzburgite and dunite (Ash and Arksey, 1990; Hansen et al., 2005). The ophiolitic material was obducted onto the Stikine and Cache Creek terranes of BC (Ash and Arksey, 1990). Serpentinization occurred via pre- and post-obduction hydrothermal-fluid alteration, followed by a carbonation event resulting from CO_2-rich fluids and forming listwanite (Hansen et al., 2005).

To the east of Atlin are two large playas described as the northern playa and the southern playa. The playas formed via shallow groundwater flow that weathered the rocks in the subsurface, leaching magnesium (Mg^{2+}) from the serpentinite and then bicarbonate (HCO_3^-) from the listwanite, and forming hydromagnesite-magnesite deposits in topographic lows carved by the last glaciation (Power et al., 2014, 2019). Power et al. (2014) proposed a depositional model supported by radiocarbon dates, suggesting that deposition in the playas has been continuous since the retreat of the last glaciation (11 000 BP). Above the glacial till and glaciolacustrine sediments, a layer of calcium-magnesium

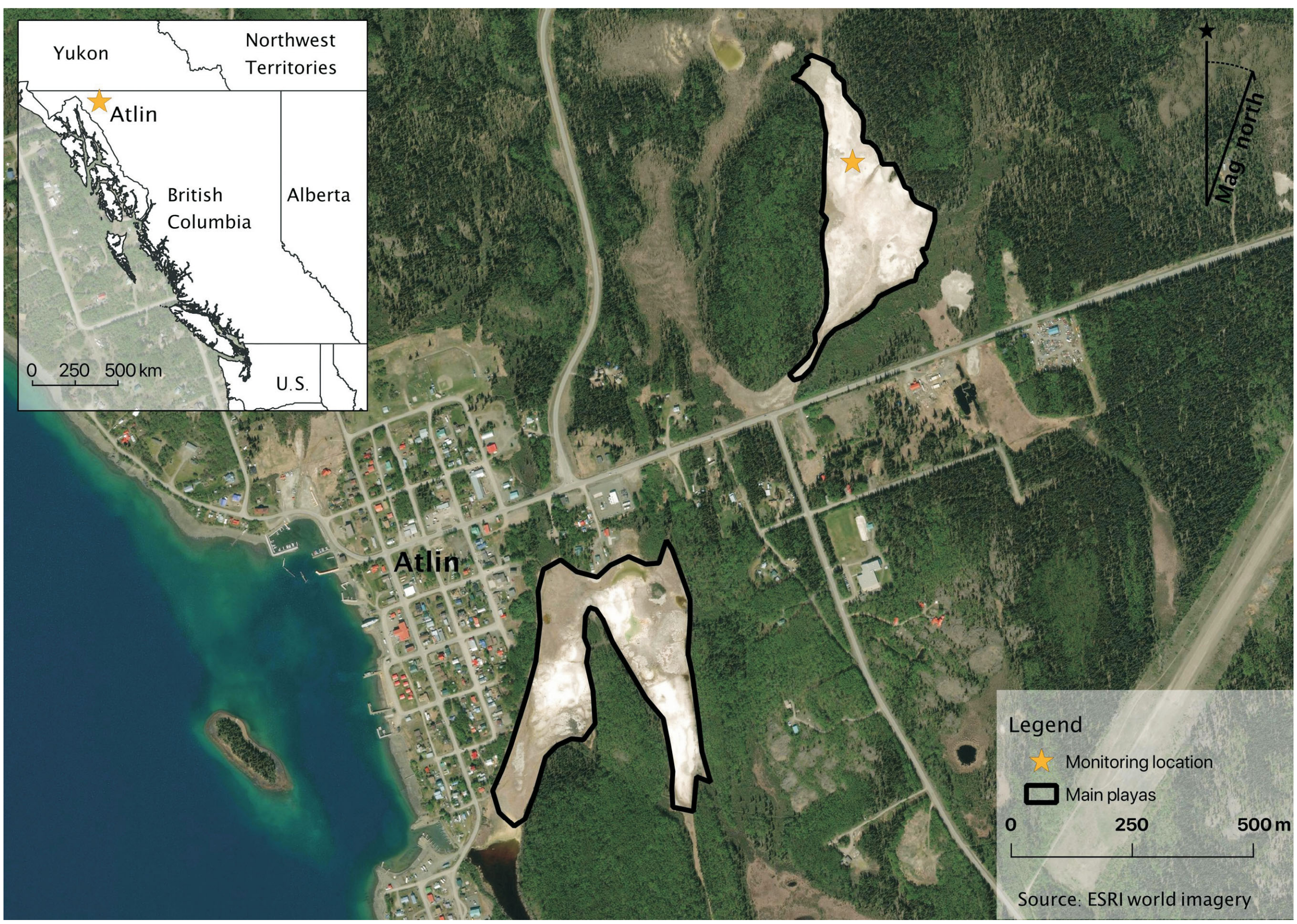

Figure 1. Location of study area in Atlin, northwestern British Columbia (map generated using QGIS V3.14, see http://qgis.org).

carbonate was deposited in subaqueous wetland fed by the alkaline groundwater (pH ~8.6). At the surface, a layer of magnesium carbonate minerals was formed and expanded in subaerial conditions due to evapoconcentration and degassing processes (Power et al., 2014). The southern playa displays four of the depositional environments (i.e., wetland, grassland, localized mounds and amalgamated mounds) described in Power et al. (2014) and the northern playa primarily displays amalgamated mounds at the surface. During the formation of the hydromagnesite-magnesite mounds, the groundwater reaching atmospheric conditions degassed CO_2 and precipitated carbonate minerals, specifically $\{Mg_5(CO_3)_4(OH)_2\ 4H_2O_{[s]}\}$, to maintain thermodynamic equilibrium (Power et al., 2014, 2019).

Site Description

The northern playa was selected for long-term monitoring of CO_2 fluxes (Figure 1). This location for pairing the EC and DCC systems was chosen due to the large areal extent of hydromagnesite-magnesite playa, the reduced surface roughness, as well as the absence of grassland influence. Data were collected over 27 days during August 2020. Long-term DCC were set in pairs at 5 m intervals toward the south of the EC tower (Figures 2, 3) surrounded by a grid of 22 soil collars for survey DCC measurements. Desiccation cracks are common at the surface of the northern playa, extending vertically into the hydromagnesite deposit.

The average air temperature for the month of August, 14°C during daytime and 6 °C at nighttime, was comparable to the multiyear averages, 18 °C and 5 °C, respectively, for the month of August (Environment Canada, 2020). Over the monitoring period, the nighttime temperatures decreased steadily. The rainfall measured by the rain gauge on the EC tower was 38 mm over this period, consistent with the historical average for August (32 ±15 mm). Historical wind direction was considered for establishing the EC tower in the best location to capture the desired fetch (i.e., monitoring area of interest). The historical prevailing-wind direction in August was from the southwest. However, during the monitoring period around 50% of the wind was from the southeast. Since the fetch was homogeneous at 180 degrees east

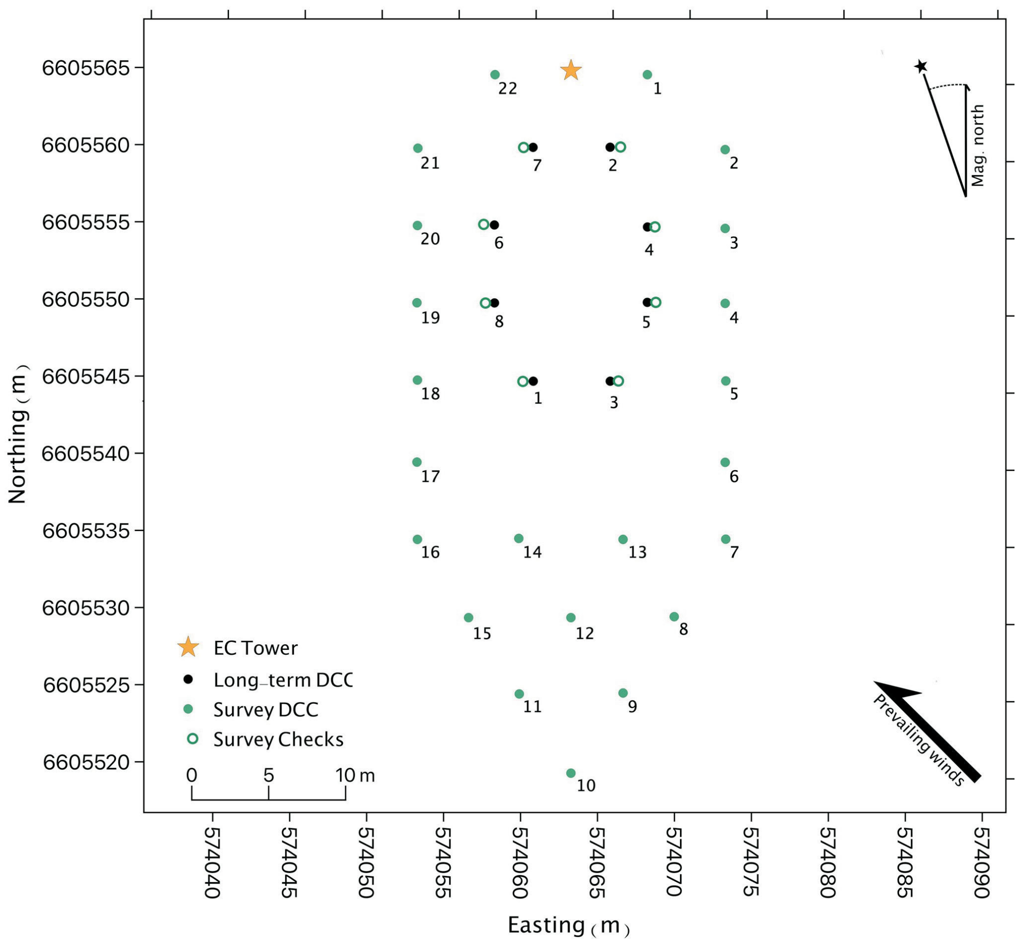

Figure 2. Location of the EC tower relative to the long-term DCC pair system and the DCC survey grid (prevailing-wind arrow for August 2020) in Atlin, northwestern British Columbia. Note that survey DCC co-located with the long-term DCC were used as checks to ground-truth the flux data (grid generated using QGIS V3.14, see http://qgis.org). All co-ordinates are in UTM Zone 8N, NAD 83.

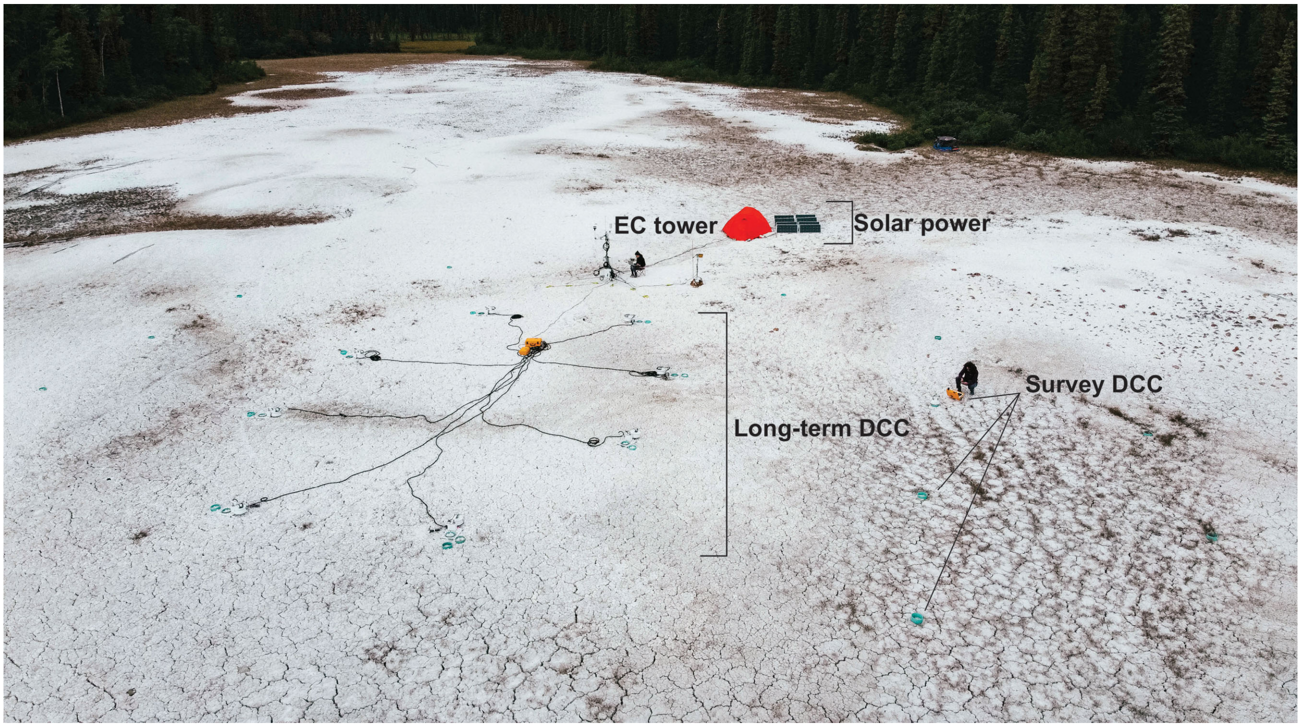

Figure 3. Drone image of the research site facing true north, showing the EC tower near the centre and the long-term DCC system to the left (two researchers for scale) on the northern playa in Atlin, northwestern British Columbia.

to west in the south direction, it was possible to collect valid EC data regardless of the historical wind direction and the choice of tower location.

Materials and Methods

Eddy Covariance

The principles of EC rely on the turbulence created by ground surface roughness, which forms eddies that transport gases, heat and momentum between the Earth's surface and the atmosphere (Baldocchi, 2003). The EC system consisted of a CSAT3B 3-D sonic anemometer manufactured by Campbell Scientific, Inc. (CSI) and a LI-COR Biosciences LI-7200RS enclosed-path CO_2/H_2O gas analyzer (Figure 4). A 7200-101 flow module was installed to maintain a precise air flow through the gas analyzer. Additionally, an analyzer interface unit was installed to integrate the data from the enclosed gas analyzer and the sonic anemometer, then the data was stored and made accessible through the unit's SMARTFlux system. The instruments were set on a tower at a height of 2.1 m above the soil with high-frequency data continuously sampled at 10 Hz for the first two weeks. For the last two weeks, the tower height was lowered to 1.65 m and the sampling frequency increased to 20 Hz to better align the EC-system sampling footprint with the area covered by DCC measurements. The footprint used to compute the area-normalized flux was proportional to the height of the instrument and inversely proportional to the wind speed, among other minor variables. It was critical that the footprint of the instrumentation not extend beyond the fetch, as it would contribute unwanted fluxes to the final estimate.

The flux from an area of interest (i.e., the footprint) was computed from the covariance using Reynolds averaging of vertical wind speed, scalar CO_2 concentrations in dry air and air density outlined in Baldocchi (2003) using the Webb-Pearman-Leuning correction (Webb et al., 1980) given by the following equation:

$$F_{CO_2} = \rho_a \overline{w'CO_2'}$$

where ρ_a is the dry-air density, $w'CO_2'$ is the time-averaged vector-path covariance of the vertical wind speed and the scalar CO_2 concentration in dry air.

The open-source EddyPro® software, released by LI-COR Biosciences, was used to process the flux data. The high-frequency data were averaged over 30 min intervals. Fluxes computed with a friction velocity (u*) less than 0.15 were rejected from this dataset, the low values resulting from the wind-speed being too low to create eddies and transport gases near the surface. Data collected from a wind direction between 270 to 90 degrees from the direction behind the anemometer (at 180 degrees) were rejected. Additionally, a water-vapour correction (Webb et al., 1980) and high-frequency corrections were applied in EddyPro®.

Additional soil and meteorological instruments at the EC station included two soil heat-flux plates (Hukseflux thermal sensor models HFP01SC and HFP01) at 5 cm depth and soil moisture probes (CSI model CS650) installed at an average depth of 10 cm, as well as sensors to measure wind speed and direction (CSI Windsonic4-L), air temperature and relative humidity (CSI model HMP 155A), barometric pressure (CSI model CS106) and rainfall (CSI model TE525WS-L). Energy balances were also calculated using incoming and outgoing radiation (CSI CNR4-L radiometer). Soil and meteorological data were averaged over 30 min intervals from 1 s measurements and were stored on a CSI CR1000X datalogger. All these components were used to measure the weather patterns and ground-truth the EC anemometer data.

Routine tests and diagnostic checks were conducted twice daily to ensure the proper functioning of the components on the flux tower. Processing, data filtering and corrections are ongoing.

Dynamic Closed Chambers

The dynamic closed chambers (DCC) in long-term and survey modes (using LI-COR Biosciences system models LI8100A with the LI-8150 and LI-8100A , respectively) were used to measure the flux from the change in concentration of CO_2 and water vapour ($H_2O_{[g]}$) in the chamber headspace over time. Eight dynamic long-term DCC were set in a grid in the direction of the prevailing winds south of the EC tower, as shown in Figures 2 and 3. The long-term chamber system consisted of eight automatic opaque chamber domes on rectangular platforms, an infrared gas analyzer unit (IRGA) and a LI-8150 multiplexer (Figure 5a). Each chamber recorded observations every 30 min, providing time-series data for fluxes directly across the ground surface. Additionally, a survey grid was set around the long-term DCC consisting of 22 soil collars permanently embedded in the ground, as shown in Figure 2. There were also 12 soil collars emplaced at selected locations, including on the forest floor, in the transition zone between grassland and carbonate materials and in excavations with a depth of 20 cm. A total of 25 observations were made at each survey collar in the grid and a further 12 at the strategically located survey collars. The survey system consisted of a portable opaque chamber dome, polyvinyl chloride (PVC) collars and an IRGA unit (Figure 5b).

The automated chamber, controlled by the analyzer unit, was placed on a collar embedded in the ground, temporarily creating a closed environment to measure accumulation of CO_2 over time in the chamber headspace. Air was circulated between the chamber and the IRGA with an internal pump, creating small-scale eddies to ensure well-mixed conditions in the system. The IRGA measured the concentration of CO_2 and $H_2O_{(g)}$ over time and subsequently corrected the concentration of CO_2 for the influence of $H_2O_{(g)}$ (i.e., dry-air CO_2 or mixing ratio). Additionally, the chamber was vented to the atmosphere to prevent a pressure gradient from affecting the flux measurement.

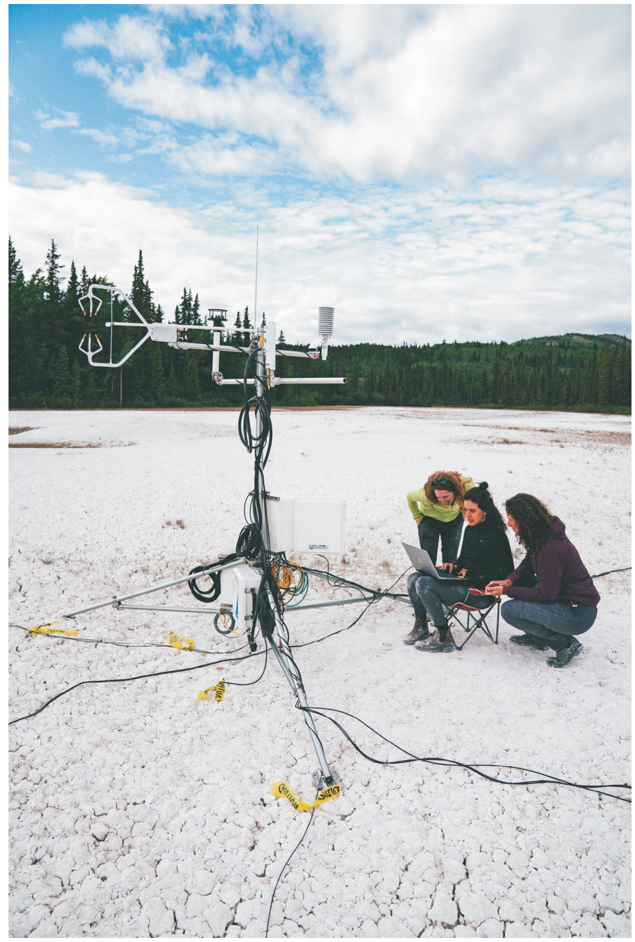

Figure 4. Eddy covariance flux tower in Atlin, northwestern British Columbia (radiometer, rain gauge and soil heat-flux plates are not shown).

Based on qualitative-data analysis, the length of each observation was 120 s during the period of chamber closure. The linear-regression method was used to fit the data points between 5 and 45 s to obtain the rate of change of water-corrected CO_2 over time (see Figure 6). To compute CO_2 fluxes across the ground surface, the temporal CO_2 gradient was then substituted into the following equation:

$$F_{CO_2} = \frac{10VP_0(1-\frac{W_0}{1000})}{RS(T_0-273.15)}\frac{\partial C'}{\partial t}$$

where, F_{CO2} is the gas flux (µmol/m^2/s), P_0 is the initial pressure measured inside the chamber (kPa), W_0 is the initial water-vapour mole fraction (mmol/mol), $\delta C'/\delta t$ is the rate of change in the water-corrected CO_2 mole fraction (µmol/mol/s), S is the soil surface area over which the flux occurs (cm^2), V is the total volume of the system (the sum of the volume of the chamber headspace, the volume of the

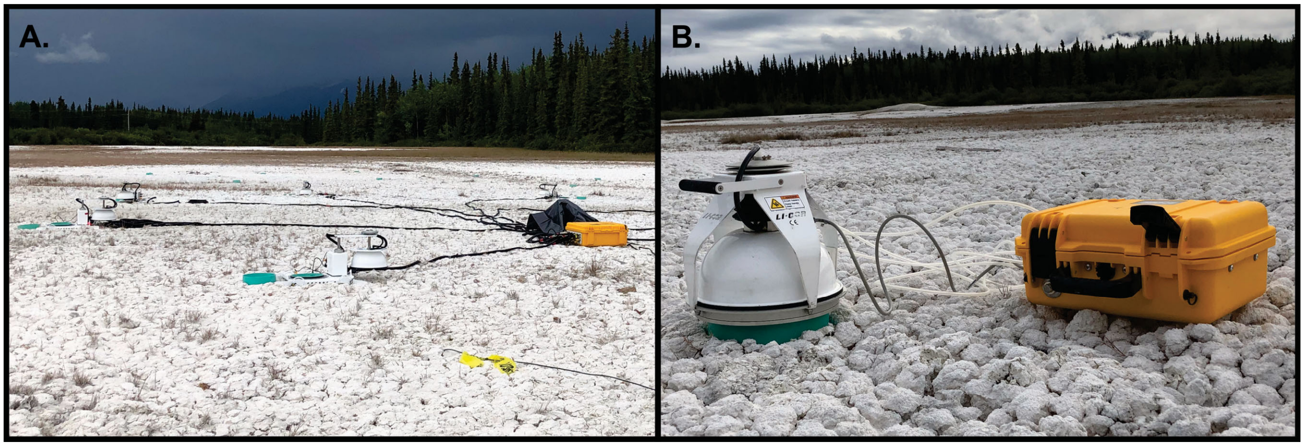

Figure 5. Study area in Atlin, northwestern British Columbia, showing **a)** a long-term chamber system, and **b)** a survey chamber system with infrared gas analyzer unit. Both are used to make gas-flux measurements and can be seen here with their associated soil collars (constructed from green polyvinyl chloride pipe sections).

collar above the ground surface and associated connection tubing and the internal analyzer volume in cm^3), R is the ideal gas constant (8.314 kPa·m^3/K/mol) and T_0 is the initial air temperature (°C).

Additionally, data and diagnostic checks were conducted five times during the observation period to ensure the proper functioning of both chamber modes. On four separate days, survey-mode observations were made alongside the long-term chambers as checks, to ground-truth the fluxes and assess the heterogeneity of the sampling. Processing, data filtering and corrections are ongoing.

Gas Sampling for Isotopic and Radiocarbon Analysis

The source of CO_2 from the subsurface and air can be distinguished based on their stable isotopes and radiocarbon composition. Carbon-dioxide gas composition will be determined via stable isotopes of carbon and oxygen, and age will be estimated using radiocarbon analysis. Following recommendations, a static chamber method was chosen for gas-sample collection and sampling from the playas (A. Wozney, I.D. Clark and K.U. Mayer, work in progress). The static chambers, consisting of a section of PVC pipe 10 cm across and 30 cm in length, were embedded 20 cm into the carbonate soil and fitted with a PVC cap equipped with a septum for sample extraction. To gain insight on the discrete and spatial variation in gas composition at depth, the static chambers were placed in excavations dug to three depths (5 cm, 40 cm and 65 cm below ground surface) at two locations on the northern playa. The static chambers were left undisturbed for a week before initial sampling.

Gas was collected from the septum with a 25 mL gas-tight syringe and 20 mL of sample was inserted into a 12 mL Labco Limited pre-evacuated Exetainer® to be analyzed for stable isotopes of CO_2, C^{13} and O^{18}. Samples for radiocarbon required a large volume of pore gas and air due to low CO_2 concentrations, ranging approximately from 400 to 1000 ppm. The method used for ^{14}C sample collection was

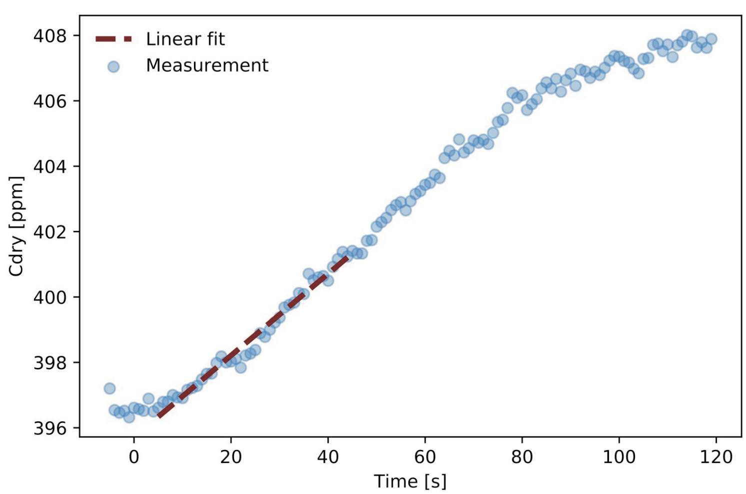

Figure 6. Determination over time of the rate of change in dry-air CO_2 (Cdry) with a linear fit (in red) for CO_2-flux calculation.

recently developed at the A.E Lalonde AMS Laboratory (Ottawa, ON). Using this method, the gas sample is bubbled through a 0.4 mL solution of barium hydroxide {$Ba(OH)_2$}, leading to the sequestration of CO_2 as a barium carbonate precipitate (Reynolds, 2019). Twenty-five samples for stable-isotope analysis were taken over three days and nine (six static chamber and three atmospheric) samples were collected for radiocarbon analysis.

Work in Progress and Expected Outcomes

Data analysis is in progress to compare the CO_2 fluxes from the EC and DCC methods. A correlation analysis of the EC and DCC data will be conducted. Additional weather correlation plots will be prepared to observe any climatic phenomena influencing the fluxes. Finally, the associated errors and limitations of both methods will be evaluated. For example, the desiccation cracks at the surface of the playas are expected to add a level of spatial variability to the DCC method. The EC method is well suited to quantify average fluxes over an area with desiccation cracks; however, the DCC method requires the headspace be closed to the atmosphere to record a valid observation. Cracks and leaks around the collars during installation were kept to a minimum in this study, but a careful inspection of the data will be needed to assess potential impact on data quality and interpretation due to the presence of cracks. The desiccation-crack problem and other potential limitations can be transposed to mine tailings with mudcracks; therefore, insights from the outcomes of this pilot study will be reflected in the application of this method to mine sites. The deployment of the technologies in the field and a preliminary review of the data suggest that these methods show promise for application at an active mine site where the ore is hosted in ultramafic rock to measure real-time CO_2 sequestration in mine tailings.

Carbon dioxide stable isotopes and radiocarbon in CO_2 from pore and atmospheric gas samples are currently being analyzed. These results will provide a better understanding of the origin of the CO_2 given the age and composition of the gas. If the CO_2 is from a deep geological source, there is an option to further study the Atlin playas as a natural analogue for scenarios for the leakage of CO_2 following injection and, also, to develop a better understanding of these unique geological features. Studying the mechanism and kinetics of carbonate precipitation as a mean for scrubbing the CO_2 as it is emitted at this site could be warranted.

Conclusions

This study on the hydromagnesite-magnesite playas in Atlin, BC has shown that EC and DCC methods show promise as a means of measuring CO_2 fluxes in a geological environment to characterize CO_2 exchange with the atmosphere. As suggested in the literature, passive CO_2 fluxes in mine tailings containing ultramafic minerals are significant and can substantially impact the carbon budget of the mine facility. The eddy covariance method may provide an efficient and continuous real-time approach to quantifying rates of CO_2 capture. The dynamic closed-chamber method is useful in cross-validating fluxes from EC measurements to corroborate the validity of the findings. The overarching goal of this project was to inform verification protocols for use by mining companies to secure carbon credits, not only in BC but also in other localities in the world where ultramafic deposits have the potential to passively capture atmospheric CO_2. These protocols would need to be recognized by the provincial and federal governments and, eventually, these environmental monitoring technologies would aid mine operators in proving reductions in their carbon footprint, which in turn could be traded for carbon credits and to market low-carbon metals at a premium.

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