

A novel empirical approach to measuring pore gas compositional change in mine waste storage facilities: A case study from northern Europe

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Abstract

The transition to a net-zero economy will place increased demand upon global mine production of key resources, increasing waste generation and life-of-mine (LOM) greenhouse gas emissions. The increased waste generation accompanying this transition will increase the need for sustainable waste management/closure. Pore gas compositions in waste rock facilities are a key control factor on sulfide oxidation and subsequent acid rock drainage (ARD) onset. Understanding pore gas changes in waste facilities post closure is essential to understand long term management risks and feasibility of closure engineering strategies (e.g. covers) to meet long term closure goals. To date no standardised empirical testing method exists to estimate the relative change in pore gases (e.g. O₂ and CO₂), and long term flux rates of these gases within waste facilities once encapsulation of waste has been undertaken. Silicate weathering and carbonate precipitation provide a key source of alkalinity in low carbonate waste materials and are a key control on acid generation potential. These silicate minerals also provide a potential feedstock for both active and passive carbon capture, through enhanced weathering/mineral carbonation. Previous studies have recorded evidence of both CO₂ release and carbon sequestration within waste rock and tailings of several operating and closed sites across the globe, yet the concept remains underdeveloped with regards to policy and standardisation. Understanding the relative rates of sulfide oxidation, silicate weathering, carbonate buffering and pore gas/water geochemical interaction is key in predicting long term pore gas changes in waste rock facilities.

This study presents a method of measuring the relative balance between sulfide oxidation, carbonate buffering and silicate mineral carbonation rates of mine wastes using bespoke sealed experimental cells partially filled with sulfidic ultramafic mine waste. Fitted high-accuracy probes within the air space of cells track CO₂ and O₂ flux, with changes being indicative of processes including carbon sequestration and sulfide oxidation/carbonate buffering. Reported relative gas concentrations are converted to absolute values using known variables in conjunction with the ideal gas law, allowing for estimates to be made regarding CO₂ and O₂ uptake/release over a given logging period. Variables such as waste type, moisture content, temperature and starting gas composition are modified to simulate various site conditions. This study aims to outline the workings of the measurement method along with displaying its potential as a tool to assess post closure pore gas changes in waste storage facilities.

Keywords: *gas flux, silicate weathering, carbonate precipitation, sulfide oxidation, pore gas*

1 Introduction

1.1 Study rationale

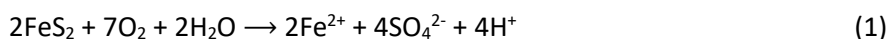
Acid rock drainage (ARD), produced via the oxidation of sulfides, represents a major challenge to waste management within the mining sector. The typically low pH (<6) and high dissolved metal load of ARD mean additive treatment is often both technically complex and expensive (Jiao et al. 2023; Nyström et al. 2019); instead, strategies to inhibit generation (preventative methods) are increasingly preferred (Vaziri et al. 2021).

As oxygen (O₂) supply is the limiting factor regarding ARD generation at most sites, one approach to minimising generation involves reducing the atmospheric exposure of sulfidic mine wastes (Anekwe & Isa 2023). The application of cover/capping systems (ranging from dry, wet and organic systems) at waste storage facilities is a common closure engineering strategy that aims to prevent O₂ ingress into wastes (Skousen et al 2017). Measuring of pore-gas concentration within wastes has previously been used as a parameter assess the effectiveness of cover systems, although O₂ has been the focus of intrusive on-site investigations (Harries & Ritchie 1985; Ramasamy & Power 2019). Site-specific variability (e.g., mineralogy and climate) and the inter-play of O₂ and carbon dioxide (CO₂) during relevant reactions (sulfide oxidation, silicate and carbonate weathering) mean the development of a standardised empirical measurement method of pore-gas change would be of benefit to the mining industry.

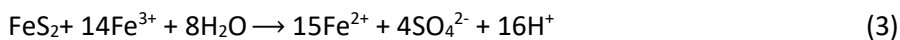
1.2 Role of pore-gas in ARD generation and associated reactions

1.2.1 Sulfide oxidation

Whilst covers can limit O₂ ingress into wastes from the atmosphere, existing O₂ present within the pore spaces of waste material can continue to drive oxidation of sulfides. Upon contact with interstitial O₂ and pore-water (H₂O), sulfide minerals can oxidise to produce hydrogen ions (H⁺), sulfate ions (SO₄²⁻) and ferrous iron (Fe²⁺). The acidity generated by this reaction can lead to the leaching of additional metals into solution. Equation 1 (Tomiyaama & Igarashi 2022) displays the oxidation of pyrite (FeS₂), a sulfide mineral considered to be the principal cause of ARD in most cases (Yuan et al. 2022).



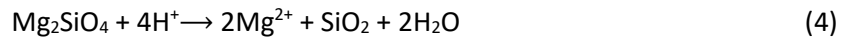
The oxidation of pyrite can be further driven by the presence of ferric ion (Fe³⁺) (itself generated by the oxidation of Fe²⁺). These reactions are shown in Equations 2 and 3 (Tomiyaama & Igarashi 2022).



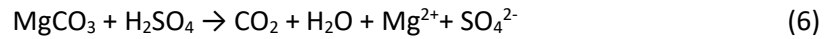
1.2.2 Importance of silicates and carbonates

In addition to sulfides, silicate minerals and primary carbonate minerals are common constituents of waste material (Lindsay et al. 2015). The dissolution of silicates and carbonates consumes H⁺ ions, providing acid-buffering/neutralisation capacity within waste materials (Brough et al. 2013).

Furthermore, CO₂ flux is a key feature of silicate and carbonate reactions within wastes (Ross et al. 2018). The dissolution of CO₂ within the pore-water of wastes forms carbonic acid (H₂CO₃) which dissociates into bicarbonate (HCO₃⁻) and carbonate (CO₃²⁻) ions. The dissolution of silicate minerals (particularly Mg and Ca-bearing silicates) under acidic conditions can operate as a CO₂ sink, with released metal cations reacting with HCO₃⁻ and/or CO₃²⁻ to form precipitable secondary carbonates. This is shown in Equations 4 and 5 (Wang and Giammar, 2012) using forsterite (Mg₂SiO₄) as an example.



Conversely, the dissolution of primary and secondary carbonates (for example magnesite- MgCO_3) within wastes leads to the release of CO_2 , as shown in Equation 6 (Torres et al. 2014).



2 Methodology

2.1 Research design

Measuring CO_2 and O_2 flux from waste samples was identified as a viable method of measuring the rates of the reactions explored above. As a basis to simulate the reactions occurring within covered waste material under varying conditions, a bespoke experimental sealed cell was designed and developed. It was planned to partially fill multiple cells with mine wastes and install CO_2 and O_2 probes within the available air space of cells to log gas flux over time (Figure 1). The use of different mine waste types and different starting gas concentrations in cells was planned to allow for comparability of results. Stainless steel barrels were chosen as the building blocks of the planned experimental cells. Purchasing and modifying standard barrels avoided the additional costs of custom-made vessels.

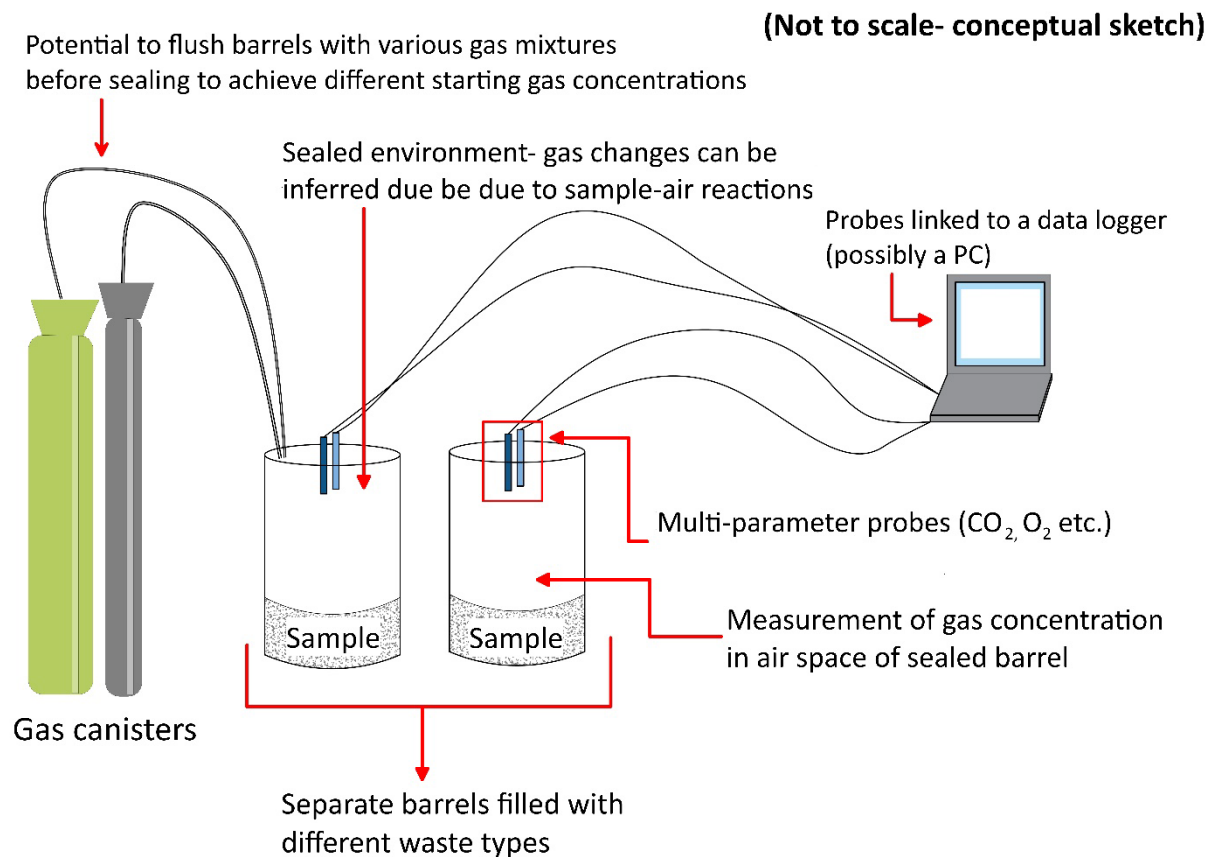


Figure 1 Conceptual sketch displaying the initial planned experimental cell set-up

2.2 Experimental set-up

2.2.1 Sample preparation

The waste samples chosen to be utilised within the experimental cells included waste rock and tailings from a nickel-copper-platinum group element mine in northern Europe. Previous in-house characterisation on material from this site had shown the typical mineralogy of waste rock and tailings to be similar- being dominated by the silicates tremolite ($\text{Ca}_2\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2$) (an amphibole); diopside ($\text{Ca}_2\text{Si}_2\text{O}_6$) (a clinopyroxene); enstatite (MgSiO_3) (an orthopyroxene) and forsterite (Mg_2SiO_4) (an olivine). The dominant sulfide mineral present was pyrrhotite (Fe_{1-x}S) (where x can range from 0-0.125) with pentlandite ($(\text{Fe,Ni})_9\text{S}_8$) and chalcopyrite (CuFeS_2) present at lower concentrations. Dolomite ($\text{CaMg}(\text{CO}_3)_2$) and calcite (CaCO_3) were the most significant carbonates present.

Waste rock samples were sourced from a waste rock inventory located at Geochemic Ltd., Pontypool, United Kingdom. This material was originally obtained from a drilling project at the mine site during 2017. Creating a composite of various waste rock samples was determined to be most appropriate as the masses of single samples were insufficient to appropriately fill the planned cells. Samples were first sieved to remove existing weathered material before being crushed to <6.3mm and mixed. This procedure was performed to maximise reactivity over the planned logging period.

Tailings samples arrived directly from site in three separate ~20 kg shipments and were processed at Geochemic Ltd. All tailings samples were wet and were mixed to ensure homogenisation before being coned and quartered.

2.2.2 Cell construction

Stainless steel barrels were sourced to act as the main body of each cell. Stainless steel was chosen over plastics such as high-density polyethylene (HDPE) due to its lower gas permeability (Stern and Fried, 2007), minimising the potential of gas flux in/out of the closed system. The resistance of stainless steel to acidic corrosion (Chen et al., 2019) was also important due to the predicted generation of acidic conditions. Based on the volume of waste material available, two larger (110 litre) barrels were planned to be filled with for the waste rock composite material and five smaller (60 litre) barrels for the tailings.

To measure CO_2 , the non-dispersive infrared (NDIR) GMP-251 (%-level) and GMP-252 (ppm-level) probes from Vaisala® were chosen. To measure O_2 , the SO-421 SDI-12 galvanic cell oxygen probe by Apogee® was chosen. Once the probes were received (7 SO-421, 8 GMP-251, 9 GMP-252) they were connected to a laptop using Universal Serial Bus (USB) adapters.

Three probes (one GMP-251, one GMP-252 and one SO-421) were inserted into each barrel to provide CO_2 readings (a high-threshold and low-threshold reading) and O_2 readings. To fit the probes and associated cables, the larger rubber bung of each barrel was removed and fitted with a threaded plastic hex plug. This hex plug was drilled with holes to accommodate the three probe cables, two input/output steel pipes and a supporting central threaded bolt. Once the probes and pipes were fed through the barrel lids, the plug was filled with epoxy resin and probes were bound with cable ties to keep all components in place (Figures 2 and 3).

Temperature control was important to simulate field conditions. To maintain a constant temperature, it was decided to place the barrels in a cold room unit. A value of 8 °C (an approximate median value of recorded temperature extremes at the mine site) was chosen. The probes of assembled barrels were connected to external multi-port USB hubs which were used to connect to two laptops (the data loggers). An additional CO_2 probe (GMP-252) was fitted at the corner of the cold room to measure ambient CO_2 levels outside the barrels.

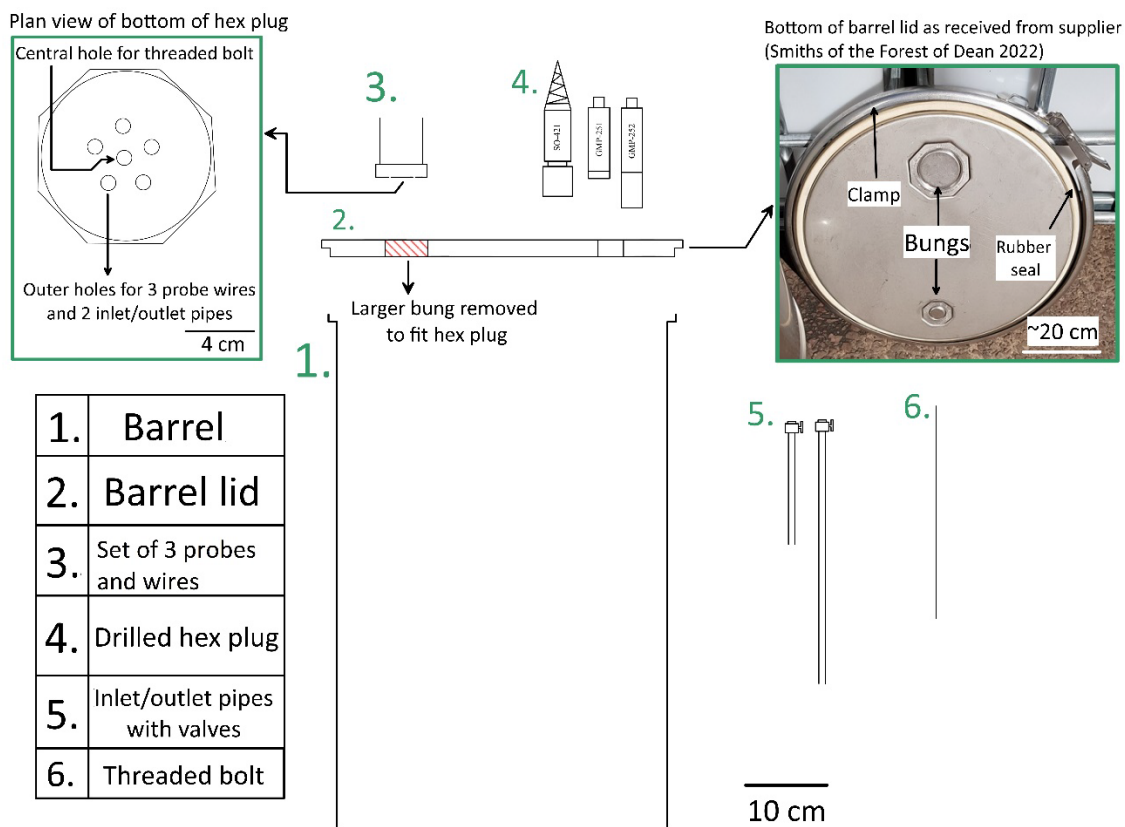


Figure 2 Key components of the barrel set-up drawn to scale

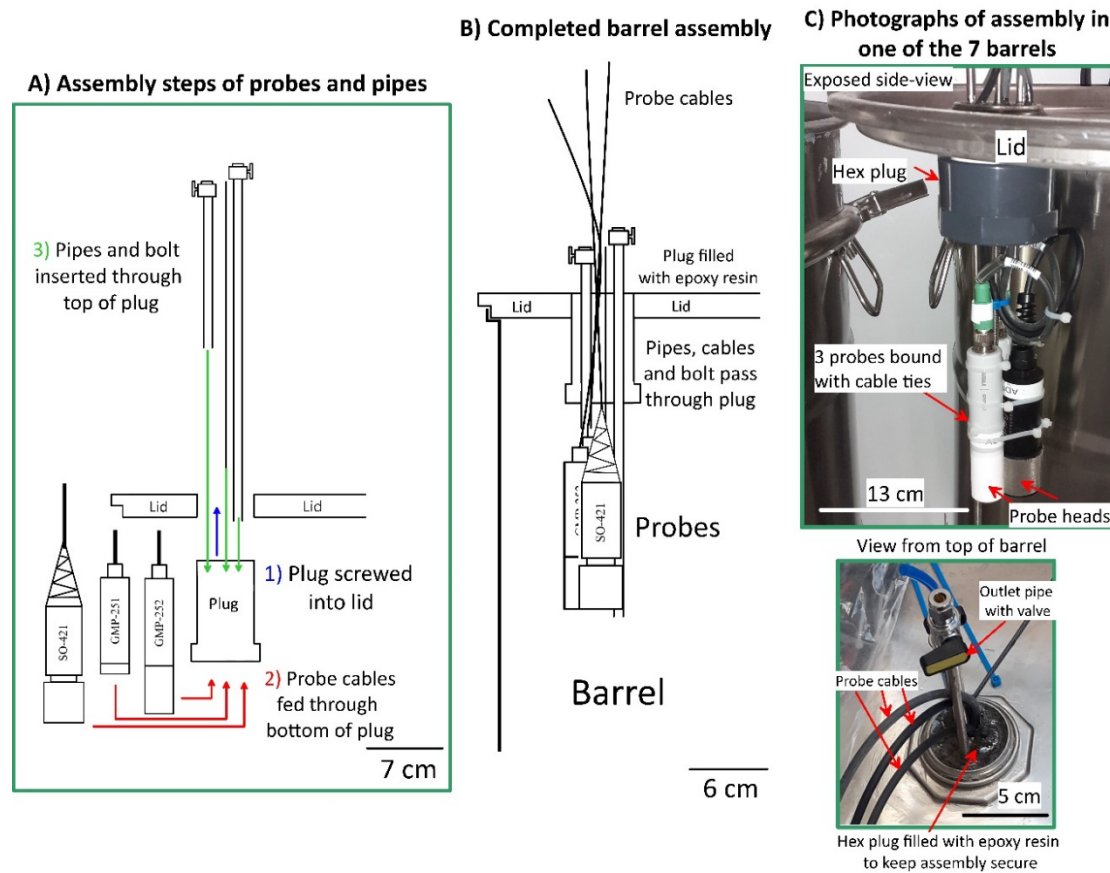


Figure 3 Example of an assembled barrel

2.2.3 Cell filling and gas flushing

The barrels were filled as described in Table 1 below. The smaller 60 litre barrels were all filled with equal masses of tailings composite. These tailings were used as received from site (with a moisture content of ~15%) to ensure they were representative. To assess the impact of elevated levels of CO₂ (and by extension, negligible O₂) on chemical reactions, two of these barrels were chosen to be flushed with a 10% CO₂ in nitrogen (N₂) mixture before sealing, whilst two additional barrels were flushed with compressed air to achieve approximate atmospheric CO₂ and O₂ concentrations. Inert quartz sand was used as a control for these tailings cells. As the waste rock composite was dry, deionised water was added once the sample was loaded to achieve a moisture content of 7.5%, simulating field conditions as closely as possible. A control cell was created using inert quartz chips and an identical water content used (though it should be noted that the mass of quartz chip used was lower than the waste rock due to limited supply). Quartz chips were used in place of sand as they were more representative of the grain size of the waste rock composite. Both barrels were flushed with compressed air before sealing to achieve atmospheric gas concentrations. Diaphragm pressures gauges were fitted to each barrel to show pressure change over time. Probe logging was started following sealing.

Table 1 Description of cell contents and starting gas composition

Barrel number	Cell contents	Mass (kg)	Starting gas composition
1	Tailings composite	4.98	Elevated CO ₂
2	Tailings composite	4.98	Elevated CO ₂
3	Tailings composite	4.98	Atmospheric
4	Tailings composite	4.98	Atmospheric
5	Quartz sand (control)	4.98	Atmospheric
6	Waste rock composite	50.2	Atmospheric
7	Quartz chip (control)	14.64	Atmospheric

2.2.4 Expected results

The change in CO₂ and O₂ concentrations the air space of various cells was expected to differ throughout the logging period. An initial rise in CO₂ concentration, coupled with an O₂ decrease, was expected in those with atmospheric gas composition due to the oxidation of sulfides leading to acidity-induced carbonate dissolution (Stokreef et al. 2022). This was hypothesised to occur at a more rapid rate within the tailings than the waste rock due to a finer particle size and therefore a higher specific surface area to accommodate reactions (Opara et al. 2022). The CO₂ within the cells containing elevated starting CO₂ was expected to decrease, with the negligible O₂ supply leading to the rate of CO₂-sink reactions (silicate dissolution) being dominant over CO₂-source reactions (sulfide oxidation and subsequent carbonate dissolution).

3 Results

3.1 Elevated CO₂ barrels

The GMP-251 probe readings were used for Barrel 1 and 2 as CO₂ concentrations were outside of the range of the GMP-252 at >1%. Starting CO₂ concentrations in Barrel 1 and 2 were 7.81 and 8.23% respectively (Figure 4). CO₂ in both barrels decreased over 60 days, reaching 6.78% in Barrel 1 and 7.20% in Barrel 2 (a reduction of just over 1%). Both barrels showed the sharpest decline in CO₂ within the first 5 days of logging.

Concentrations of O₂ started at near 0 in both barrels (0.8% in Barrel 1 and -0.02% in Barrel 2) (Figure 4). The O₂ in Barrel 1 showed a series of stepped rises and falls resulting in a net O₂ increase of 0.024% over 60 days. The O₂ in Barrel 2 decreased most sharply within the first 5 days of logging, reaching -0.18% after 40 days and remaining steady for the following 20 days. An overall decrease of 0.16% in O₂ concentration was seen.

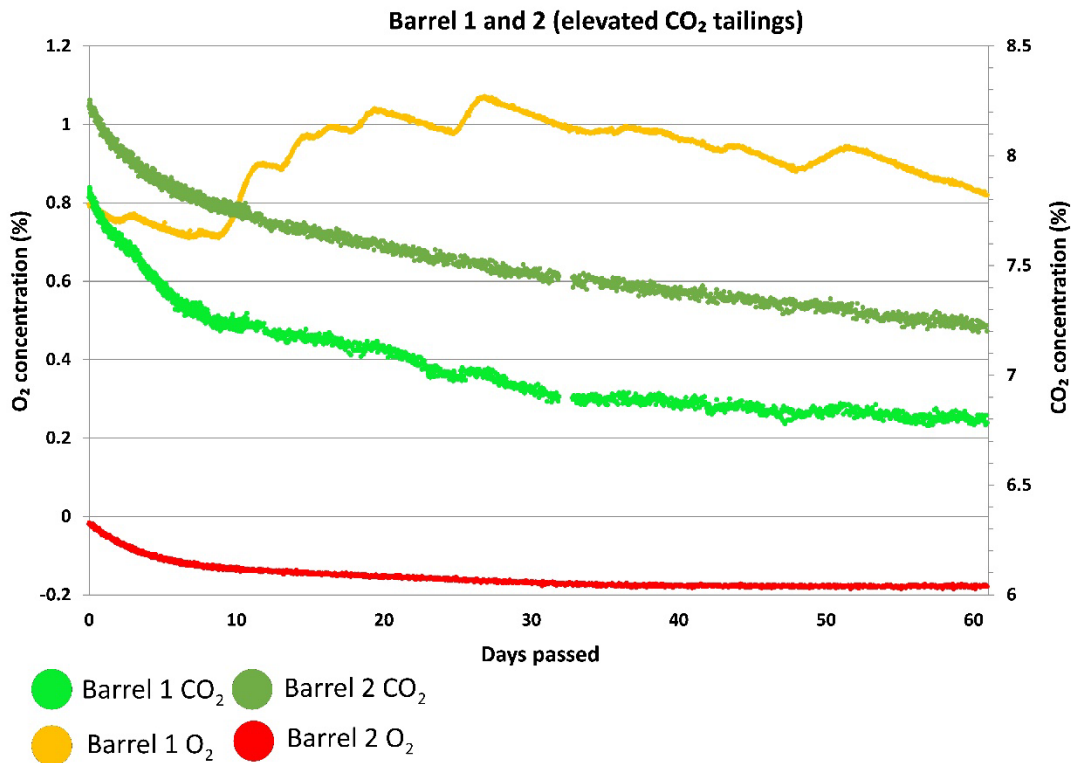


Figure 4 CO₂ and O₂ concentration within Barrels 1 and 2

3.2 Atmospheric tailings barrels

The GMP-252 probe readings were used for Barrel 3, 4 and 5 as CO₂ concentrations were suitably within range (0-10,000 ppm). Barrel 3 had a starting CO₂ concentration of 490 ppm, while starting CO₂ concentrations in Barrels 4 and 5 were identical at 345 ppm (Figure 5). The CO₂ in all barrels (including the control) showed a decrease over 60 days (217 ppm in Barrel 3, 255 ppm in Barrel 4 and 227 ppm in Barrel 5). The sharpest decline in CO₂ occurred within the first 5 days of logging in all barrels and the rate of decrease slowed with time.

Starting concentrations of O₂ were similar in all barrels (20.43% in Barrel 3, 20.53% in Barrel 4 and 20.56% in Barrel 5) (Figure 5). Barrels 3 and 4 showed a decrease in O₂ of 0.38% and 0.56% respectively, with the rate of decrease appearing highest from 40 days onwards. The O₂ concentration in Barrel 5 showed a smaller net decrease of 0.14% and greater fluctuation in O₂ readings.

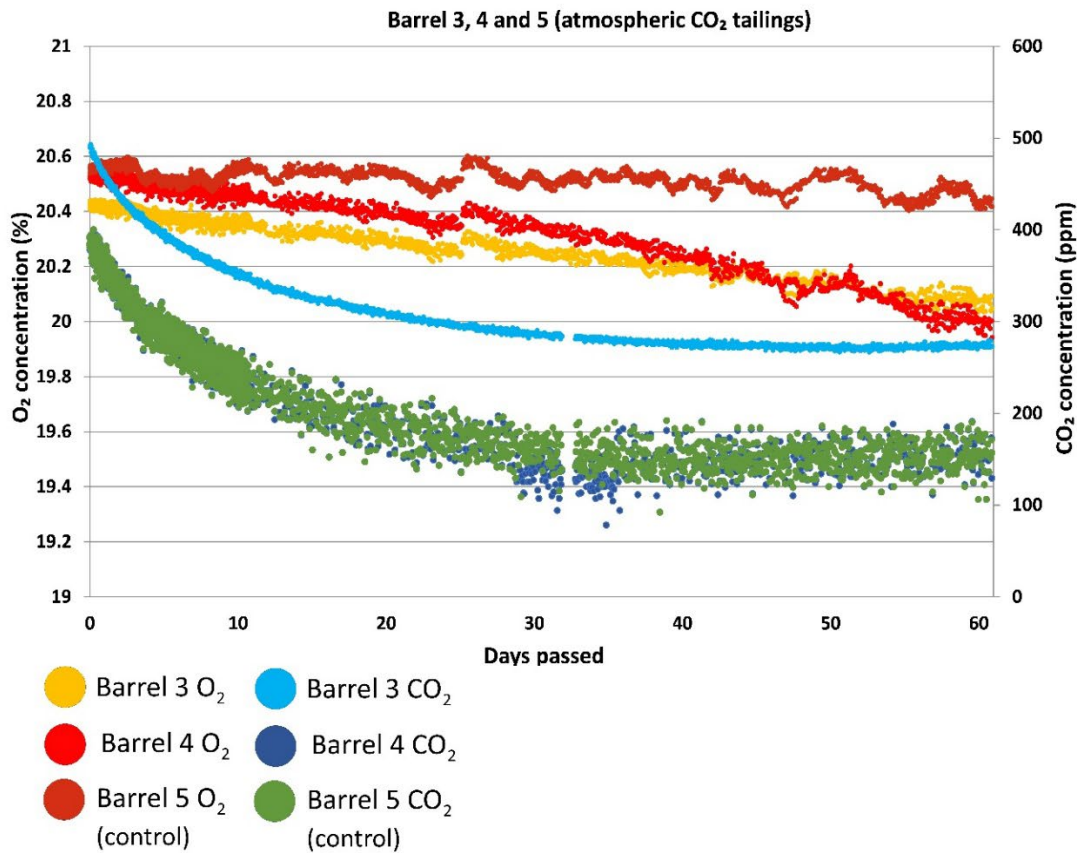


Figure 5 CO₂ and O₂ concentration within Barrels 3, 4 and 5

3.3 Atmospheric waste rock barrel

The GMP-252 probe readings were used for Barrel 6 and 7 as CO₂ concentrations were suitably within range (0-10,000 ppm). Barrel 6 had a starting CO₂ concentration of 419 ppm and Barrel 7 had a higher starting concentration of 511 ppm (Figure 6). The CO₂ change in Barrel 6 was the most significant of all the barrels—showing an initial decrease of 165 ppm after ~5 days of logging before increasing and reaching 6131 ppm over the subsequent 55 days of logging. The CO₂ concentration in Barrel 7 remained steadier than Barrel 6, decreasing by 27 ppm after ~5 days of logging before rising slightly to reach 503 ppm after 60 days.

Starting concentrations of O₂ were similar both barrels (20.16% in Barrel 6 and 20.49% in Barrel 7) (Figure 6). Barrel 6 showed a decrease in O₂ of 8.31% over 60 days, while the O₂ concentration in Barrel 7 decreased by a smaller amount (0.26%).

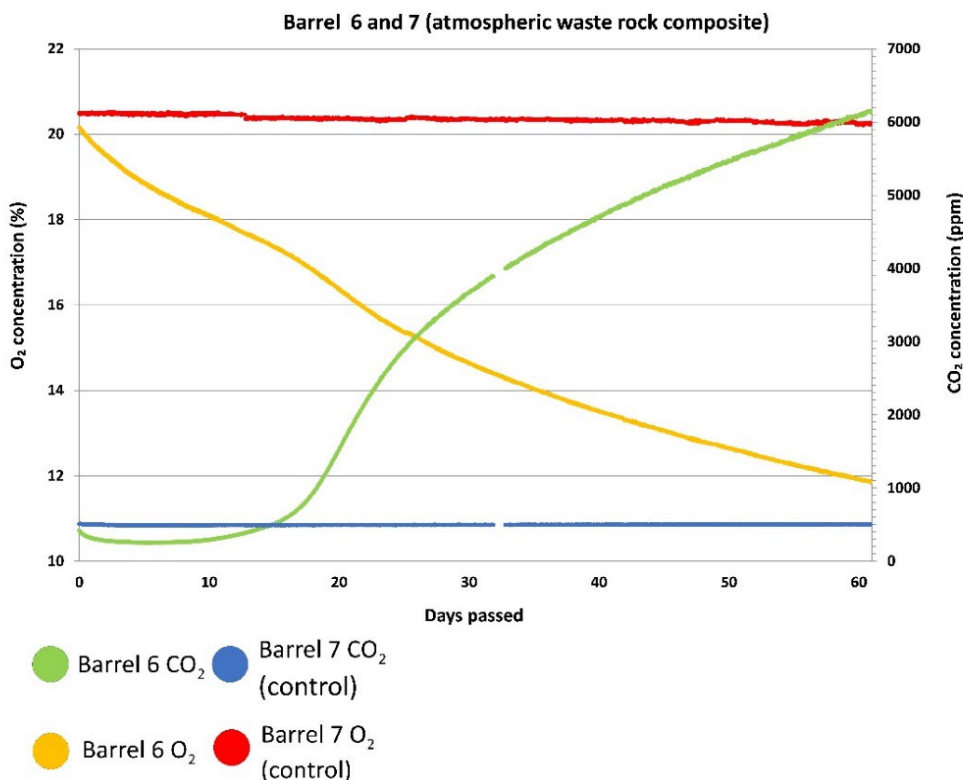


Figure 6 CO₂ and O₂ concentration within Barrels 6 and 7

4 Discussion

4.1 CO₂ and O₂ flux in tailings material

Barrel 1 and 2 showed the largest CO₂ decrease of all barrels tested, with a reduction of 1% CO₂ seen in the barrel air spaces. The lower CO₂ decrease in the corresponding atmospheric tailings barrels (3 and 4) suggests this difference was a result of an increased CO₂ supply increasing the rate of CO₂ uptake (carbonation). Carbonation rates increase with CO₂ supply as more CO₂ is available to dissolve into interstitial water to start the carbonation process (Stokreef et al. 2022). There was a net decrease in CO₂ within both barrels, suggesting CO₂ uptake from carbonation was dominant over CO₂ generation from sulphide-carbonate interaction.

The O₂ concentration in Barrels 1 and 2 did not stay constant at near-zero as expected. The increases and subsequent decreases of O₂ in Barrel 1 are notable. The increases may have been due to entry of O₂ into the barrel. The decrease in O₂ that followed (causing the steps seen in Figure 4) likely illustrates the process of sulphide oxidation and carbonate dissolution driven by an increase in O₂ supply. This is further supported by the fact that the rises and falls in O₂ correspond with minor increases in CO₂, suggesting CO₂ was released by the acidity-induced dissolution of carbonates resulting from sulphide oxidation.

Barrels 3 and 4 showed a CO₂ decrease over the logging period, with a similar CO₂ decrease also shown by the quartz sand in Barrel 5. This decrease was unlikely to have been caused by a leak, since O₂ concentrations in these barrels did not show a steady increase. As the quartz sample was inert (and so not expected to have undergone carbonation reactions), these results suggest that a similar process was occurring in all three barrels. As all samples had similar water contents, this CO₂ decrease could have been due to the diffusion of CO₂ into the interstitial water of mineral grains. Though this process can be the first step in aqueous carbonation (Rackley 2017), diffusion of CO₂ into water is a fundamental physical process that occurs

irrespective of mineralogy (Power et al. 2014). Since CO₂ is highly soluble at the temperature range at which the barrels were kept (Stokreef et al. 2022), the CO₂ uptake in all barrels may have been largely due to CO₂ diffusion into water and not necessarily potential subsequent carbonation reactions.

The O₂ concentration in Barrels 3 and 4 showed a decrease that was not mirrored by the control and is theorised to have been due to sulphide oxidation (sulphides and carbonates were absent in the control). This O₂ decrease did not correspond with an increase in CO₂, suggesting that the CO₂ produced from sulphide oxidation was less than that which was taken up via diffusion into water. The fact that the tailings did not seem to undergo carbonation reactions (in addition to the fact that their rate of oxidation was relatively low) suggests a general lack of reactivity. This contrasts with the expectation that the tailings used in this experiment would be reactive due to their fine particle size and higher specific surface area. This lack of reactivity could be due to diffusion limitations relating to the relatively low mass (4.98 kg) of tailings used and their relatively low permeability (d₁₀= 2.80 µm; d₅₀ = 42.51 µm; d₉₀= 153.70 µm).

4.2 CO₂ and O₂ flux in waste rock composite

The results from the waste rock barrel were indicative of sulphide oxidation of the waste rock sample, leading to O₂ drawdown and subsequent CO₂ release via carbonate dissolution. This theory is supported by the lack of major change observed in the waste rock control barrel which did not contain sulphides or carbonates. Although a net increase in CO₂ in Barrel 6 was observed (the only net CO₂ increase of all the barrels), there was an initial decrease in CO₂ in Barrel 6 during the first 5 days of logging. This decrease may have been due to the diffusion of CO₂ into the interstitial water of the waste rock sample, similarly to the decreases seen in Barrel 3, 4 and 5.

There was a net increase in CO₂ within Barrel 6, suggesting that CO₂ generation from sulphide-carbonate reactions was dominant over CO₂ uptake from carbonation. This contrasted with the tailings samples, which all showed a net CO₂ decrease over the logging period. The rate of O₂ decrease within the waste rock was also far higher than in the atmospheric tailings, suggesting a much higher oxidation rate. The greater mass (50.2 kg) and higher permeability of the waste rock (d₁₀= 0.13 mm; d₅₀ = 2.94 mm; d₉₀= 7.96 mm) when compared with the tailings may explain the difference in reactivity between the two waste types.

4.3 Conversion to absolute gas concentrations

The relative concentrations of CO₂ and O₂ reported by the probes were used in conjunction with the ideal gas law (Equation 7- Francis 2016) and Dalton's Law of partial pressures (Equation 8- Hernández 2016) and known parameters to estimate the change in grams of gas within each barrel over time (Table 2).

$$n = PV/RT \quad (7)$$

Where: n = moles, P= pressure, V= volume, T= temperature and R= relative gas constant

$$P_t = P_1 + P_2 + P_3 \quad (8)$$

Where: P_t = total pressure and P_{1,2,3} = partial pressures of individual gases in a given mixture

Table 2 Net changes of CO₂ and O₂ in barrel air space over the logging period

Barrel number	Net CO ₂ change (g)	Net O ₂ change (g)
1	-0.0009	+0.022
2	-1.37	-0.15
3	-0.029	-0.38
4	-0.034	-0.59
5	-0.030	-0.16
6	+0.99	-13.46
7	-0.0018	-0.43

4.4 Applicability and drawbacks of experimental method

The experimental method developed allowed for the inter-play of CO₂ and O₂ flux within wastes to be mapped. Comparing the waste-filled barrels with the control barrels gives confidence that gas flux is due to reactions such as sulfide oxidation and carbonate dissolution. The known volume of the barrels paired with pressure and temperature measurement make conversion to absolute gas concentrations convenient. The standardised nature of the barrels also means that different types of waste from additional mine sites could easily be incorporated and used to compare the dominance of certain reactions in material of varying mineralogy. Whilst the temperature of the cold room unit was maintained at 8 °C during data collection, temperature could be easily changed through the unit’s interface and allow for the impact of seasonality (e.g. temperature rise in summer months) to be assessed.

Identified drawbacks of the set-up includes the fact that the barrels are a closed system and therefore do not account for possible O₂ ingress that may otherwise occur due to inadequate capping systems. Incorporating the measurement of pH within each barrel would also have been useful to infer about ARD generation and potential buffering. In terms of sample usage, testing waste rock under elevated CO₂ concentrations would have provided an effective comparison between waste rock and tailings barrels at these levels. The mass of tailings used in the experiments was significantly lower than the waste rock and using equal masses would allow for improved comparability.

5 Conclusion

The experimental method developed showed potential with regards to the direct measurement of CO₂ and O₂ flux within mine wastes. A limited leak only appeared to occur in one barrel (Barrel 1), with the remainder appearing to maintain their seals. Maintaining variables proved largely successful, with the mass and water content of samples able to be fixed and control of temperature possible. Control over these variables, in addition to the option of using mine wastes from other sites, lends this method a high degree of versatility. A continuation in the logging of gas flux within the barrels in addition with modification (increased instrumentation such as the addition of a pH probe) would be beneficial in increasing the robustness of results.

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