CARBON SEQUESTRATION IN MINE TAILINGS

Year 1 Report of Activities

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Project Overview

The Carbon Sequestration in Mine Tailings project is a three-year research initiative that formally began in May 2005. The project examines the potential for using mine waste to store atmospheric carbon dioxide, thereby mitigating industrial greenhouse gas emissions. The project is sponsored by BHP Billiton (formerly WMC Resources, Ltd.), Diavik Diamond Mine Inc. (DDMI), and the Natural Sciences and Engineering Research Council of Canada (NSERC). It focuses on tailings from two active mines: the Mount Keith Nickel mine, Western Australia and the Diavik Diamond Mine, NWT.

The goals of the project are to document and quantify carbon sequestration already occurring within mine tailings, develop a verification protocol for mineral sequestration so that carbon fixation can be claimed for offsets or traded for carbon credits, and construct a geochemical model for predicting sequestration rate as a function of environment and microbial activity so that sequestration can be accelerated. This will be accomplished by integrated field examination and sampling of mine waste from the two mine sites; experimental analysis and geochemical modeling of mineral sequestration reaction pathways; and X-ray diffraction, transmission/scanning electron microscopy, energy dispersive spectroscopy, and geochemical analysis of sequestration products. The program has site-specific as well global objectives.

The global objectives, which are shared among sites, are:

1. Establish dissolution rate laws for serpentine, the silicate mineral feedstock to the mineral sequestration process in biotic and abiotic environments.
2. Identify mechanisms and establish rate laws for carbonate mineral precipitation.
3. Identify the controlling environmental factors for CO2 fixation.
4. Develop an objective, scientific verification protocol for documenting the amount of mineralogically bound CO2 and fingerprinting the source of carbon.

Research objectives specific to individual mines are:

5. Identify mineral sinks of bound atmospheric CO2 in mine tailings.
6. Demonstrate crystallographic binding of CO2 and isotopically fingerprint the source of bound carbon.
7. Quantify the rate of CO2 uptake by mine residues so that CO2 fixation that is directly attributable to the mining process can be properly claimed for CO2 offsets.

Research results from global objectives are shared with both industrial sponsors, while site-specific research results are reported first to the industrial sponsor for that site.

Research Team

This is a collaborative research project between Professor Greg Dipple of the Mineral Deposit Research Unit at the University of British Columbia (UBC) and Canada Research Chair Gordon Southam of the University of Western Ontario (UWO). The project is the basis for three Ph.D. research projects: Ian Power (UWO), James Thom (UBC), and Sasha Wilson (UBC). Ian's research examines biologically mediated mineral dissolution and mineral precipitation pathways. James' research is to extract abiotic rate laws for mineral dissolution and precipitation from bench top experiments and integrate them into a geochemical model for carbon sequestration. Sasha's research project documents the mineralogical hosts of carbon, uses isotopic fingerprinting to identify the source of carbon, and develops and applies a verification protocol to bound carbon.

Overview of Research Activities

The first field visit to Diavik was delayed until September 2005 due to accommodation shortages onsite associated with the summer construction schedule. G. Dipple and G. Southam visited Diavik from 13th to 15th September to initiate the project, gain familiarity with the industrial operation, and make field observations and collect samples to orient future studies. During field work, three morphologically distinct types of mineral efflorescences were noted on tailings deposits. The sub-zero temperatures, however, prevented assessment of mineral cementation within tailings because the tailings were partially cemented by ice crystals. Eleven samples were collected (Table 1) and returned to UBC and UWO. Subsequent examination and analysis of these samples includes: mineral identification, mineral abundance measurement, surface area measurement, and biofilm microbe identification. These preliminary data are reported here and used to estimate the sequestration potential of Diavik tailings, to note possible carbon sequestration activities already occurring on-site, and to estimate potential cation (Mg^{2+}) release rates from tailings.

Table 1. Sample descriptions and locations

		UTM (NAD27)	
SAMPLE	**Description**	**northing**	**easting**
05DVK1	PK fine mud from bottom of tailings lake at barge		
05DVK2	0 to 3 cm depth exposed, unsaturated tailings	7152138	533040
05DVK3	3 to 20 cm depth exposed, unsaturated tailings	7152138	533040
05DVK4	0 to 0.5 cm depth, nodular surface on tailings	7152099	533035
05DVK5	Cumulative scrapings from white crusts in vicinity of 05DVK4		
05DVK6	0 to ~ 2 cm depth of coarse PK tails including white crust	7151716	534033
05DVK7	2 to ~ 20 cm depth of coarse PK tails, below white crust	7151716	534033
05DVK8	Fine PK tails sampled from pipeline 14 Sept. 2005		
05DVK9	PK water sample at barge		
05DVK10	Diavik fault zone, weathered kimberlite		
05DVK11	Biofilm at kimberlite-groundwater interface, Diavik Pit		

Field Sampling and Observations

A total of ten solid samples and one water sample were collected (Table 1). These include bulk PK fine tailings directly from the processing plant (sample obtained by A. Forest), fine PK tails sediment from the centre of the tailings pond (collected with Ekman sampler by Colleen English), bulk samples of PK fine and PK coarse tailings (Fig. 1A), selective sampling of three mineral efflorescences that develop as surface or subsurface crusts on tailings that are exposed above water level (unsaturated), and samples of pit biofilm and tailings impoundment facility water.

Two morphologically distinct mineral efflorescences were observed on the surface of PK fine tailings where exposed above the waterline of the tailings containment pond. One type consists of nodular, cemented(?) encrustations on some exposed horizontal surfaces (Fig. 1B). The lighter colour of these nodules may reflect lower moisture content or may represent a mineral precipitate. They do not have a salty taste, and do not react strongly with dilute HCl. Reaction with HCl has aided in the field identification of hydrated magnesium carbonate minerals at other mine sites, but is difficult at Diavik because of the abundance of calcite (which also reacts with HCl) in tailings. Wilson (2005) noted nodular encrustations on horizontal surfaces in chrysotile mine tailings from Clinton Creek, Yukon. The Clinton Creek "spires" are cemented by the hydrated magnesium carbonate minerals nesquehonite, dypingite and hydromagnesite that contain atmospheric CO_2 (Wilson, 2005).

The second efflorescence in PK fine tailings is distinct white powder precipitates located on indentations and scalloped surfaces on the sides of tailings piles (Fig. 1C). These precipitates appear to be mineralogically distinct. They do not have a salty taste, and do not react with dilute HCl.

A third type of efflorescence was observed immediately (~1mm) below the surface of PK coarse tailings (Fig. 1D). This crust is unusual in being laterally continuous for more than a metre. The white powder precipitate does not have a salty taste but did react noticeably with dilute HCl, although this is difficult to distinguish from reaction of bulk calcite in the tailings.

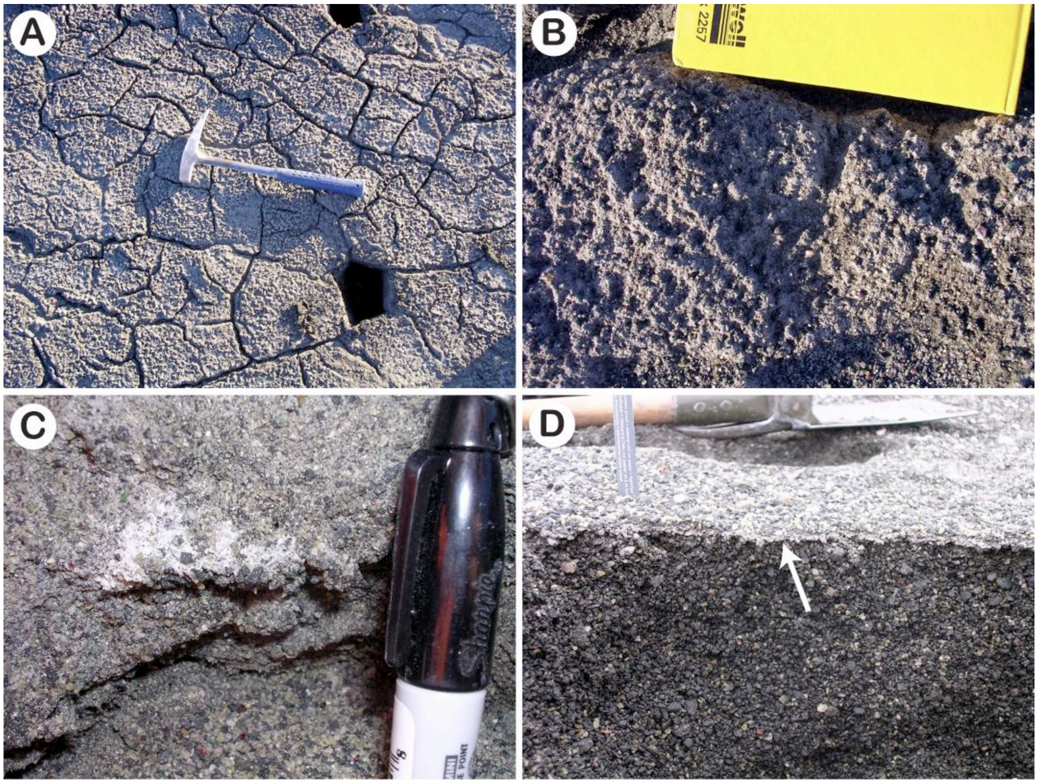

Figure 1. Field sampling sites and mineral efflorescences. ***a****) Surface of PK fine tailings above tailings pond water line at sampling site 05DVK2 and 3. Note desiccation cracks in surface deposits.* ***b****) Nodular efflorescence on horizontal exposure of PK fine tailings. Powder X-ray diffraction data indicate that these nodular crusts may contain the hydrated magnesium carbonate mineral nesquehonite* ***c****) White powdery mineral efflorescence on sub-vertical surface of PK fine tailings exposure. White mineral precipitates are gypsum.* ***d****) Continuous thin white crust just below surface of PK coarse tailings (white arrow). The crust contains nesquehonite.*

Mineralogical Analysis

Mineral phases were identified with X-ray powder diffraction (XRPD). XRPD data for mineral identification were collected on a Siemens D5000 θ-2θ diffractometer. Constituent mineral phases were identified with reference to the ICDD PDF-4 database using DIFFRACplus EVA (Bruker AXS 2004). The normal-focus Co X-ray tube was operated at 35 kV and 40 mA. Two scanning modes were utilized. Mineral identification and mineral abundance were assessed from bulk scans with 1 second and 0.02 degrees 2-theta per step, over 3 to 80 degrees 2-theta. Total scan time per sample was 35 minutes. A slow scan over a restricted range of 2-theta was used to search for trace amounts of magnesium carbonate minerals. These scans focused on the (101) peak for nesquehonite at 40 seconds per step, 0.02 degrees per step over 15.3 to 16.5 degrees 2-theta for a total scan time of 120 minutes per sample.

Mineral identification scans can detect minerals present at abundances greater than about one weight percent. These data are presented in table 2. The mineral content of bulk tailings are similar to that predicted by Baker et al., (2001). Serpentine and forsterite represent the two principal mineralogical sources for Mg^{2+} which could be used for carbon sequestration. Calcite is ubiquitous and is the only carbonate mineral present at an abundance of 1 to 2 wt % or greater.

Recycling of bedrock carbon provides an alternative source of carbon that might be incorporated into tailings efflorescences. Dissolution of calcite and re-precipitation of Ca-carbonate yields no net carbon sequestration, and has been noted in tailings of the Cassiar chrysotile mine, B.C. (Wilson, 2005). At Cassiar, the precipitates are aragonite which can be readily distinguished from Mg-carbonate efflorescences that result from dissolution of Mg-silicate minerals. Dissolution of Mg-silicates and precipitation of Mg-carbonate mineral efflorescences is the principal carbon sequestration process observed in chrysotile mine tailings (Wilson, 2005).

The mineral identification scans also indicate that the white powder efflorescences on PK fine tailings (Fig. 1C) are composed of gypsum. They may represent dissolution of bedrock calcite and re-precipitation as sulfate. Sulfate precipitation could release to the atmosphere the CO_2 that was liberated during calcite dissolution, but the amount occurring in the tailings containment facility appears to be insignificant.

Detailed scans for hydrated magnesium carbonates can detect minerals present at abundances of less than one weight percent. Detailed scans (e.g., Fig. 2; results indicated as "tr" in Table 2) indicate the presence of nesquehonite in the nodular efflorescences on PK fine tailings (sample 05DVK4) and in the continuous white crusts in the PK coarse tailings (05DVK6). Even in selective crust samples, the abundance of nesquehonite is probably less than 1 weight percent, indicating that the amount of precipitate is not significant from a greenhouse gas perspective. The low abundance has also prevented isotopic analysis of the nesquehonite to fingerprint the source of bound carbon. Future work will employ microsampling techniques to collect relatively pure nesquehonite for isotopic analysis.

Field, mineralogical, isotopic, and experimental work by Power et al. (2005) and Wilson (2005) indicate that nesquehonite is associated with abiotic carbonate mineral precipitation in mine tailings and in bog environments, whereas hydromagnesite and dypingite form from microbially mediated precipitation. The formation of nesquehonite at Diavik therefore may represent abiotic precipitation, which is is not surpising for this climatic and depositional environment. Future stable isotopic analysis of nesquehonite from Diavik will confirm or refute this hypothesis. One potential pathway to accelerate carbon sequestration would be to promote microbial precipitation (Power et al., 2005).

Table 2. Mineral content from powder X-ray diffraction data.

SAMPLE	05DVK2	05DVK3	05DVK4	05DVK5	05DVK6	05DVK7
Serpentine	+	+	+	+	+	+
Vermiculite-2M/Clinochlore*	-	-	-	-	-	-
Forsterite	+	+	+	+	+	+
Phlogopite-1M	-	-	-	-	-	-
Ilmenite	-	n/d	-	-	-	-
Quartz	-	-	-	-	-	-
Almandine	-	-	-	-	-	-
Calcite	-	-	-	-	-	-
Albite	n/d	n/d	-	-	-	-
Diopside	-	-	-	-	-	-
Gypsum	n/d	n/d	n/d	-	n/d	n/d
Nesquehonite	n/d	n/d	tr	tr?	tr	tr

+	Present as a major phase
-	Present as a minor phase
tr	Present in trace amounts
n/d	Not detected
*	Vermiculite and Clinochlore may both be present (Need to use glycerol at a later date to determine if clinochlore present or if just vermiculite.)

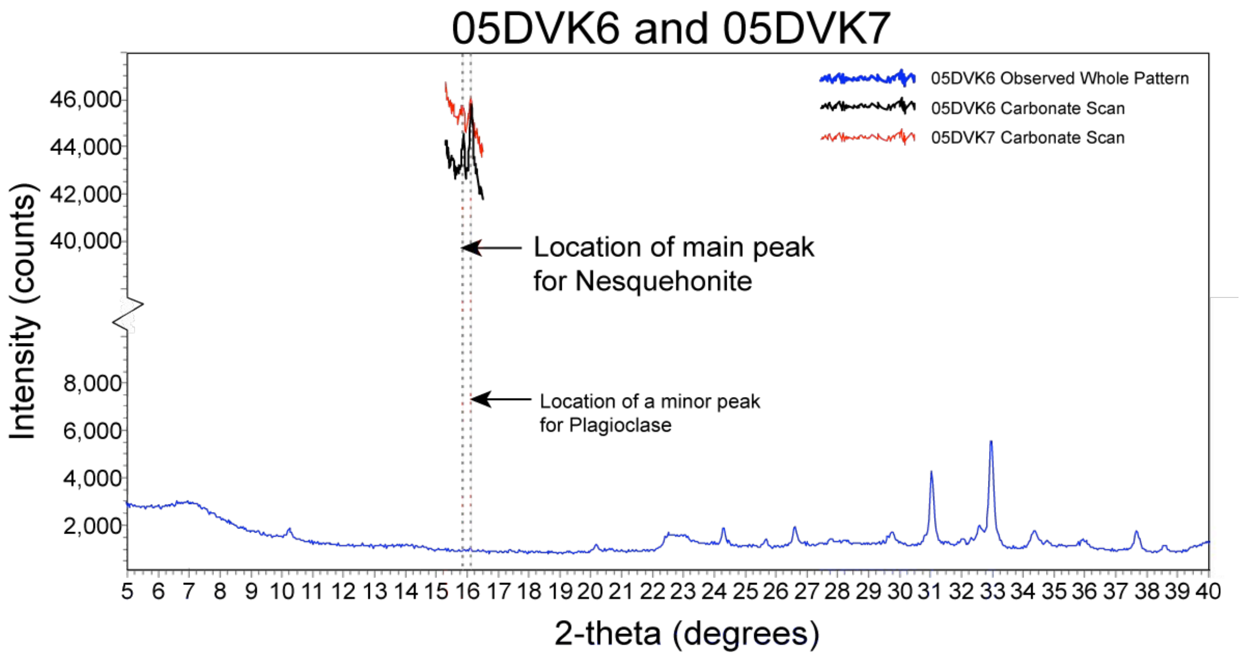

Figure 2. Powder X-ray diffraction pattern of samples 05DVK6 and 05DVK7. Detailed scans detected the 101 peak of nesquehonite, indicating trace amounts of the hydrated magnesium carbonate in these samples.

Mineral abundance data in select samples was assessed using the Rietveld method and X-ray powder diffraction data (following Wilson, 2005). An aliquot of each sample was powdered using a tungsten carbide ringmill. A 10 wt.% spike of annealed CaF_2 was added to 3.00 g of each sample of tailings and the mixture was ground under anhydrous ethanol with synthetic corundum grinding elements for 10 minutes in a McCrone micronising mill to reduce the mean grain size and to ensure homogenization. Samples were mounted in a back-loading aluminum cavity holder of the design described by Raudsepp and Pani (2003). Preferred orientation of inequant crystallites was minimized by covering the top of the cavity with a sheet of ground glass and loading powdered samples against the roughened surface. To further inhibit preferred orientation of crystallites for bulk compositional scans, the surface of each sample was serrated with a razor blade along two axes: one parallel to the axis of the diffractometer goniometer and the second in the perpendicular direction. No blades were used for the carbonate scans because surface roughness promotes the scattering of X-rays, thus reducing the intensity of Bragg reflections and making trace phases more difficult to identify.

The data are presented in Table 3 and a sample scan included in Fig. 3. Sample 05DVK3 is bulk sample of PK fine tailings to 20 cm depth. This sample contains 58 weight percent serpentine and 28 % forsterite. Sample 05DVK7 is bulk sample of PK coarse tailings. It contains 69 % serpentine and 17 % forsterite. These data will be confirmed with more systematic and representative sampling in future years, but allow a first prediction of the sequestration capacity of Diavik tailings. The sequestration capacity based on complete reaction of end-member stoichiometric serpentine and forsterite and sequestration within nesquehonite (Mg:CO_2 of 1:1 on a molar basis) is listed in Table 3, assuming a tailings production of 2 million tonnes per year. Sample 05DVK3 likely presents the best estimate because of the predominance of fine tailings at Diavik. On this basis the sequestration capacity of Diavik tailings is approximately 900,000 tonnes. This represents 640% of annual greenhouse gas emissions (141,600 tonnes CO_2 equivalent).

Table 3. Mineral abundance (weight %) from Rietveld analysis of powder X-ray diffraction data.

SAMPLE	**05DVK2**	**05DVK3**	**05DVK4**	**05DVK5**	**05DVK7**
Serpentine	**65.71**	**57.88**	**25.31**	**29.76**	**68.86**
Vermiculite-2M	4.80	4.15	4.91	6.65	3.11
Forsterite	**15.86**	**28.39**	**50.94**	**45.07**	**16.86**
Phlogopite-1M	0.01	0.00	0.00	0.05	0.00
Ilmenite	0.33	0.00	0.82	0.27	0.31
Quartz	2.76	2.25	3.74	1.98	2.84
Almandine	3.81	1.94	5.40	3.05	3.33
Calcite	5.27	4.06	2.52	2.73	3.22
Albite	0.00	0.00	3.86	5.32	0.00
Diopside	1.45	1.34	2.50	4.16	1.46
Gypsum	0.00	0.00	0.00	0.95	0.00
Sum	100.00	100.00	100.00	100.00	100.00
CO2 capacity*	824,445	906,520	878,431	847,343	866,988

NB The unit-cell and space-group data for Lizardite-1T (Melini and Vitti 1994) were used to model the serpentine-group minerals with the Pawley method.

NB There is phlogopite-1M in all of these specimens, but it is significantly less than 1 wt.%

* Annual CO2 sequestration capacity (tonnes) based on 2 million tonnes tailings production per year.

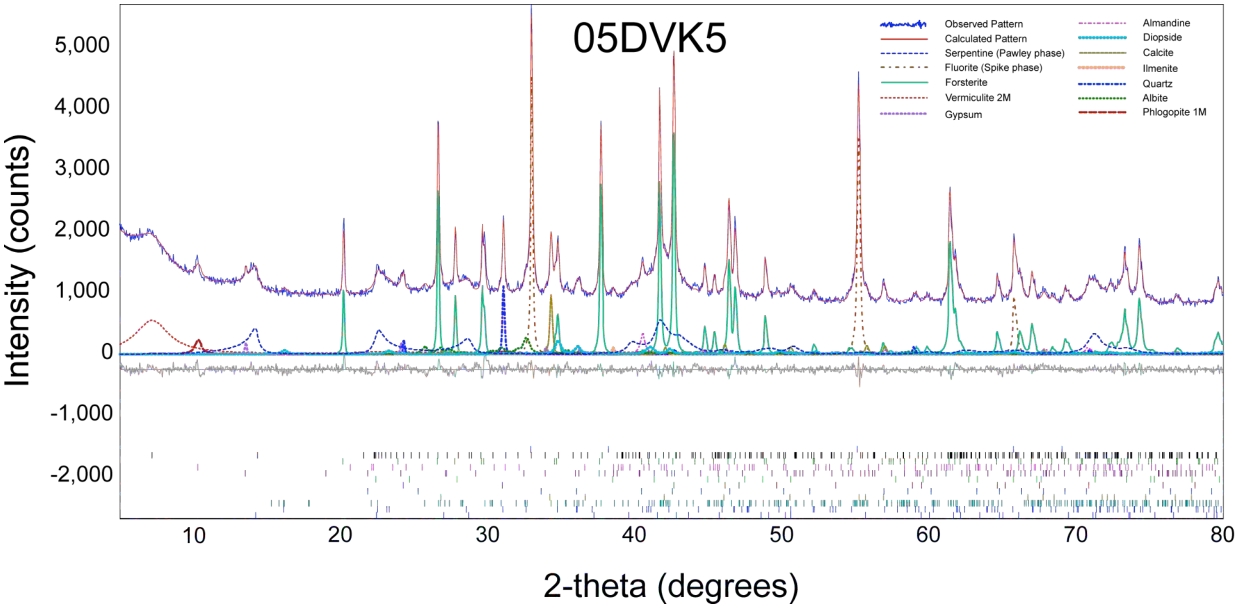

Figure 3. Powder X-ray diffraction scan of sample 05DVK5. The Rietveld method best-fits (in a least squares sense) a forward model pattern to the observed pattern by adjusting mineral abundance. The Rietveld method and analytical techniques employed here are explained in detail by Wilson (2005).

Surface Area Analysis and Mineral Dissolution Rates

Surface area can exert a first order control on mineral dissolution rates. Typically the Brunauer-Emmett-Teller isotherm is used to model adsorption-desorption of water, nitrogen, argon, or krypton (Brunauer et al., 1938). The BET method measures surface area of a material by relating it to the area of the gas molecule used in the measurement. This would imply that different molecules yield different values for the measured surface area. The BET isotherm is based upon an adsorption-desorption dynamic equilibrium model (Brantley et al., 1999). The BET equation conveys a relationship between the number of moles adsorbed on a gram of material, n, and the number of moles adsorbed as a monolayer on a gram of material, n_m,

$$\frac{P/P_o}{n(1-P/P_o)} = \frac{1}{n_m c} + \frac{c-1}{n_m c}(P/P_o)$$

P is the gas pressure, P_o is the saturated vapor pressure of the gas, and c is related to the heat of adsorption of the gas on the surface. Surface area is determined by plotting $\frac{P/P_o}{n(1-P/P_o)}$ against P/P_o where the value of n_m can be determined from

$$n_m = (slope + intercept)^{-1}$$

Once n_m is known, the specific surface area, A_s, can be calculated

$$A_s = n_m a_m N_A$$

Where N_A is Avogadro's number and a_m is the cross sectional area of the gas molecule.

The surface area of Diavik tailings was calculated using the BET method on a multi-point N_2 gas adsorption isotherm. The N_2 gas adsorption isotherm measurements were made using a Quantachrome-1A system. Samples were first degassed at 150°C for 24 hours and were subsequently analyzed at liquid nitrogen temperatures. Surface area measurements for samples were found to range from 42 to 61 m^2/g (Table 4).

Table 4. Surface area data.

SAMPLE	**05DVK1**	**05DVK3**	**05DVK7**	**05DVK8**
Surface Area (m^2/g)	61.03	42.50	42.64	53.11

Diavik tailings have unusually high surface areas (42 to 61 m^2/g) that may translate into very fast mineral dissolution rates. For comparison, the surface area of finely ground forsterite used in dissolution kinetic experiments by Porovsky and Schott (2000) was about 0.08 m^2/g. In other studies, we have attritubed the unusually high surface area of chrysotile mine tailings, 15 m^2/g (our unpublished data), to the morphology of chrysotile. Diavik tailings have surface area that is even higher, and difficult to rationalize with any simple surface geometric model. Unless there is substantial internal porosity to serpentine and forsterite, it is unlikely that the measured surface areas apply to these minerals. If they do apply, the mineral dissolution rates within tailings may be very rapid. Alternatively, the high bulk surface areas may be due to internal surfaces of vermiculite. We will image mineral surfaces in Diavik tailings with high resolution scanning electron microscopy to determine in these high surface areas apply to forsterite and serpentine.

Cation (Mg) release via silicate mineral dissolution may be the rate-limiting step in the formation of Mg-carbonates in the mine tailings environment (Wilson, 2005). Mineral dissolution varies with temperature and fluid chemistry. The dissolution kinetics of forsterite were documented by Porovsky and Schott (2000), but until recently no reliable published rate law for serpentine dissolution existed. Ph.D. student James Thom has extracted a dissolution rate law for the serpentine mineral chrysotile from continuously-stirred reactor dissolution experiments conducted at UBC over the past year. We apply his rate law (Thom and Dipple, 2005) to serpentine dissolution in Diavik tailings on a per gram basis because the reactive surface area of chrysotile is difficult to measure. Forsterite dissolution was estimated using a conservative surface area of 0.08 m^2/g and the measured value of 40 m^2/g (Figs. 4A and 4B respectively). These calculations assume dissolution in water at 25 C and far from equilibrium conditions, and use mineral abundance data from sample 05DVK3.

The total amount of Mg released depends on the amount of time per year that the tailings are in contact with aqueous solution (noted as reaction time in Fig. 4). The rate calculations indicate total amount of CO_2 sequestered from a 2 million tonne per year tailings operation assuming that silicate dissolution is the rate limiting step and 100% efficiency in binding all released Mg with CO_2. The calculations are at best crude approximations of sequestration capacity and rate, but emphasize the potential for acceleration of carbon sequestration at Diavik. Residence times of a few weeks may be sufficient to offset a substantial fraction of Diavik greenhouse gas emissions (Fig. 4). Future experiments will directly measure the rate of Mg release from bulk tailings dissolution over a range of pH.

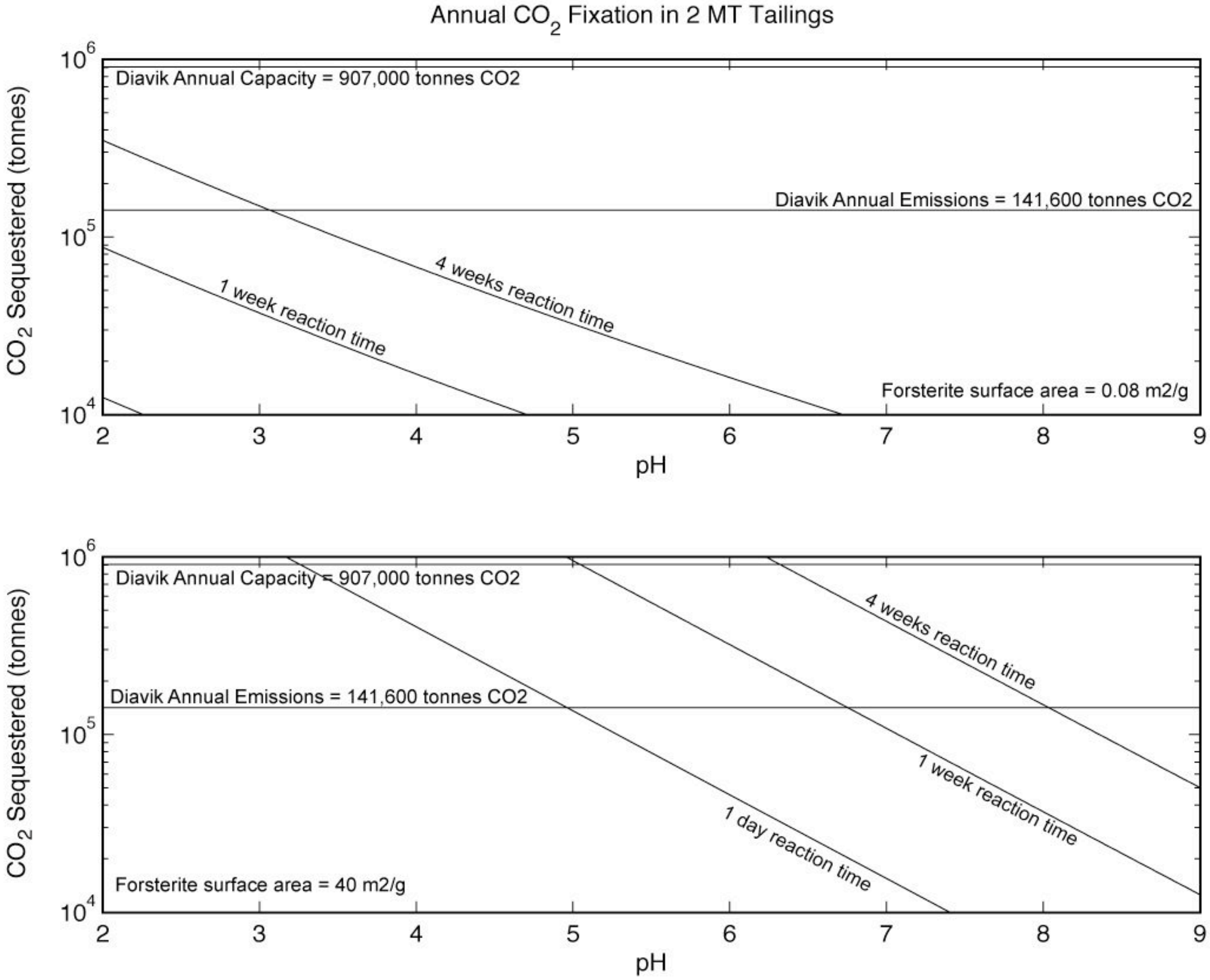

Figure 4. Carbon dioxide sequestration rate for Diavik tailings based on serpentine and forsterite dissolution kinetics.

Geomicrobiology

Microbial communities that have demonstrated the ability to enhance the formation of carbonate minerals were identified in the biofilm samples collected during the site visit, September 2005. Initial light- and scanning electron- microscopy indicate minor traces of carbonate present within a biofilm. Traditional field fizz tests for analyzing carbonate, that were modified for use using light microscopy, demonstrate the formation of minor amounts of carbon dioxide, i.e., that carbonate is present within biofilm samples. Further microbial sampling and analysis is needed confirm the ability of local microbial communities to aid in carbonate formation at the Diavik site.

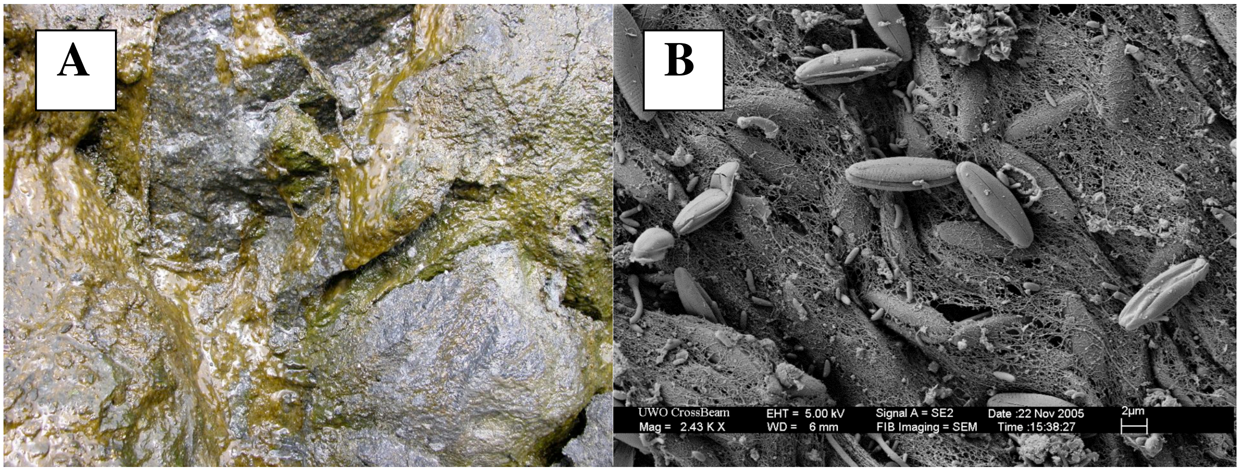

Figure 5. Microbial biofilm collected at the kimberlite groundwater interface at the Diavik fault zone. The biofilm was found to be composed of diatoms, cyanobacteria and heterotrophic bacteria all held with a fibrous carbohydrate matrix (B).

Summary and Future Work

On the basis of field work and samples collected in September, 2005 we have:

1) Identified three morphologically and mineralogicaly distinct efflorescences in Diavik mine tailings. Two of the efflorescences contain the Mg-carbonate mineral nesquehonite. Future work will assess if these precipitates are binding atmospheric CO_2 and determine bulk abundance within Diavik tailings deposits. These results address research objectives 5, 6 and 7 (as listed in **Project Overview**).
2) Determined the mineral content of a limited number of tailings samples using a new methodology for serpentine-bearing tailings (Wilson, 2005; research objective 4). These data allow for the total sequestration potential of Diavik tailings to be assessed (900,000 tonnes per year, table 2). This sequestration potential exceeds total Diavik greenhouse gas production by a factor of more than six. Future work will refine this estimate with more extensive and representative sampling programs.
3) Combined new Mg-silicate mineral dissolution rate data (research objective 1) with surface area measurements to calculate order-of-magnitude Mg release rates. If the unusually large surface areas of Diavik tailings are representative of serpentine and/or forsterite surface areas, then the potential rate of carbon sequestration may be much greater than previously thought. Even if surface areas are small (Fig. 4A), reaction times of weeks may suffice to extract sufficient Mg to offset Diavik greenhouse gas emissions. Future work will use high-resolution imaging to examine serpentine and forsterite surfaces. Dissolution rate law data for other serpentine minerals will be measured (research objective 1) and the new rate data incorporated into more rigorous geochemical models of mineral dissolution and precipitation.
4) Analyzed microbial biofilms from the Diavik pit and identified indigenous flora that could be used to promote the precipitation of mineral carbonates (research objective 2).

At this early stage we see no reason to modify the original work plan as outlined in the research proposal. The work proposed here falls within that outline and our results to date are consistent with the milestones of that proposal. This work will continue on existing samples until the spring or summer of 2006, at which time an extended field visit and sampling program can be initiated.

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