PHASE 2 INITIAL BOREHOLE DRILLING AND TESTING, IGNACE AREA

WP4f Data Report – CEC and BET Investigations of Core Samples for IG_BH04

APM-REP-01332-0355 **May 2022**

Wood Plc.

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Phase 2 Initial Borehole Drilling and Testing, Ignace Area

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6th Floor, 22 St. Clair Avenue East, Toronto, Ontario, M4T 2S3

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Table of Contents

Page

1.0 Introduction 1
2.0 Sampling and Sample Preparation 1
3.0 Experimental Set-ups and Analytical Methods 2
3.1 Determination of Specific Surface Area using the BET Method 2
3.2 Determination of Cation Exchange Capacity (CEC) 3
4.0 Results 5
4.1 BET Specific Surface Area 5
4.2 CATION Exchange Capacity 6
4.2.1 CECNi 6
4.2.2 CEC_{cation} 11
4.2.3 "Zero Exchange Experiment" 16
5.0 Summary 19
6.0 References 20

List of Figures

Figure 4-1: The Specific Surface Area Measured at Different Grain Size Fractions and at A Core Slice Using the BET Method 6
Figure 4-2: Nickel Concentration (Left) and CEC_{Ni} Values of the Largest Grain Size Fraction 10-20 mm Measurements over Experimental time. Experiments run 71 days 10
Figure 4-3: Results of the CEC_{Ni} Values of the Different Grain Size Fractions (0.065-0.2 mm, 2-4 mm, 10-20 mm) and Rock/Test Solution Mass Ratios Calculated by Nickel Concentration 11
Figure 4-4: Results of the $CEC_{Cations}$ Values of the Different Grain Size Fractions (0.065-0.2 mm, 2-4 mm, 10-20 mm) and Rock / Test Solution Ratios Calculated by the Sum of Concentrations of All the Cations in the Solution 15
Figure 4-5: Experimental Bottles with the Grain Size Fraction (2-4 mm) Experiments Immediately After Start (Left) and After 96 h Shaking (Right) 17
Figure 4-6: Results of the CEC Values of the Different Grain Size Fractions (0.065-0.2 mm, 2-4 mm, 10-20 mm) and Rock / Test Solution Ratios Calculated by the Ni-Concentration (CEC_{Nickel}, Grey Bar), the Sum of All Cations in the Solution (CEC_{cation}, Blue Bar) and Corrected with the Zero Exchange Experiment Values ($CEC_{cation\ corr.}$; Orange Bar) 18

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List of Tables

Page

Table 2-1: Overview of the core sample taken from IG_BH04 for the CEC/BET measurements 2
Table 3-1: Used Masses of Crushed Rock Material for the Determination of Specific Surface Area by BET Method 2
Table 3-2: Overview of the Experimental Set-ups for CEC Measurements 3
Table 4-1: Results of the Specific Surface Area Measured at Different Grain Size Fractions by the BET Method 6
Table 4-2: Element Concentrations of the Final CEC Experiment Solutions Using Different Rock / Test Solution Mass Ratios Conducted with the Smallest Grain Size Fraction (0.065-0.2 mm) After 48 h Sample Name IG_BH04_SA001 65-200 µm 7
Table 4-3: Element Concentrations of the Final CEC Experiment Solutions Using Different Rock / Test Solution Ratios Conducted with the Medium Grain Size Fraction (2-4 mm) After 96 h Sample Name IG_BH04_SA001 2-4 mm 8
Table 4-4: Element Concentrations of the Final CEC Experiment Solutions Using Different Rock / Test Solution Ratios Conducted with the Largest Grain Size Fraction (10-20 mm) After 71 days Sample Name IG_BH04_SA001 10-20 mm 9
Table 4-5: Calculated CEC_{Ni} Values, Mean CEC_{Ni} Values and Standard Deviation from the CEC Experiments with Three Grain Size Fraction 0.065-0.2 mm, 2-4 mm and 10-20 mm 10
Table 4-6: CEC Values of Each Cation (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe, Si^{4+}) and CEC Values of the Sum of Cations of the Smallest Grain Size Fraction (0.065-0.2 mm). Sample Name IG_BH04_SA001 0.065-0.2 mm. All Values in meq/kg rock 12
Table 4-7: CEC values of Each Cation (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe, Si^{4+}) and CEC Values of the Sum of Cations of the Medium Grain Size Fraction (2-4 mm). Sample Name IG_BH04_SA001 2-4 mm. All Values in meq/kg rock 13
Table 4-8: CEC values of Each Cation (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe, Si^{4+}) and CEC Values of the Sum of Cations of the Largest Grain Size Fraction (10-20 mm). Sample Name IG_BH04_SA001 10-20 mm. All Values in meq/kg rock 14
Table 4-9: Calculated CEC_{cation} Values (Mean CEC-Cation Values) from the CEC Experiments with the Three Grain Size Fractions 0.065-0.2 mm, 2-4 mm and 10-20 mm 15
Table 4-10: Overview of the Experimental Set-ups of the "Zero Exchange Experiment" 17
Table 4-11: Calculated $CEC_{cation\ zero}$ Values (Mean CEC-Cation Values) Based on all Cation concentrations from the Zero Exchange Experiments with the Three Grain Size Fraction 0.065-0.2 mm, 2-4 mm and 10-20 mm after experimental time. The CEC_{cation} Values and the $CEC_{cation\ coor.}$ Values After Correction with the $CEC_{cation\ zero}$ Values 18

List of Appendices

Appendix A: Analytical Raw Data

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1.0 Introduction

Within the characterisation of the Ignace investigation site, one core sample was taken from borehole IG_BH04 for the measurement of cation exchange capacity (CEC) and specific surface area (by BET method) at different grain size fractions. The CEC measurements were conducted by Hydroisotop GmbH. The BET measurements were performed at the University of Bern. The results of the experimental measurements are documented in the following sections of this report.

2.0 Sampling and Sample Preparation

One core sample from borehole IG_BH04 (sample ID: IG_BH04_SA001) with a length of 0.36 m and mass of 2.8 kg was taken at an average depth of 565.36 mbgs (downhole) on January 21, 2020 for the determination of CEC (cation exchange capacity) and specific surface area using the BET method (Table 2-1). Sampling was conducted by Wood PLC according to the instructions provided by Hydroisotop GmbH (Hydroisotop) via personal training. After recovery from the borehole, the core sample was photographed and immediately packed in a plastic bag, evacuated and sealed airtight. This procedure was repeated for a second plastic bag and a final Al-coated plastic layer. The sample was stored in a refrigerator on site and then sent to Hydroisotop, Germany, in a cooler.

The sample was sent by Wood on January 24, 2020 and arrived at the Hydroisotop lab on January 30, 2020. The sample was well packed and arrived in the lab with preserved vacuum. At Hydroisotop the sample was stored in the fridge at 4°C.

The core sample was prepared for the CEC and BET measurements on May 8, 2020. The core sample was unpacked, photographed and immediately wrapped into Parafilm™ and cut by dry-sawing into full-diameter sections of 1 to 2 cm thickness. After sawing the core sections were crushed using a steel mortar and the crushed rock material was sieved using a vibration sieving tower (Retsch AS 450) and analytical stainless-steel sieves with mesh sizes of 0.065, 0.2, 2.0, 4.0, 10 and 20 mm. The obtained crushed rock material with grain size fractions of 10-20 mm, 2-4 mm,0.065-0.2 mm and < 0.065 mm was stored in PE bottles. After the first step of crushing and sieving, sufficient rock material of the largest grain size fraction 10-20 mm was achieved. Crushing and sieving was repeated until sufficient rock material with a grain size of 2-4 mm was obtained. Afterwards the grain size fraction of 0.2-2 mm was milled with a zirconium oxide grinding beaker and balls (Retsch AS 450) (300 rpm, 1 min) and sieved again to obtain the smallest grain size fraction (0.065-0.2 mm) needed for CEC and BET experiments.

One core section with a thickness of 3 cm and mass of 293.8 g was retained for BET experiments on a rock cylinder. Before sending this core slice and about 35 g of each grain size fraction to University of Bern for BET measurements, the rock materials were dried at 105 °C for two weeks.

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Table 2-1: Overview of the core sample taken from IG_BH04 for the CEC/BET measurements

Sample description		Depth			Sampling					
Sample ID	Hydro Lab No.	from (m)	to (m)	ave (m)	Length (m)	Test Type	Date sampled	Time recovered (hh:mm)	Date sent	Date arrived
IG_BH04_SA001	339045	565.18	565.54	565.36	0.36	SA	21. Jan, 2020	12:27	24.01.2020	30.01.2020

3.0 Experimental Set-ups and Analytical Methods

The specific surface areas for different grain size fractions were determined at the University of Bern. CEC experiments were conducted at Hydroisotop. Unless otherwise specified, the analytical work was conducted at Hydroisotop GmbH, Germany.

3.1 Determination of Specific Surface Area using the BET Method

The specific surface area (SSA) was determined on the four grain size fractions (crushed rock) and a 3 cm thick rock slice cut out of the intact core by nitrogen adsorption isotherms at -196°C, using a BELSORP miniX surface analyser at the University of Bern (Dr. Urs Eggenberger).

A mass of 5 – 30 g of crushed rock material (Table 3-1; maximum volume 18 cm^3) was weighed (to an accuracy of 0.001 g) in a glass container until $^1/_3$ to maximum $^2/_3$ of the container was filled. After inserting the glass container containing the rock sample into a BELPREP vac III, the vacuum pump was started and the sample was heated up to 105 °C overnight. Hereby the rock sample was desorbed of primary adsorbed gases under vacuum before measuring the adsorption isotherm. After the glass container cools down for a minimum of six hours, the vacuum pump was stopped and the valves of the glass container were closed. To determine the weight loss, the glass container was weighed before heating and after cooling.

Table 3-1: Used Masses of Crushed Rock Material for the Determination of Specific Surface Area by BET Method

Hydro Lab-No.	Grain size fraction	Used masses (g)
	<0.065 mm	4.670 / 4.724
344886	0.065-0.2 mm	5.558
344887	2-4 mm	30.302
344888	10-20 mm	21.624
344889	Core slice (3 cm thickness, 6 cm diameter)	27.602

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For the BET measurement the neck of the glass container was fixed in the BELSORP-miniX surface analyzer and the measurement was started using the BELSORP-miniX software. After approximately three hours the measurement was finished. The specific surface area, expressed as m^2/g, was then calculated from the amount of adsorbed N_2 and the sample weight according to the BET method (Brunauer et al. 1938) for a relative pressure of P/P_0 (measured vacuum pressure to the atmospheric pressure) from 0.05 to 0.3.

Certified standard materials (four different BET-standards with specific surface areas from 0.18 to 214 m^2/g) were used for calibration and quality checks. The reproducibility for BET surface area is < 2 % CV (Coefficient of Variation) at 1 m^2 measured area.

3.2 Determination of Cation Exchange Capacity (CEC)

The CEC is determined by the nickel-ethylenediamine (Ni-en) method originally developed for clay-rich sedimentary rocks (Baeyens and Bradbury 1994; Bradbury and Baeyens 1997, 1998) and adapted to crystalline rocks according to Eichinger and Waber (in progress). This method is based on the premise that Ni, as a strong sorbent, will exclusively displace (and replace) all cations from the exchange sites in sheet silicates (the concentrations of the displaced cations will increase in solution, while the Ni concentration will accordingly decrease, allowing determination of the cation exchange capacity).

The CEC measurements were conducted on three crushed rock size fractions (0.065 - 0.2 mm, 2 - 4 mm and 10 -20 mm). Ni-en extractions were performed in duplicate at four different solid to liquid mass ratios (0.1, 0.25, 0.5, 1). In addition, a blank was carried out for each experimental run as a control.

The experimental stock solution (0.0005 molar Ni-en solution) was prepared by adding ethylenediamine to a $Ni(NO_3)_2$ solution. The Ni-en solution was prepared using degassed, oxygen- and CO_2-free water that was prepared in the glove box by boiling and N_2 bubbling for 30 minutes.

For the determination of the CEC values, the batch volumes were selected according to the grain size and the experiment duration. Table 3-2 gives an overview of the respective rock and test solution masses for the individual experiments.

Table 3-2: Overview of the Experimental Set-ups for CEC Measurements

Rock/Water mass ratio		1:1		1:2	1:4	1:10	Blank
Experiment 0.065-0.2 mm (48 h) and 2-4 mm (96 h)							
Rrock material mass	g	40		20	10	10	0
Test solution mass	g	40		40	40	100	75
Experiment 10-20 mm							
Rock material mass	g	60		30	15	10	0
Test solution mass	g	60		60	60	100	75

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The weighted rock material was added into a 100 ml polyethylene bottle. To start the experiment, the experimental solution was added to the rock material in the polyethylene bottle in the glove box using a graduated cylinder. Immediately after addition of the solution, the pH and redox potential of the experimental solution were measured. The bottles were then closed and continuously shaken upside down.

The samples were shaken end-over-end in the polyethylene bottles. The reaction time depended on the sizes of the crushed rock. The smallest size (0.065-0.2 mm) samples were shaken for 48 h and the size 2-4 mm samples for 96 h. The largest size fraction (10-20 mm) samples were shaken until stable Ni-concentrations in the solution were achieved. After nickel concentration was stable, an aliquot of 0.5 ml sub-sample was taken at 7, 14, 21 and 28 days. After 36 and 62 days, an aliquot of 1 ml sub-sample was taken. The experimental solutions were sampled after 7, 14, 21, 28, 36 and 62 days as sub-samples until the final sampling at day 71.

500/1000 µl sub-sample was taken from the polyethylene bottle with a pipette and filled into a 1.5 ml reaction tube. The reaction tube was centrifuged until the solution was clear. The solution was then carefully removed with a pipette. 100 µl solution was directly added to a 10 ml centrifuge tube containing 9900 µl of 2% HNO_3 for Ni concentration measurement (1:100 dilution). The remaining solution was transferred to a clean 1.5 ml reaction tube for the cation concentration analyses by IC.

For the final sampling of all experimental set-ups, the pH value and the redox potential of the final solution were measured first in the glove box. The solution was then decanted into a 50 ml centrifuge tube and centrifuged until the solution was clear. The solution was divided into 3 vials in the glove box for concentration analysis.

1. For the photometer and IC measurements, a partial sample (vial 1) was filtered with a 0.45 µm syringe filter and filled into a tube.

2. For the Fe^{2+} analysis, a 2nd aliquot of the sample (vial 2) was filled unfiltered into a tube with 20 µl of HNO_3 prepared.

3. For all cations measured by the AAS, the 3rd subsample (vial 3) was filtered with a 0.45 µm syringe filter and filled into a tube with 20 µl of HNO_3 pre-filled.

All samples were immediately sent to the laboratory for measurement after the experiment was finished.

The Ni-en solution was analysed for Ni^{2+} concentration before and after extraction by AAS with a detection limit of 0.01 mg/L. The analytical error of the AAS is ± 10 % based on multiple measurements of high-grade, commercial check-standard solutions.

For the sub-samples, nickel, calcium and magnesium concentrations were determined by AAS and potassium, sodium and ammonium concentrations by IC.

For the final samples, Ni^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+}, Ba^{2+}, Fe^{2+}, Fe^{3+}, Al^{3+} concentrations were determined by AAS. Na^{+}, K^{+}, Cl^{-}, SO_4^{2-}, F^{-}, nitrate and ammonium concentrations were measured by IC, and silicon concentration by photometric method.

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The cation exchange capacity (CEC) was then derived from the Ni consumption, i.e., the difference between the Ni concentration in the initial and the final extract solutions. The cumulative error of the entire procedure (i.e., extraction and analysis) is approximately 10 %.

In order to confirm the CEC value obtained by measuring the nickel concentration, major and trace cation concentrations (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Sr^{2+}, Ba^{2+}, Fe^{2+}, Fe^{3+}, Al^{3+}) in the final extract solution were determined. The nickel, major and trace cation concentrations were measured in duplicate. pH was measured in the individual solutions before and after the experiments using a Schott Titroline alpha system.

To obtain the concentrations of redox specific ions like $Fe^{2/3+}$ and NH_4^+, the CEC experiments were conducted under anoxic conditions in the glove box using oxygen-free test solution (for production, see section above). The glove box was filled with nitrogen and a possible ingress of air was controlled by an oxygen measurement in the inner atmosphere. The redox potential of the solution was analyzed before and after the experiments for all CEC solutions.

4.0 Results

The results of the BET and CEC experimental measurements of the core (IG_BH04_SA001) taken from borehole IG_BH04 are explained in the following sections.

4.1 BET Specific Surface Area

The results of the BET measurements are summarized in Table 4-1and Figure 4-1. The accuracy of the measurement for the larger grain size fractions (2-4 mm and 10-20 mm) and the core slice is ± 30%. Additionally, the specific surface area of the grain size fractions 0.065-0.2 mm and <0.065 mm was determined in repeat measurements.

In general, the specific surface area increases with decreasing particle size and increasing porosity. Even the same rock material with different grain size fraction, same weight and volume has varied specific surface areas and therefore the varied surface activity and adsorption volume. In other words, a rock sample with the same volume and weight shows different surface areas for different particle sizes.

The specific surface area of the grain size fraction 2-4 mm is 0.11 m^2/g, of the grain size fraction 10-20 mm is 0.14 m^2/g, and of the core slice is 0.12 m^2/g. For the grain size fraction < 0.065 mm, the specific surface area was measured twice and the mean value 0.97±0.02 m^2/g (n=2) is significantly higher. The specific surface area of the grain size fraction 0.065-0.2 mm was measured four times. The mean value of the four measurements is 0.22±0.02 m^2/g (n=4).

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Table 4-1: Results of the Specific Surface Area Measured at Different Grain Size Fractions by the BET Method

Hydro Lab-No.	Grain size fraction	Specific surface area (m^2/g)
	<0.065 mm	0.96 / 0.98
344886	0.065-0.2 mm	0.22 (0.21 / 0.23 / 0.22 / 0.23)
344887	2-4 mm	0.11
344888	10-20 mm	0.14
344889	Core slice (3 cm thickness, 6 cm diameter)	0.12

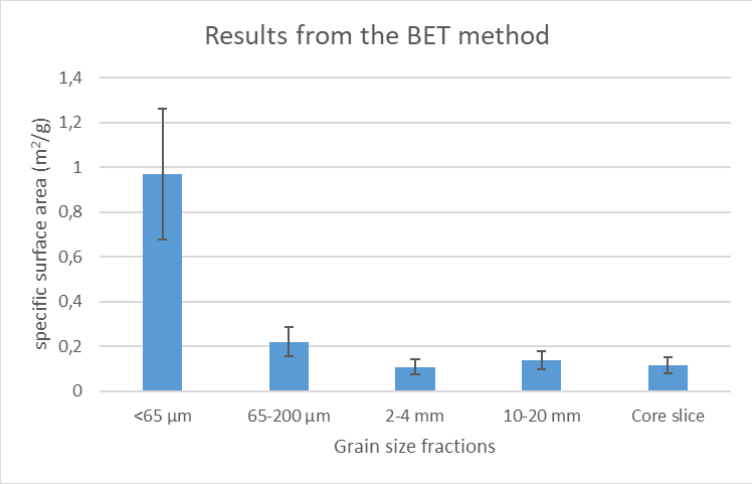


Figure 4-1: The Specific Surface Area Measured at Different Grain Size Fractions and at A Core Slice Using the BET Method

4.2 CATION Exchange Capacity

4.2.1 CEC_{Ni}

The results from the CEC experiments performed on the smallest grain size fraction (0.065-0.2 mm) are summarized in Table 4-2, the middle grain size fraction (2-4 mm) in Table 4-3 and the largest grain size fraction (10-20 mm) in Table 4-4.

The initial Ni concentration of the Ni-ethylenediamine stock solution was 33.1±3.0 mg/l. Each experiment was performed in duplicate indicated as No. 1 and No. 2 in Table 4-2 to Table 4-4.

The pH value and redox potential were measured at the beginning of the experiment right after adding the experimental solution to the grinded rock material and after the experiment (after 48 h, 96 h, 71 days).

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During the experiments the pH value increased from 8.83-9.28 to 9.43-9.58 for the grain size range 0.065-0.2 mm, from 8.59-8.98 to 9.47-10.33 for the grain size range 2-4 mm and from 9.11-9.22 to 9.61-10.51 for the grain size range 10-20 mm.

Table 4-2: Element Concentrations of the Final CEC Experiment Solutions Using Different Rock / Test Solution Mass Ratios Conducted with the Smallest Grain Size Fraction (0.065-0.2 mm) After 48 h
Sample Name IG_BH04_SA001 65-200 µm

Hydro Lab.-No.		350529	350530	350531	350532	350533	350534	350535	350536	350537
Sample		1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock / Test Solution Mass Ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
Rock mass	g	39.999	39.997	20.004	20.007	9.998	9.999	10	10.002	0
Solution mass	g	40	40	40	40	40	40	100	100	70
pH value start		8.89	8.83	9.25	9.28	9.25	9.25	9.2	9.24	8.51
pH value end		9.57	9.58	9.48	9.5	9.43	9.46	9.46	9.44	8.51
Redox potential start	mV	55	50	61	65	58	58	58	47	95
Redox potential end	mV	96	41	69	73	36	55	50	60	95
Nickel (Ni^{2+})	mg/l	3.08	3.5	10.3	11.5	16.6	18.8	24.1	24.7	32.9
Barium (Ba^{2+})	mg/l	0.009	0.007	0.015	0.09	0.006	0.015	0.008	0.009	0.081
Strontium (Sr^{2+})	mg/l	0.04	0.04	0.05	0.06	0.04	0.04	0.03	0.03	0.04
Aluminum (Al^{3+})	mg/l	0.67	0.65	1.51	3.66	0.48	1.32	0.37	0.37	0.59
Iron tot. (Fe_{tot})	mg/l	26	25.2	21.3	21.2	5.28	5.33	< 0.01	< 0.01	< 0.01
Iron-II (Fe^{2+})	mg/l	0.05	0.09	0.98	4.12	0.01	0.75	< 0.01	0.02	< 0.01
Iron-III (Fe^{3+})	mg/l	26	25.1	20.3	17.1	5.27	4.58	< 0.01	< 0.01	< 0.01
Sodium (Na^{+})	mg/l	17	17	9.3	9.3	4.9	5.1	2.4	2.4	1
Potassium (K^{+})	mg/l	37	28	18	19	10	10	4.8	4.8	2.6
Calcium (Ca^{2+})	mg/l	6.57	5.78	14.5	14.2	9.71	8.64	10.7	7.68	0.897
Magnesium (Mg^{2+})	mg/l	1.9	5.8	4.7	3.9	2.4	1.6	0.36	1.5	< 0.2
Ammonium (NH_4^{+})	mg/l	0.89	0.91	0.26	0.24	0.17	0.16	0.14	0.13	0.10
Silicon (Si^{4+})	mg/l	3.7	4.1	3.3	5.7	2.7	2.8	1.9	1.9	< 0.1

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Table 4-3: Element Concentrations of the Final CEC Experiment Solutions Using Different Rock / Test Solution Ratios Conducted with the Medium Grain Size Fraction (2-4 mm) After 96 h Sample Name IG_BH04_SA001 2-4 mm

Hydro Lab.-No.		352653	352654	352655	352656	352657	352658	352659	352660	352662
Sample		1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock/ Test Solution Mass Ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
Rock mass	g	39.988	40.005	19.999	19.998	10.006	10.005	10.008	10.004	0
Solution mass	g	39.063	39.304	38.778	38.743	33.898	39.183	98.86	98.807	73.673
pH value start		8.59	8.79	8.94	8.98	8.93	8.93	8.89	8.86	8.56
pH value end		10.31	10.33	10.14	10.18	9.95	10.01	9.48	9.47	8.42
Redox potential start	mV	69.4	64.0	47.8	46.9	71.6	64.0	69.4	78.5	77
Redox potential end	mV	-35	-39.6	-17.6	-20.6	0.7	-4.1	27.6	28.4	108.5
Nickel (Ni^{2+})	mg/l	3.75	3.77	4.50	3.59	5.40	4.25	18.3	18.1	29.6
Barium (Ba^{2+})	mg/l	1.25	1.01	0.472	0.326	0.022	0.03	0.03	0.029	0.292
Strontium (Sr^{2+})	mg/l	0.25	0.22	0.12	0.13	0.06	0.09	0.02	0.01	< 0.001
Aluminium (Al^{3+})	mg/l	110	99	44.2	45.8	18.5	24.6	1.08	1.07	0.005
Iron tot. (Fe_{tot})	mg/l	200	125	58.3	64.8	18.5	30.7	3.53	2.17	< 0.01
Iron-II (Fe^{2+})	mg/l	154	125	58.3	64.8	18.5	30.7	0.31	0.3	< 0.01
Iron-III (Fe^{3+})	mg/l	46	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	3.22	1.86	< 0.01
Sodium (Na^{+})	mg/l	65	44	28	24	19	18	4.5	2.9	0.55
Potassium (K^{+})	mg/l	59	90	41	56	31	48	10	9.7	5
Calcium (Ca^{2+})	mg/l	20	37	6.3	14	7.8	18	2.9	2.8	3.1
Magnesium (Mg^{2+})	mg/l	340	72	9.7	26	6.9	21	0.51	0.53	0.58
Ammonium (NH_4^{+})	mg/l	2.3	2.2	1.3	1.3	0.77	0.92	0.22	0.21	0.21
Silicon (Si^{4+})	mg/l	31.1	16.8	17.3	14.4	10	7.8	2.8	2.7	< 0.5

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Table 4-4: Element Concentrations of the Final CEC Experiment Solutions Using Different Rock / Test Solution Ratios Conducted with the Largest Grain Size Fraction (10-20 mm) After 71 days Sample Name IG_BH04_SA001 10-20 mm

Hydro Lab.-No.		356425	356426	356427	356428	356429	356430	356431	356432	356433
Sample		1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock/ Test Solution Mass Ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
Rock mass	g	59.529	59.803	29.985	30.556	15.354	14.872	10.202	10.375	0
Solution mass	g	58.625	59.025	59.668	59.32	59.198	58.836	98.453	98.971	74.364
pH value start		9.22	9.18	9.14	9.14	9.11	9.15	9.14	9.15	9.14
pH value end		10.51	10.30	10.42	10.32	9.67	10.22	9.61	9.96	8.92
Redox potential start	mV	9.1	24.1	5.6	19.1	15.8	23.2	7.3	14.8	9.9
Redox potential end	mV	-88.0	-104.7	-88.7	-85.6	-83.1	-78.7	-68.6	-74.4	-24.2
Nickel (Ni^{2+})	mg/l	4.20	3.46	2.80	2.54	2.71	1.97	11.8	18.5	30.8
Barium (Ba^{2+})	mg/l	2.18	2.04	0.804	0.598	0.114	0.268	0.09	0.072	0.13
Strontium (Sr^{2+})	mg/l	1.03	0.77	0.38	0.32	0.05	0.09	0.06	0.04	0
Aluminium (Al^{3+})	mg/l	241	165	89.5	87.5	12.2	23.5	9.25	3.25	0.003
Iron tot. (Fe_{tot})	mg/l	82.6	87.6	33.0	28.8	5.38	11.1	2.13	0.63	< 0.001
Iron-II (Fe^{2+})	mg/l	82.6	79.7	33.0	28.8	2.66	10.2	2.13	0.60	< 0.001
Iron-III (Fe^{3+})	mg/l	< 0.1	7.9	< 0.1	< 0.1	2.72	0.9	< 0.01	0.03	< 0.001
Sodium (Na^{+})	mg/l	86.7	87.9	59.2	55	28.1	34.3	19.0	5.9	0.28
Potassium (K^{+})	mg/l	7.3	7.9	8.2	8.4	11.2	12.6	7.5	6.5	0.99
Calcium (Ca^{2+})	mg/l	62.5	45.9	22.6	21.4	3.25	5.20	3.80	3.54	<0.1
Magnesium (Mg^{2+})	mg/l	24.0	21.3	7.74	10.1	0.65	3.15	0.77	< 0.1	0.17
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Silicon (Si^{4+})	mg/l	49.0	44.9	32.0	29.6	11.7	14.7	8.8	5.3	< 0.1

Figure 4-2 shows the nickel concentrations and calculated CEC_{Ni} values during the course of the experiment for the largest grain size (10-20 mm) versus experimental time. The nickel concentration in the solution decreases with the experimental time, because the nickel exchanges with the cations on the rock surface, as a result the CEC value increases with time. The time-series show that the Ni-equilibration time depends on the rock/ test solution ratio. The experiments with high rock/ test solution ratios (1:1, 1:2) show a faster Ni-equilibrium state than those with lower ratios (1:4, 1:10). As a result, the nickel concentration is nearly zero or levels off at a constant low value, confirming the equilibrium within the experimental time (Figure 4-2). Because less nickel in the solution can be exchanged in the 1:1 experiments compared to the rock surface, an equilibrium state was reached after 21 days. In the 1:10

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experiments, on the other hand, more nickel in the solution is available for cation exchange compared to the rock surface, so that a state of equilibrium was nearly reached after 62 and 71 days, respectively.

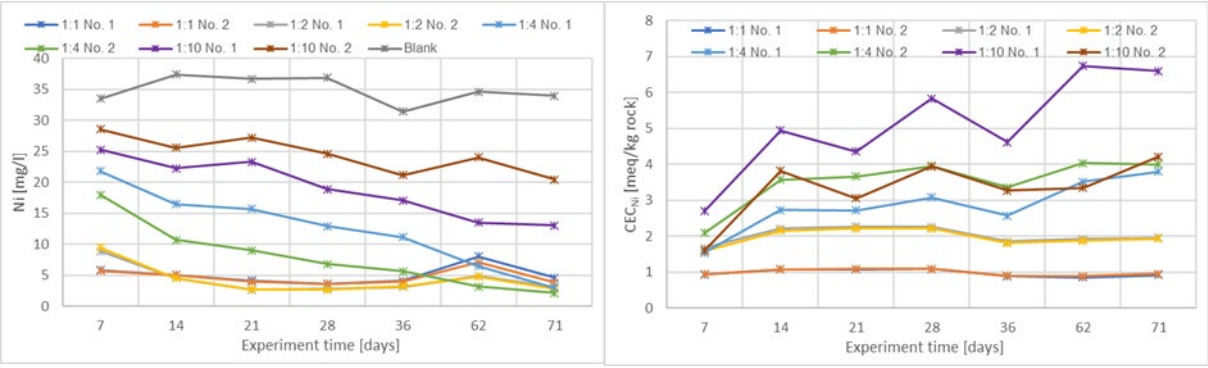


Figure 4-2: Nickel Concentration (Left) and CEC_{Ni} Values of the Largest Grain Size Fraction 10-20 mm Measurements over Experimental time. Experiments run 71 days

Table 4-5 and Figure 4-3 give an overview of the calculated mean CEC values based on the nickel concentrations. Depending on the rock/ test solution ratio and the grain size fraction, the CEC value varies from 0.82 to 5.99 meq/kg rock. The results of the duplicates of the individual experiments agree well with each other (Table 4-5). This shows that the experiments are comparable and reproducible. An exception is the 1:10 experiment of grain size 10-20 mm, where the replicates differ from each other. The reason is probably that the 10 g rock weight corresponds to only a few rock grains and thus the reactive surface can differ significantly between the 2 replicates. The results also show that the CEC value increases as the volume of solution increases in relation to the mass of rock. At a rock/ test solution ratio of 1:1, CEC_{Ni} values range from 0.82 to 1.02 meq/kg rock, whereas at a rock/ test solution ratio of 1:10, CEC_{Ni} values range from 2.79 to 5.99 meq/kg rock (see Table 4-5).

Table 4-5: Calculated CEC_{Ni} Values, Mean CEC_{Ni} Values and Standard Deviation from the CEC Experiments with Three Grain Size Fraction 0.065-0.2 mm, 2-4 mm and 10-20 mm

Rock / Test Solution Mass Ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10
0.065-0.2 mm	CEC_{Ni} (meq/kg rock)	1.02	1.00	1.54	1.46	2.22	1.92	3.00	2.79
	Mean CEC_{Ni} (meq/kg rock)	1.01±0.01		1.50±0.06		2.07±0.21		2.90±0.14	
2-4 mm	CEC_{Ni} (meq/kg rock)	0.82	0.82	1.57	1.63	2.64	3.21	3.37	3.43
	Mean CEC_{Ni} (meq/kg rock)	0.82±0.003		1.60±0.04		2.93±0.40		3.40±0.05	
10-20 mm	CEC_{Ni} (meq/kg rock)	0.83	0.85	1.77	1.74	3.43	3.61	5.99	3.83
	Mean CEC_{Ni} (meq/kg rock)	0.84±0.02		1.75±0.02		3.52±0.13		4.91±1.52	

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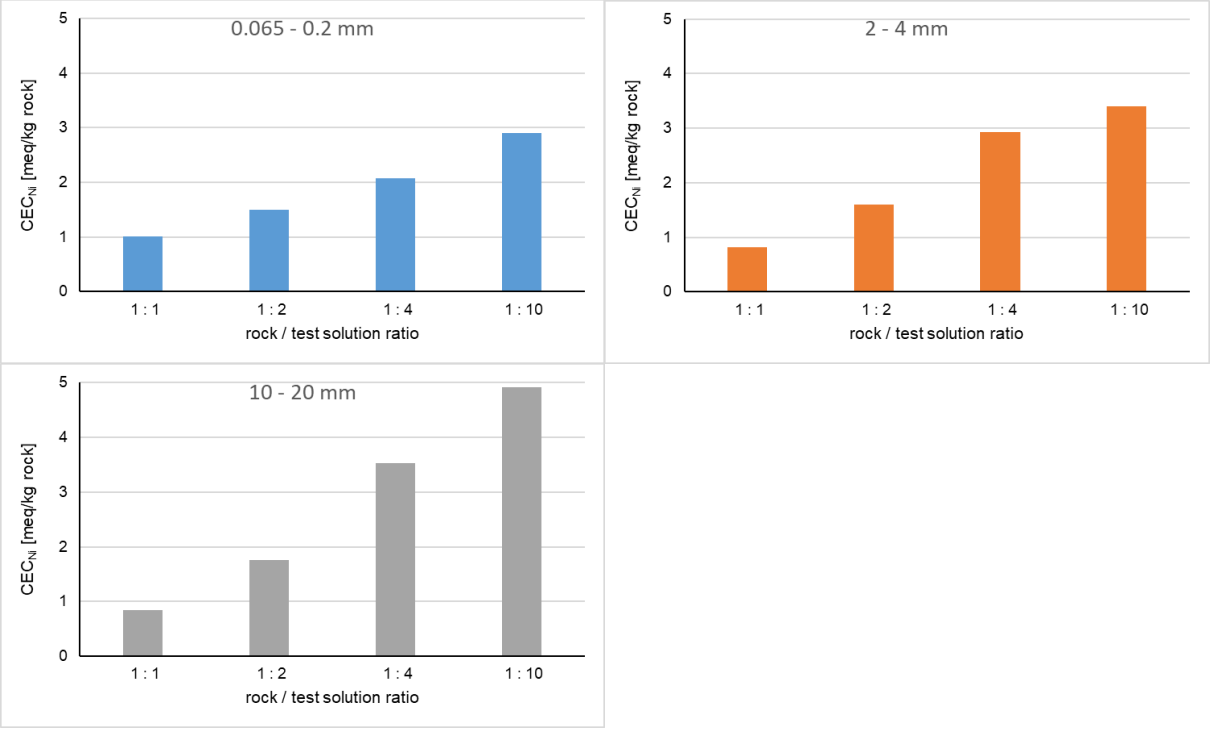


Figure 4-3: Results of the CEC_{Ni} Values of the Different Grain Size Fractions (0.065-0.2 mm, 2-4 mm, 10-20 mm) and Rock/Test Solution Mass Ratios Calculated by Nickel Concentration

4.2.2 CEC_{cation}

To verify the measured CEC values calculated by nickel concentration measurement, the concentrations of major and trace cations (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+ Al^{3+}, Ba^{2+}, Sr^{2+}, Fe, Si^{4+}) in the final extract solution are measured by ion chromatography and AAS. The results of concentrations of each cation and the CEC calculations based on the sum of cations ($CEC_{cations}$) are summarized in Table 4-6, Table 4-7 and Table 4-8. The results of the CEC calculations based on the sum of all the cations are summarized in Table 4-9 and Figure 4-4.

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Table 4-6: CEC Values of Each Cation (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe, Si^{4+}) and CEC Values of the Sum of Cations of the Smallest Grain Size Fraction (0.065-0.2 mm). Sample Name IG_BH04_SA001 0.065-0.2 mm. All Values in meq/kg rock

Hydro Lab.-No.	350529	350530	350531	350532	350533	350534	350535	350536
Sample	1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2
Rock/Test Solution Mass Ratio	1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10
Nickel (Ni^{2+})	1.02	1.00	1.54	1.46	2.22	1.92	3.00	2.79
Barium (Ba^{2+})	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Strontium (Sr^{2+})	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Aluminium (Al^{3+})	< 0.01	< 0.01	0.14	0.45	< 0.01	0.21	< 0.01	< 0.01
Iron tot. (Fe_{tot})	0.93	0.90	1.53	1.52	0.76	0.76	< 0.1	< 0.1
Iro-II (Fe^{2+})	0.002	0.003	0.07	0.29	< 0.01	0.11	< 0.01	< 0.01
Iro-III (Fe^{3+})	0.93	0.90	1.45	1.22	0.76	0.66	< 0.1	< 0.1
Sodium (Na^+)	0.70	0.70	0.72	0.72	0.68	0.71	0.61	0.61
Potassium (K^+)	0.88	0.65	0.79	0.84	0.76	0.76	0.56	0.56
Calcium (Ca^{2+})	0.28	0.24	1.36	1.33	1.76	1.55	4.89	3.38
Magnesium (Mg^{2+})	0.16	0.48	0.77	0.64	0.79	0.53	0.30	1.23
Ammonium (NH_4^+)	0.04	0.04	0.02	0.02	0.02	0.01	0.02	0.02
Silicon (Si^{4+})	0.53	0.58	0.94	1.62	1.54	1.60	2.71	2.71
$CEC_{main\text{-}cations}$ (Na^+, K^+, Ca^{2+}, Mg^{2+})	2.02	2.07	3.64	3.53	3.99	3.54	6.36	5.79
$CEC_{cations}$ (all cation/sum)	3.52	3.60	6.26	7.14	6.26	6.13	8.91	8.33

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Table 4-7: CEC Values of Each Cation (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe, Si^{4+}) and CEC Values of the Sum of Cations of the Medium Grain Size Fraction (2-4 mm). Sample Name IG_BH04_SA001 2-4 mm. All Values in meq/kg rock

Hydro Lab.-No.	352653	352654	352655	352656	352657	352658	352659	352660
Sample	1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2
Rock/ Test Solution Mass Ratio	1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10
Nickel (Ni^{2+})	0.82	0.82	1.57	1.63	2.64	3.21	3.37	3.43
Barium (Ba^{2+})	0.02	0.01	0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Strontium (Sr^{2+})	0.006	0.005	0.005	0.006	0.005	0.008	0.005	0.002
Aluminium (Al^{3+})	7.96	7.21	6.35	6.57	4.64	7.14	0.78	0.77
Iron tot. (Fe_{tot})	7.00	4.40	4.05	4.50	2.24	4.31	1.25	0.77
Iron-II (Fe^{2+})	5.39	4.40	4.05	4.50	2.24	4.31	0.11	0.11
Iron-III (Fe^{3+})	1.61	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	1.14	0.66
Sodium (Na^+)	2.75	1.87	2.34	2.01	2.77	3.03	1.85	1.16
Potassium (K^+)	1.47	2.26	2.03	2.77	2.68	4.80	2.50	2.42
Calcium (Ca^{2+})	0.94	1.82	0.57	1.30	1.25	3.46	1.25	1.22
Magnesium (Mg^{2+})	3.20	5.82	1.57	4.17	1.95	6.63	0.50	0.51
Ammonium (NH_4^+)	0.12	0.11	0.12	0.12	0.11	0.16	0.03	0.03
Silicon (Si^{4+})	4.33	2.35	4.78	3.97	4.83	4.35	3.94	3.80
$CEC_{main\text{-}cations}$ (Na^+, K^+, Ca^{2+}, Mg^{2+})	8.37	11.76	6.51	10.25	8.65	17.92	6.09	5.32
$CEC_{cations}$ (all cation/sum)	27.79	25.85	21.82	25.43	20.47	33.88	12.09	10.67

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Table 4-8: CEC Values of Each Cation (Na^+, K^+, Ca^{2+}, Mg^{2+}, NH_4^+, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe, Si^{4+}) and CEC Values of the Sum of Cations of the Largest Grain Size Fraction (10-20 mm). Sample Name IG_BH04_SA001 10-20 mm. All Values in meq/kg rock

Hydro Lab.-No.	356425	356426	356427	356428	356429	356430	356431	356432
Sample	1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2
Rock/ Test Solution Mass Ratio	1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10
Nickel (Ni^{2+})	0.83	0.85	1.77	1.74	3.43	3.61	5.99	3.83
Barium (Ba^{2+})	0.06	0.06	0.04	0.03	< 0.01	0.02	< 0.01	< 0.01
Strontium (Sr^{2+})	0.03	0.02	0.02	0.02	< 0.01	0.01	0.02	0.01
Aluminium (Al^{3+})	7.51	5.15	5.65	5.39	1.49	2.95	2.91	1.01
Iron tot. (Fe_{tot})	2.57	2.74	2.08	1.77	0.66	1.39	0.67	0.20
Iron-II (Fe^{2+})	2.57	2.49	2.08	1.77	0.33	1.28	0.67	0.19
Iron-III (Fe^{3+})	< 0.01	0.25	< 0.01	< 0.01	0.33	0.11	< 0.01	< 0.01
Sodium (Na^+)	2.69	2.74	3.72	3.37	3.40	4.27	5.90	1.75
Potassium (K^+)	0.20	0.22	0.45	0.46	1.25	1.46	2.05	1.72
Calcium (Ca^{2+})	1.95	1.43	1.43	1.32	0.40	0.65	1.20	1.10
Magnesium (Mg^{2+})	0.74	0.66	0.48	0.61	0.06	0.37	0.19	< 0.01
Ammonium (NH_4^+)	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Silicon (Si^{4+})	1.53	1.40	2.02	1.82	1.43	1.84	2.77	1.65
$CEC_{main\text{-}cations}$ (Na^+, K^+, Ca^{2+}, Mg^{2+})	5.58	5.04	6.08	5.75	5.11	6.75	9.34	4.52
$CEC_{cations}$ (all cation/sum)	17.29	14.42	15.89	14.79	8.69	12.96	15.70	7.37

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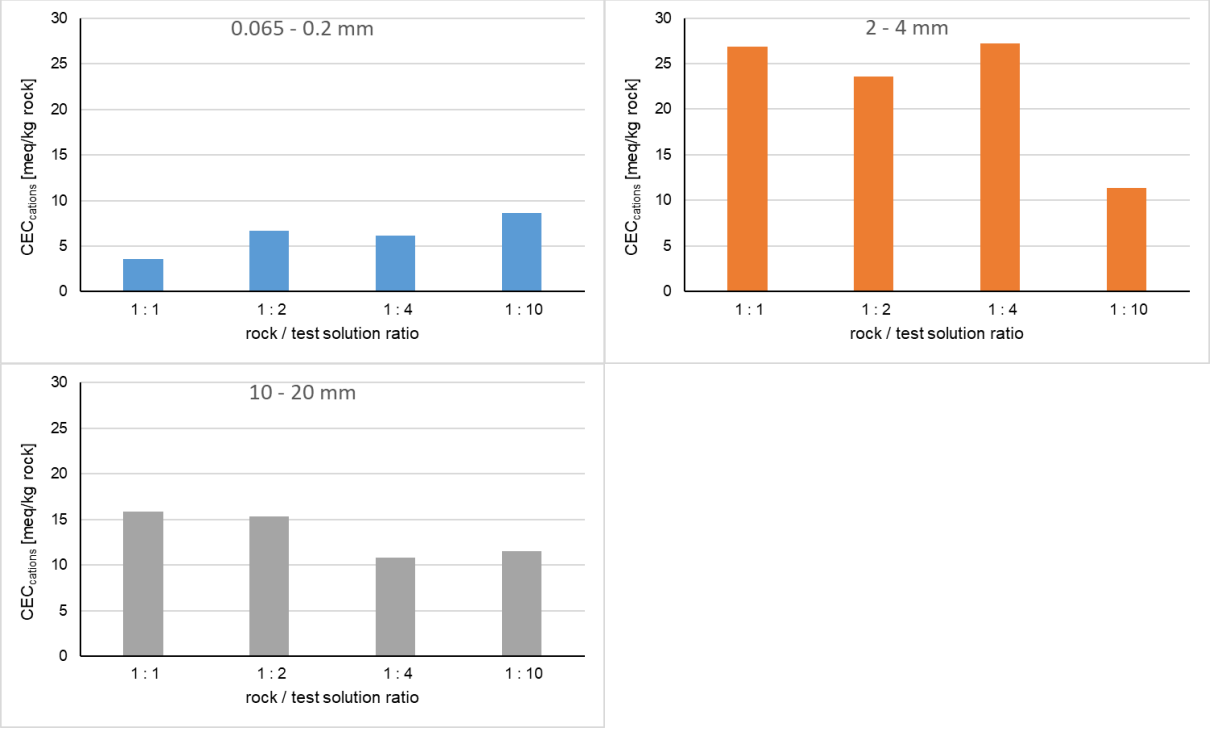


Figure 4-4: Results of the $CEC_{Cations}$ Values of the Different Grain Size Fractions (0.065-0.2 mm, 2-4 mm, 10-20 mm) and Rock / Test Solution Ratios Calculated by the Sum of Concentrations of All the Cations in the Solution

Table 4-9: Calculated CEC_{cation} Values (Mean CEC-Cation Values) from the CEC Experiments with the Three Grain Size Fractions 0.065-0.2 mm, 2-4 mm and 10-20 mm

Rock / Test Solution Mass Ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10
0.065-0.2 mm	CEC_{cation} (meq/kg rock)	3.52	3.60	6.26	7.14	6.26	6.13	8.91	8.33
	Mean CEC_{cation} (meq/kg rock)	3.56±0.06		6.70±0.62		6.19±0.09		8.62±0.41	
2-4 mm	CEC_{cation} (meq/kg rock)	27.79	25.85	21.82	25.43	20.47	33.88	12.09	10.67
	Mean CEC_{cation} (meq/kg rock)	26.82±1.37		23.63±2.55		27.18±9.48		11.38±1.00	
10-20 mm	CEC_{cation} (meq/kg rock)	17.29	14.42	15.89	14.79	8.69	12.96	15.70	7.37
	Mean CEC_{cation} (meq/kg rock)	15.85±2.03		15.34±0.78		10.82±3.02		11.54±5.89	

The results show that the CEC_{cation} values calculated by the sum of all the cations (Table 4-9) are 2 to 33 times higher (3.52-33.88 meq/kg rock) than the CEC_{Ni} values calculated from the nickel concentrations. The CEC_{cation} values significantly increased during the experiment time of the largest grain size fraction (10-20 mm) (Appendix Tab. A-3 to A-9). The experimental solutions showed very high cation concentrations at the end of the experiment - probably due to abrasion and erosion and water-rock

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interactions of the rock caused by end-over-end shaking of the experiments - leading to high calculated CEC_{cation} values.

4.2.3 "Zero Exchange Experiment"

To evaluate the influence of possible disturbing effects, which lead to the overestimation of exchanged cations, aqueous extraction experiments, following the same procedures as the Ni-exchange experiments were conducted without the addition of Ni.

Potential processes, which lead to the entrance of cations are:

- Crushing and grinding of the rock material liberates fluids trapped in mineral fluid inclusions.
- During leaching, all salts in the rock material will become dissolved, in addition to the limited dissolution of the original mineral assemblage. The mineralization of a leach solution is therefore the sum of: (i) the constituents originally dissolved in the porewater, (ii) the constituents present in fluid inclusions, and (iii) water-rock interactions during the leaching process. Thus, aqueous leach solutions represent a complex composition in rocks with abundant fluid inclusions and/or rapidly reacting mineral phases.
- Fluid inclusions, a high salt content in porewater and the presence of carbonates, such as calcite and dolomite (Ca^{2+} and Mg^{2+}) and other minerals, for example, feldspars (K^+, Na^+ and Ca^{2+}) in the rock material can induce an overestimated $CEC_{cations}$ value. Carbonates with concentrations > 1 wt.% are not expected in crystalline rocks from the Ignace site.
- K^+ and Na^+ cations originating from porewater with a high salt content dissolve in the Ni-en complex solution. Hence, the measured cation concentration has to be corrected for the porewater composition (Waber et al., 2003). Therefore, a blank experiment has to be realized parallel to the CEC experiments. Exchangeable cations that are not analyzed also may lead to an underestimated CEC value (e.g. NH_4^+, H^+) (Waber et al., 2003).

To evaluate these effects, a "zero exchange experiment" was conducted. The aim was to determine the basic dissolution of elements from the rock by rock-water interaction processes during the experiments and to use this as a basic value for the CEC calculation ($CEC_{cation\ corr.} = CEC_{cation}-CEC_{cation\ zero}$; $CEC_{cation\ zero}$ is the CEC measured by the "zero exchange experiment", $CEC_{cation\ corr.}$ is the CEC after the correction). For this purpose, rock material was added to ultrapure water following the procedures for the normal CEC-experiment (same grain size for a certain period of time: 0.065-0.2 mm for 48 h, 2-4 mm for 96 h, 10-20 mm for app. 71 d) for one rock / test solution ratio (see Table 4-10). All "zero exchange experiments" were sampled and analysed followed the same protocols as the CEC experiments.

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Table 4-10: Overview of the Experimental Set-ups of the "Zero Exchange Experiment"

Grain Size Fraction	Rock/ Test Solution Mass Ratio	Reaction Time	Mass (rock material) (g)	Mass (solution) (g)
0.065-0.2 mm	1:2	48 h	20	40
0.065-0.2 mm Blank	-	48 h	0	40
2-4 mm	1:1	96 h	40	40
2-4 mm Blank	-	96 h	0	40
10-20 mm	1:1	71 days	60	60
10-20 mm Blank	-	71 days	0	60

It was shown that shaking the rock in the bottle caused massive abrasion of the rock particles (see Figure 4-5 and Table 4-11). The $CEC_{cation\ zero}$ values without any Ni-cation-exchange range from1.85 to 16.17 meq/kg rock (Table 4-11). This resulted in an increased dissolution of elements, including cations, as well as a partial fragmentation of the rock particles (suspension) and finally to enhanced rock-water interactions. This led to significantly higher cation concentrations than expected and significantly higher CEC values than those determined by nickel consumption (see Figure 4.6). In addition, the erosion of the rock particles caused an increase in the specific surface area potentially available for cation exchange. Therefore, for the future CEC experiments, we recommend avoiding overhead shaking and instead carefully shaking or agitating the bottles on a regular basis in order to ensure mixing during the experiment without forcing strong erosion.



Figure 4-5: Experimental Bottles with the Grain Size Fraction (2-4 mm) Experiments Immediately After Start (Left) and After 96 h Shaking (Right)

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Table 4-11: Calculated $CEC_{cation\ zero}$ Values (Mean CEC-Cation Values) Based on all Cation Concentrations from the Zero Exchange Experiments with the Three Grain Size Fraction 0.065-0.2 mm, 2-4 mm and 10-20 mm after Experimental Time. The CEC_{cation} Values and the $CEC_{cation\ coor.}$ Values After Correction with the $CEC_{cation\ zero}$ Values

Grain Size Fraction		$CEC_{cation\ zero}$ (Zero Exchange Experiment)				CEC_{cation}		$CEC_{cation\ corr.}$	
		1:1	1:1	1:2	1:2	1:1	1:2	1:1	1:2
0.065-0.2 mm	(meq/kg rock)	-	-	3.16	3.07	-	-	-	-
	Mean (meq/kg rock)	-		3.12±0.06		3.56	6.70	-	3.58
2-4 mm	(meq/kg rock)	4.01	1.85	-	-	-	-	-	-
	Mean (meq/kg rock)	2.93±1.53		-		26.82	23.63	23.89	-
10-20 mm	(meq/kg rock)	14.31	16.17	-	-	-	-	-	-
	Mean (meq/kg rock)	15.24±1.31		-		15.85	15.34	0.61	-

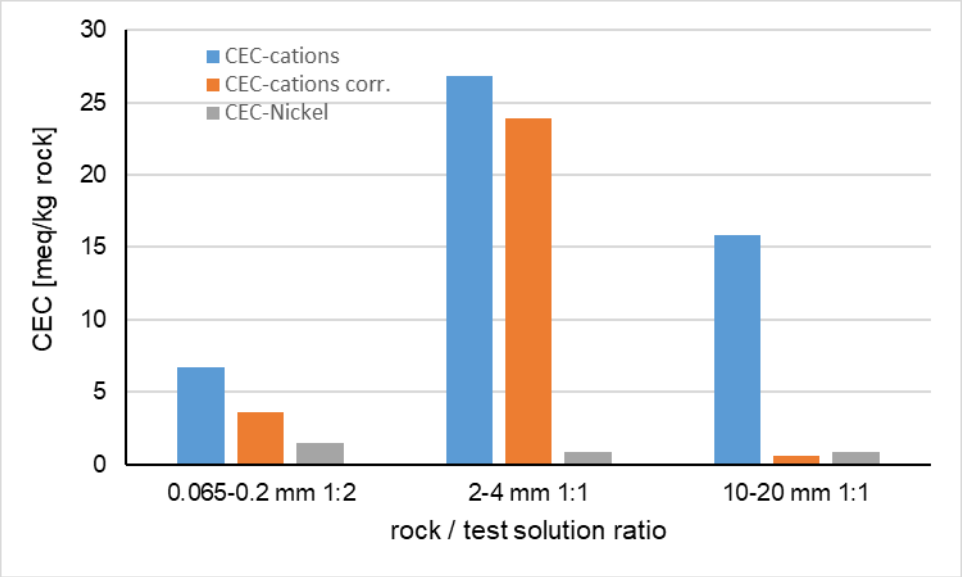


Figure 4-6: Results of the CEC Values of the Different Grain Size Fractions (0.065-0.2 mm, 2-4 mm, 10-20 mm) and Rock / Test Solution Ratios Calculated by the Ni-Concentration (CEC_{Nickel}, Grey Bar), the Sum of All Cations in the Solution (CEC_{cation}, Blue Bar) and Corrected with the Zero Exchange Experiment Values ($CEC_{cation\ corr.}$; Orange Bar)

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5.0 Summary

The investigations of the surface area by the BET method and the cation exchange capacity conducted on a crystalline core sample from the borehole IG_BH04 (sample ID: IG_BH04_SA001) can be summarized as follows:

- In general, the specific surface area increases with decreasing particle size.
- The specific surface area of the grain size fraction< 0.065 mm is 0.97±0.02 m^2/g (n=2).
- The specific surface area of the grain size fraction 0.065-0.2 mm is 0.22±0.02 m^2/g (n=4).
- The specific surface area of the grain size fraction 2-4 mm is 0.11 m^2/g.
- The specific surface area of the grain size fraction 10-20 mm is 0.14 m^2/g.
- The specific surface area of the core slice (3 cm thickness and 6cm diameter) is 0.12 m^2/g.
- Depending on the rock/ test solution ratio and the grain size fraction, the CEC_{Ni} values vary from 0.82 to 5.99 meq/kg rock.
- The results also show that the CEC_{Ni} value increases as the volume of test solution increases in relation to the mass of rock.
- The CEC_{cation} values calculated from the sum of all cations (Na^+, K^+, Mg^{2+}, Ca^{2+}, $NH4^+$, Al^{3+}, Ba^{2+}, Sr^{2+}, Fe_{tot}, Si^{4+}) vary between 3.52 and 33.88 meq/kg rock depending on the rock/ test solution ratio and are of a factor 2 to 33 (mean 9) higher than the CEC_{Ni} values calculated from the nickel concentrations.
- CEC_{cation} values significantly increased during the experiment time of the largest grain size fraction (10-20 mm).

It can be summarized that the CEC_{Ni} value depends on or is affected by various factors and processes, which include, e.g., the release of porewater, the opening of fluid inclusions, the grain size, the rock/test solution ratio, the mineralogical composition of the sub-samples and the experimental setup.

Besides the calculation of the CEC value from the nickel concentration, the calculation from the sum of the cations ($CEC_{cations}$) is used to check the CEC_{Ni} values. Here it is important to note which cations (elements) are included in the calculation, as this can change the CEC value significantly.

It was also shown that the grain size and consequently the reactive surface as well as the rock / test solution ratio have a large influence on the measured CEC value.

Consequently, the CEC_{Ni} values should be used, but always taking into account the previously discussed factors and checking the CEC_{cation} values in order to know all possible influencing factors and to take them into account if relevant.

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Appendix A

Analytical Raw Data

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CEC experiment 0.065-0.2 mm

Appendix Table A-1: Element concentrations of the final CEC experiment solutions with the smallest grain size fraction (0.065-0.2 mm) after 48 h. Sample name IG_BH04_SA001 0.065-0.2 mm.

Hydro Lab.-No.		350529	350530	350531	350532	350533	350534	350535	350536	350537
Sample		1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock/Water ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
m (rock)	g	39.999	39.997	20.004	20.007	9.998	9.999	10	10.002	0
m (solution)	g	40	40	40	40	40	40	100	100	70
pH-value start		8.89	8.83	9.25	9.28	9.25	9.25	9.2	9.24	8.51
pH-value end		9.57	9.58	9.48	9.5	9.43	9.46	9.46	9.44	8.51
Redox potential start	mV	55	50	61	65	58	58	58	47	95
Redox potential end	mV	96	41	69	73	36	55	50	60	95
Nickel (Ni^{2+})	mg/l	3.08	3.5	10.3	11.5	16.6	18.8	24.1	24.7	32.9
Barium (Ba^{2+})	mg/l	0.009	0.007	0.015	0.09	0.006	0.015	0.008	0.009	0.081
Strontium (Sr^{2+})	mg/l	0.04	0.04	0.05	0.06	0.04	0.04	0.03	0.03	0.04
Aluminum (Al^{3+})	mg/l	0.67	0.65	1.51	3.66	0.48	1.32	0.37	0.37	0.59
Iron tot. (Fe_{tot})	mg/l	26	25.2	21.3	21.2	5.28	5.33	< 0.01	< 0.01	< 0.01
Iron-II (Fe^{2+})	mg/l	0.05	0.09	0.98	4.12	0.01	0.75	< 0.01	0.02	< 0.01
Iron-III (Fe^{3+})	mg/l	26	25.1	20.3	17.1	5.27	4.58	< 0.01	< 0.01	< 0.01
Sodium (Na^{+})	mg/l	17	17	9.3	9.3	4.9	5.1	2.4	2.4	1
Potassium (K^{+})	mg/l	37	28	18	19	10	10	4.8	4.8	2.6
Calcium (Ca^{2+})	mg/l	6.57	5.78	14.5	14.2	9.71	8.64	10.7	7.68	0.897
Magnesium (Mg^{2+})	mg/l	1.9	5.8	4.7	3.9	2.4	1.6	0.36	1.5	< 0.2
Ammonium (NH_4^{+})	mg/l	0.89	0.91	0.26	0.24	0.17	0.16	0.14	0.13	0.1
Silicon (Si^{4+})	mg/l	3.7	4.1	3.3	5.7	2.7	2.8	1.9	1.9	< 0.1
Hydrogencarbonate (HCO_3^{-})	mg/l	36.4	38.3	34.4	6.1	21.2	21.3	14.3	14.5	13.3
Chloride (Cl^{-})	mg/l	22	14.	6.5	7.4	4.	4	2	2	2.6
Sulfate (SO_4^{2-})	mg/l	1.2	0.89	0.69	0.6	0	0.58	0	0	1.1
Nitrate (NO_3^{-})	mg/l	68	68	66	750	67	70	68	67	67
Fluoride (F^{-})	mg/l	0.43	0.38	0.2	0.22	0.12	0.12	0	0	0

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CEC experiment 2-4 mm

Appendix Table A-2: Element concentrations of the final CEC experiment solutions with the middle grain size fraction (2-4 mm) after 96 h. Sample name IG_BH04_SA001 2-4 mm.

Hydro Lab.-No.		352653	352654	352655	352656	352657	352658	352659	352660	352662
Sample		1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock/Water ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
m (rock)	g	39.988	40.005	19.999	19.998	10.006	10.005	10.008	10.004	0
m (solution)	g	39.063	39.304	38.778	38.743	33.898	39.183	98.86	98.807	73.673
pH-value start		8.59	8.79	8.94	8.98	8.93	8.93	8.89	8.86	8.56
pH-value end		10.31	10.33	10.14	10.18	9.95	10.01	9.48	9.47	8.42
Redox potential start	mV	69.4	64.0	47.8	46.9	71.6	64.0	69.4	78.5	77
Redox potential end	mV	-35	-39.6	-17.6	-20.6	0.7	-4.1	27.6	28.4	108.5
Nickel (Ni^{2+})	mg/l	3.75	3.77	4.50	3.59	5.40	4.25	18.3	18.1	29.6
Barium (Ba^{2+})	mg/l	1.25	1.01	0.472	0.326	0.022	0.03	0.03	0.029	0.292
Strontium (Sr^{2+})	mg/l	0.25	0.22	0.12	0.13	0.06	0.09	0.02	0.01	< 0.001
Aluminium (Al^{3+})	mg/l	110	99	44.2	45.8	18.5	24.6	1.08	1.07	0.005
Iron tot. (Fe_{tot})	mg/l	200	125	58.3	64.8	18.5	30.7	3.53	2.17	< 0.01
Iron-II (Fe^{2+})	mg/l	154	125	58.3	64.8	18.5	30.7	0.31	0.3	< 0.01
Iron-III (Fe^{3+})	mg/l	46	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	3.22	1.86	< 0.01
Sodium (Na^{+})	mg/l	65	44	28	24	19	18	4.5	2.9	0.55
Potassium (K^{+})	mg/l	59	90	41	56	31	48	10	9.7	5
Calcium (Ca^{2+})	mg/l	20	37	6.3	14	7.8	18	2.9	2.8	3.1
Magnesium (Mg^{2+})	mg/l	340	72	9.7	26	6.9	21	0.51	0.53	0.58
Ammonium (NH_4^{+})	mg/l	2.3	2.2	1.3	1.3	0.77	0.92	0.22	0.21	0.21
Silicon (Si^{4+})	mg/l	-	16.8	17.3	14.4	10	7.8	2.8	2.7	< 0.5
Hydrogen carbonate (HCO_3^{-})	mg/l	59.2	55.2	40.9	42.3	38.3	42.1	9.27	8.97	34
Chloride (Cl^{-})	mg/l	18	5.8	4.9	2.8	3.5	4.9	1.2	0.7	3.8
Sulfate (SO_4^{2-})	mg/l	5	1.1	1.1	0	1.1	0	0	0	0
Nitrate (NO_3^{-})	mg/l	67	65	64	65	65	65	65	65	67
Fluoride (F^{-})	mg/l	0.69	0.41	0.13	0.14	0	0	0	0	0

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CEC experiment 10-20 mm

Appendix Table A-3: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 7 days. Sample name IG_BH04_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
354170	1:1 No. 1	5.87	37.4	15.5	25.1	11
354171	1:1. No. 2	5.7	40.6	21.4	28.3	12
354172	1:2 No. 1	8.93	22.4	13.3	10.6	3.9
354173	1:2 No.2	9.5	22.1	13.2	10.1	3.9
354174	1:4 No. 1	21.8	6.2	5.6	5.51	0.42
354175	1:4 No 2	18.0	9.7	9.4	4.89	0.95
354176	1:10 No. 1	25.3	4.2	4.0	2.75	0.17
354177	1:10 No. 2	28.6	1.5	2.4	2.27	0.24
354178	Blank	33.5	0.25	0.91	0.30	0.19

Appendix Table A-4: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 14 days. Sample name IG_BH04_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
354170	1:1 No. 1	5.09	51.6	13.3	27.9	11
354171	1:1. No. 2	5.08	53.8	18.9	30.5	14
354172	1:2 No. 1	4.58	33.3	13.1	10.6	4.3
354173	1:2 No.2	4.56	30.7	13.8	9.26	3.7
354174	1:4 No. 1	16.5	11.0	9.5	5.87	0.53
354175	1:4 No 2	10.7	16.0	12.0	5.08	1.4
354176	1:10 No. 1	22.3	6.1	5.2	3.90	0.31
354177	1:10 No. 2	25.6	2.2	3.2	2.47	0.18
354178	Blank	37.4	0.32	0.89	0.41	0.14

Appendix Table A-5: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 21 days. Sample name IG_BH04_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
354170	1:1 No. 1	4.15	63.3	17.1	36.0	0.04
354171	1:1. No. 2	3.98	63.1	19.1	32.4	14
354172	1:2 No. 1	2.65	40.8	12.6	10.2	5.1
354173	1:2 No.2	2.72	36.6	13.9	8.69	3.0
354174	1:4 No. 1	15.7	13.4	8.7	5.08	0.32
354175	1:4 No 2	9.05	20.4	13.4	4.77	1.1
354176	1:10 No. 1	23.3	7.5	5.4	3.68	0.17
354177	1:10 No. 2	27.2	3.5	4.4	2.76	0.12
354178	Blank	36.7	0.32	0.89	0.35	0.09

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Appendix Table A-6: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 28 days. Sample name IG_BH04_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
354170	1:1 No. 1	3.59	70.7	12.5	37.1	19
354171	1:1. No. 2	3.67	69.3	19.2	34.0	17
354172	1:2 No. 1	2.88	40.8	12.7	< 0.1	< 0.1
354173	1:2 No.2	2.69	36.6	13.3	8.34	4.7
354174	1:4 No. 1	12.9	13.4	9.3	7.05	0.59
354175	1:4 No 2	6.85	20.4	13.7	5.45	1.9
354176	1:10 No. 1	18.9	7.5	6.5	6.04	0.34
354177	1:10 No. 2	24.6	3.5	4.5	4.62	0.47
354178	Blank	36.9	0.13	1.5	0.80	0.12

Appendix Table A6-7: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 36 days. Sample name IG_BH04_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
354170	1:1 No. 1	4.11	82.6	26.7	44.1	19
354171	1:1. No. 2	3.96	79.4	28.1	41.3	22
354172	1:2 No. 1	3.10	49.3	13.1	14.3	6.8
354173	1:2 No.2	3.23	43.4	13.9	10.1	4.5
354174	1:4 No. 1	11.2	17.6	10.0	5.9	0.94
354175	1:4 No 2	5.62	25.6	14.2	6.37	3.3
354176	1:10 No. 1	17.1	10.4	7.0	3.80	0.35
354177	1:10 No. 2	21.2	4.4	4.9	3.29	0.29
354178	Blank	31.4	0.11	0.7	0.15	< 0.1

Appendix Table A-8: Element concentrations of the CEC experiment solution with the largest grain size fraction (10-20 mm) after 62 days. Sample name IG_BH04_SA001 10-20 mm.

Hydro Lab.-No.	Sample	Nickel (Ni^{2+})	Sodium (Na^{+})	Potassium (K^{+})	Calcium (Ca^{2+})	Magnesium (Mg^{2+})
		mg/l	mg/l	mg/l	mg/l	mg/l
354170	1:1 No. 1	8.04	99.5	25.7	71.4	45
354171	1:1. No. 2	7.06	91.3	20.9	72.9	47
354172	1:2 No. 1	4.88	59.1	11.2	21.9	13
354173	1:2 No.2	4.78	53.4	11.2	17.5	9.9
354174	1:4 No. 1	6.48	24.6	11.0	4.00	0.8
354175	1:4 No 2	3.17	31.2	14.1	3.54	1.7
354176	1:10 No. 1	13.5	14.8	7.7	3.18	0.4
354177	1:10 No. 2	24.0	4.6	5.1	3.05	< 0.1
354178	Blank	34.6	< 0.0	0.8	0.08	< 0.1

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Appendix Table A-9: Element concentrations of the final CEC experiment solution with the largest grain size fraction (10-20 mm) after 71 days. Sample name IG_BH04_SA001 10-20 mm.

Hydro Lab.-No.		356425	356426	356427	356428	356429	356430	356431	356432	356433
Sample		1:1 No.1	1:1 No.2	1:2 No. 1	1:2 No. 2	1:4 No. 1	1:4 No. 2	1:10 No. 1	1:10 No. 2	Blank
Rock/Water ratio		1:1	1:1	1:2	1:2	1:4	1:4	1:10	1:10	-
m (rock)	g	59.529	59.803	29.985	30.556	15.354	14.872	10.202	10.375	0
m (solution)	g	58.625	59.025	59.668	59.32	59.198	58.836	98.453	98.971	74.364
pH-value start		9.22	9.18	9.14	9.14	9.11	9.15	9.14	9.15	9.14
pH-value end		10.51	10.30	10.42	10.32	9.67	10.22	9.61	9.96	8.92
Redox potential start	mV	9.1	24.1	5.6	19.1	15.8	23.2	7.3	14.8	9.9
Redox potential end	mV	-88.0	-104.7	-88.7	-85.6	-83.1	-78.7	-68.6	-74.4	-24.2
Nickel (Ni^{2+})	mg/l	4.20	3.46	2.80	2.54	2.71	1.97	11.8	18.5	30.8
Barium (Ba^{2+})	mg/l	2.18	2.04	0.804	0.598	0.114	0.268	0.09	0.072	0.13
Strontium (Sr^{2+})	mg/l	1.03	0.77	0.38	0.32	0.05	0.09	0.06	0.04	0
Aluminium (Al^{3+})	mg/l	241	165	89.5	87.5	12.2	23.5	9.25	3.25	0.003
Iron tot. (Fe_{tot})	mg/l	82.6	87.6	33.0	28.8	5.38	11.1	2.13	0.63	< 0.001
Iron-II (Fe^{2+})	mg/l	82.6	79.7	33.0	28.8	2.66	10.2	2.13	0.60	< 0.001
Iron-III (Fe^{3+})	mg/l	< 0.1	7.9	< 0.1	< 0.1	2.72	0.9	< 0.01	0.03	< 0.001
Sodium (Na^{+})	mg/l	86.7	87.9	59.2	55	28.1	34.3	19.0	5.9	0.28
Potassium (K^{+})	mg/l	7.3	7.9	8.2	8.4	11.2	12.6	7.5	6.5	0.99
Calcium (Ca^{2+})	mg/l	62.5	45.9	22.6	21.4	3.25	5.20	3.80	3.54	<0.1
Magnesium (Mg^{2+})	mg/l	24.0	21.3	7.74	10.1	0.65	3.15	0.77	< 0.1	0.17
Ammonium (NH_4^{+})	mg/l	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Silicon (Si^{4+})	mg/l	49.0	44.9	32.0	29.6	11.7	14.7	8.8	5.3	< 0.1
Hydrogencarbonate (HCO_3^{-})	mg/l	100	97	63	58	50	50	8.8	8.0	7.1
Chloride (Cl^{-})	mg/l	7.8	20	3.8	3.8	2.4	2.6	2.0	0.99	1.1
Sulfate (SO_4^{2-})	mg/l	0.76	2.2	< 0.5	< 0.5	< 0.5	< 0.5	0.86	< 0.5	< 0.5
Nitrate (NO_3^{-})	mg/l	64.0	64.1	64.4	63.9	63.7	64.8	63.2	61.6	64.5
Fluoride (F^{-})	mg/l	0.62	064	0.27	0.25	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1

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