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Executive summary

This report releases the laboratory results of brine and rock interaction experiments of six Clearwater shale caprock samples, collected from depths ranging from 400.80 m to 522.70 m in the Athabasca oil sands area of northeast Alberta (townships 68-74, ranges 5-7, west of the fourth meridian). In corporation with University of Regina, an 84-day CO_2-brine-rock experiment was conducted under potential CO_2 storage conditions (22 ± 1°C and 5 MPa) representative of shallow gas reservoirs. Main findings from the sample tests and experiments are as follows:

- **Mineralogical Compositions:** X-ray diffraction (XRD) revealed that the primary components of the Clearwater shale caprocks in the selected core samples are quartz (43.2–68.3 wt.%), clay minerals (26.2–45.1 wt.%), K-feldspar (1.1–3.8 wt.%), and albite (1.9–13.6 wt.%), with some samples also containing small amount of dolomite.
- **Water Chemistry**: Water analysis results showed a sharp increase, followed by a slight decrease in total dissolved solids (TDS) upon CO_2 exposure. While Ca^{2+}, Mg^{2+}, and K^{+} levels showed no significant changes over time, Na^{+}, Fe^{2+}, and Mn^{2+} exhibited some variation.
- **Indication of CO_2 saturated formation water - caprock interaction**: Based on the water chemistry and XRD results, the primary chemical reaction in the rock samples appears to involve albite/K-feldspar reacting with CO_2 and water over time to produce dawsonite and quartz. This reaction could result in a significant increase in solid volume within the system, suggesting notable carbonation, which is advantageous for long-term CO_2 storage under the Clearwater caprock.

The experimental results revealed gradual chemical changes in Clearwater shale under potential storage conditions without supercritical CO_2.

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1 Introduction

The Clearwater shale is a major caprock for natural gas accumulations and oilsands deposits in northeast Alberta. It forms the top seal of potential CO_2 storage in the Wabiskaw and McMurray sandstone reservoirs. Geotechnical and mechanical properties of the shale, which are crucial for assessing caprock integrity in steam-assisted gravity drainage (SAGD) projects, have been extensively studied (e.g., Liu et al., 2022; Shafie Zadeh and Chalaturnyk, 2015). The Geological Survey of Canada, Calgary has conducted a risk and capacity assessment of CO_2 storage in the Kirby gas field using statistical and numerical methods (Peng et al., 2023), primarily focusing on determination of the criteria and thresholds that quantify the risk of leakage through Clearwater Shale for safely storing CO_2 in shallow gas formations based on the original reservoir conditions. Huang et al. (2024) examined the geochemical properties and their implications for cap-rock integrity for CO_2 storage. However, few studies have examined the potential mineralogical changes in the Clearwater Shale following CO_2 exposure in the context of geological storage of CO_2 in the underlaying porous strata. Since the CO_2-water-caprock interaction is sensitive to mineral composition, the risk associated with dynamic changes of the Clearwater shale after exposure to CO_2 remains unknown. To fill the knowledge gap in leakage risk evaluation for long-term CO_2 storage in depleted gas reservoir in this area, a CO_2-brine-rock interaction experiment was carried out for the Clearwater Shale.

2 Materials

Six caprock core samples were collected from Clearwater shale from five wells with short names of 7-26-74-5W4, 8-16-72-6W4, 6-7-73-6W4, 10-4-73-7W4, and 11-16-68-5W4, located in the Athabasca oil sands area, townships 68-74, ranges 5-7, west of the fourth meridian. The surface locations of those wells are illustrated in Figure 1. The burial depths of rock samples range from 400.80 to 522.70 m. More detailed information on the cores, wells, and formation depths can be found in **Appendix A**.

No formation water is available to this study. Synthetic water is prepared based on information of available formation water chemical compositions (data from AccuMap database) (Figure 1).

The CO_2 used for saturating synthetic water is supplied by Linde Canada Inc. with a purity of 99.98%.

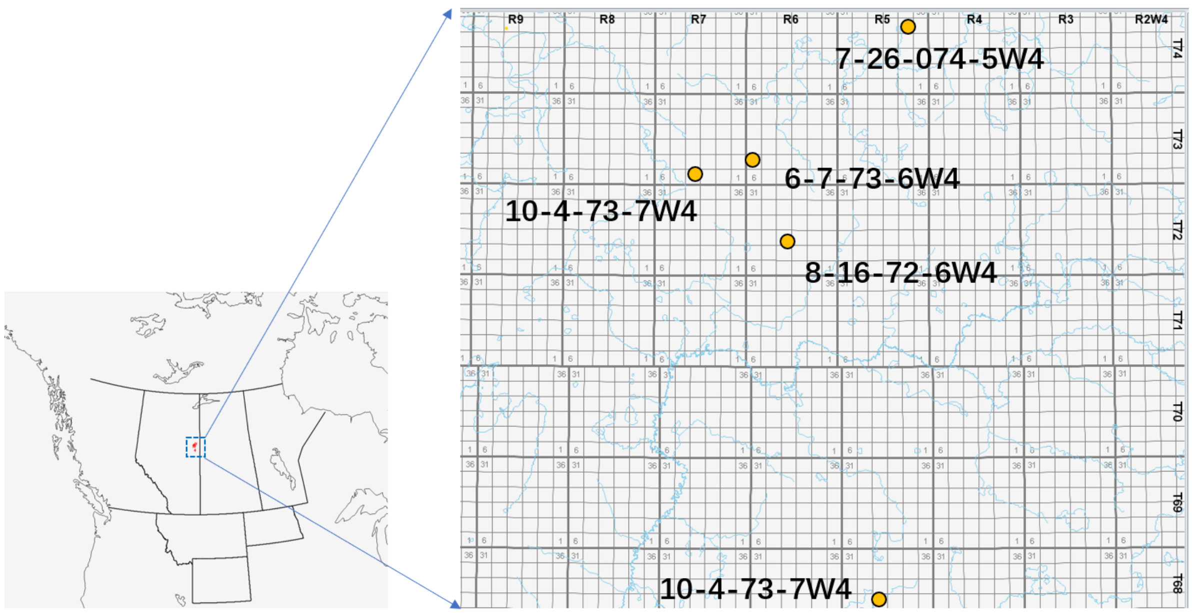

Figure 1. Locations of wells for collecting Clearwater shale caprocks (Screenshotted from AccuMap)

3 Experimental setup and procedures

An overview of the geochemical experiment procedure is shown in Figure 2, which includes materials pretreatment, the periodic reaction process, and periodic sampling of brine and rock.

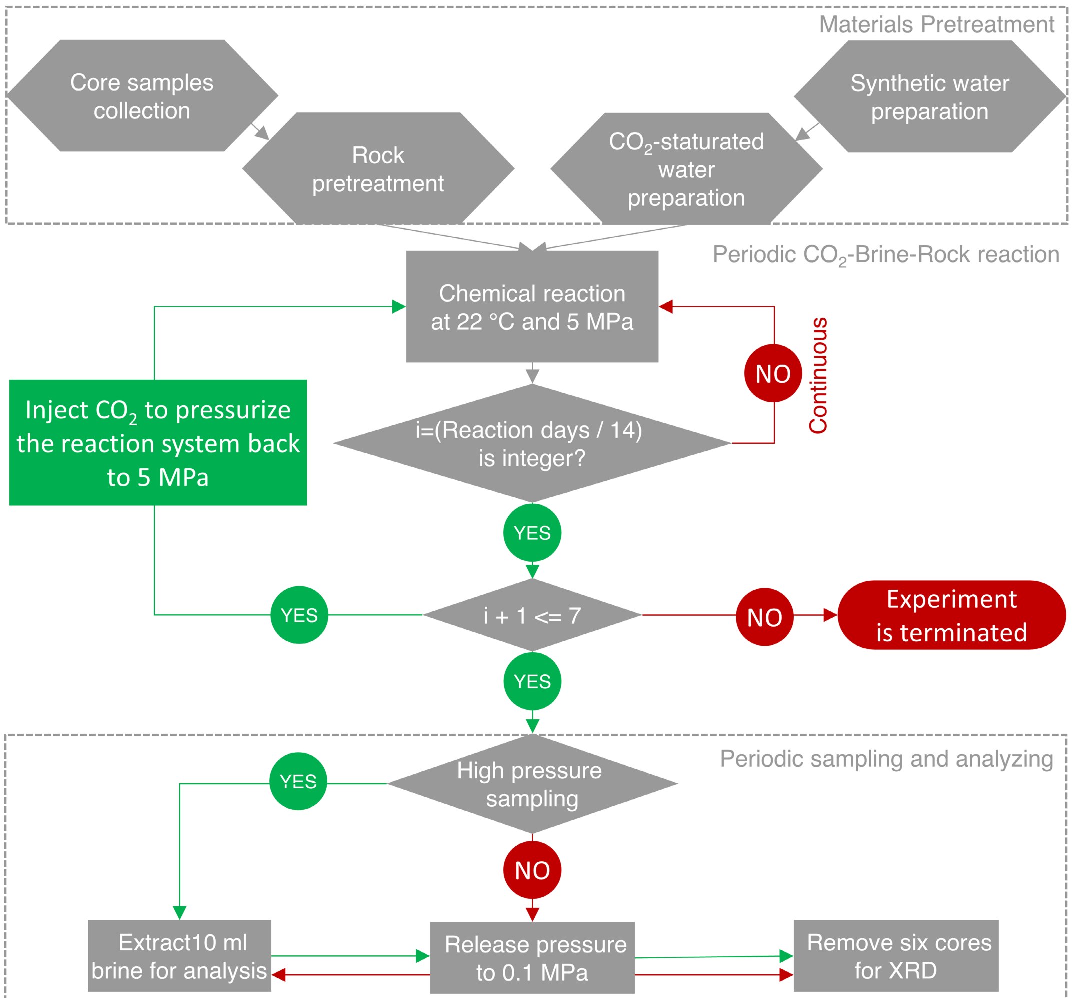

Figure 2. Workflow of the geochemical experiment.

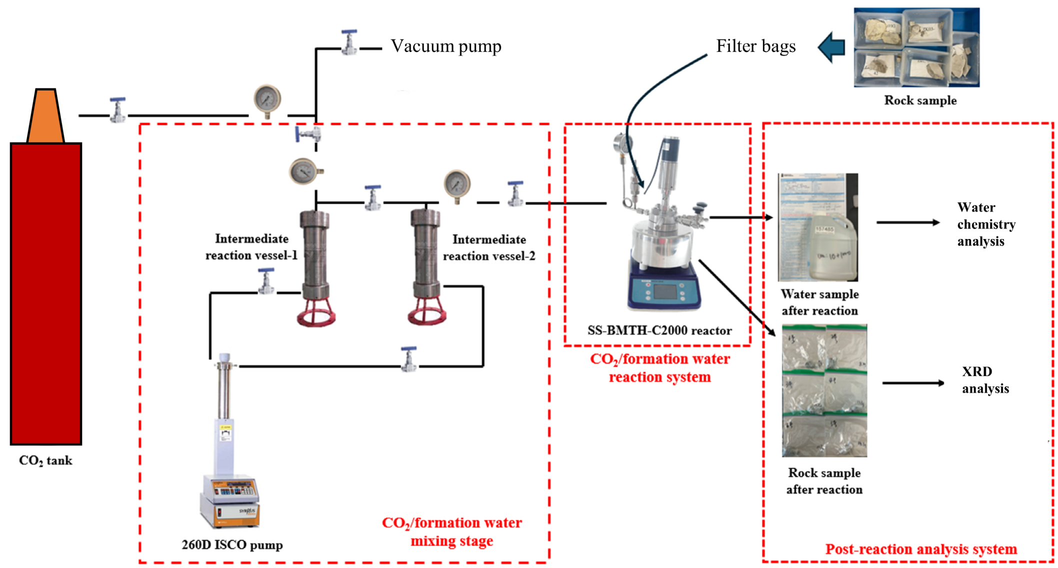

Figure 3. Experimental setup.

Figure 3 shows experimental setup used in this work, which include three subsystems: 1) CO_2/synthetic water mixing system, 2) CO_2-brine-rock interaction system (at 22 ± 1 °C and 5 MPa) and 3) post-reaction water/rock analysis system. The main apparatus for the chemical reaction experiment include a CO_2 injection gas tank, an SS-BMTH-C2000 (Shanghai Yushen Instrument Co., Ltd., with a maximum chamber volume of 660 mL, maximum working pressure of 48 MPa, and maximum working temperature of 250 ℃) reactor, an 260D ISCO injection pump (Teledyne ISCO, Inc.), two ZR-3 piston intermediate vessels (Haian Petroleum Instrument Co., Ltd.) for mixing CO_2 and synthetic brine before reactions.

(a) Materials pretreatment for reaction experiments

Cores. Before the chemical reaction test, the six original cores were firstly dried in an oven at 80°C for 24 hours. Each core was then dry-cut into seven smaller pieces of similar size. Due to the small dimensions and well-cemented nature of the original core chunks, we assumed that the pieces cut from the same core were relatively homogeneous in mineral composition, though this cannot be guaranteed. Uncertainties introduced by this assumption should be aware when interpreting mineral composition changes in cores.

For each group of core pieces from the same original core, one piece was crushed to a particle size of 40 µm for X-ray diffraction (XRD) analysis to under the initial mineral compositions. The remaining 36 small pieces were individually placed in labeled filter bags (5 cm× 7 cm, cotton, 200 mesh) for the chemical reaction test. After each designated reaction period, one piece from each original core group was dried and crushed for subsequent XRD analysis to identify any potential changes in mineral composition during CO_2 treatment.

Fluids. To mimic representative geochemical compositions of formation water, we retrieved the produced water analytical data around the selected well locations and formations from AccuMap database. The result (Table 1) indicates that the formation water is highly mineralized Na-Cl type water, with a total dissolved solids (TDS) content of approximately 13,500 mg/L.

Table 1. The geochemical properties of produced formation water and synthetic brine.

Formation water			**Synthetic Brine (1L)**		
Contents	**Concentration**	**Unit**	**Contents**	**Mass**	**Unit**
Na	4790	mg/L	NaCl	8	g
K	37.3	mg/L	NaSO4	2.5	g
Ca	108	mg/L	NaHCO3	1.5	g
Mg	59.6	mg/L	CaCl2	0.3	g
Fe	1.8	mg/L	MgCl26H2O	0.5	g
Cl	7380	mg/L	KCl	0.1	g
HCO3	1090	mg/L	NaCO3	1	g
SO4	3.3	mg/L			
TDS	13500	mg/L	Calculated TDS	13900	mg/L

The mass of mineral salts required to prepare a one liter of synthetic water solution is detailed in Table 1. This table reveals that the synthetic water primarily consists of NaCl, $CaCl_2$, KCl, $MgCl_2 \cdot 6H_2O$, Na_2SO_4, and $NaHCO_3$, with a TDS of 13900 mg/L, which is very close to the TDS of produced formation water in the Table 1.

Then afterwards, the sufficient amount of synthetic brine and CO_2 with the purity of 99.98% are mixed in vessel 1 and vessel 2 (Figure 2) through a slow back-and-forth flow process (0.1 ml/min) between the two vessels at a temperature of 22 ± 1 °C and a pressure of 5 MPa to represent the potential CO_2 storage conditions in the targeted Kirby gas reservoir. Until the pressure stabilized between the back-and-forth flow, the CO_2-staturated synthetic water is stored for later CO_2-brine-rock reaction experiments.

(b) Periodic CO_2-brine-rock reaction

After placing the 36 pretreated core samples into the chemical reactor, a gas leakage test is conducted at the working pressure of 5 MPa and room temperature (22 ± 1°C) using CO_2. Following the gas leakage test, CO_2-saturated synthetic brine is injected into the reactor chamber that has already been pressurized with CO_2. During brine injection, the previously filled CO_2 gas is allowed to flow out of the reactor under the pressure control of a back pressure regulator (BPR). Once all the gas in the reaction system is displaced, the CO_2-saturated brine supply is terminated, with approximately 600 mL of brine initially introduced.

After a 14-day reaction period, the reactor pressure is gradually released to 0.1 MPa (ambient pressure) for brine and rock sampling. A 10 mL brine sample is extracted for water chemistry analysis, and six bags of rock from different sampling depths are removed for XRD testing. To maintain the consistency of the reaction environment, no fresh CO_2-saturated brine is added. Instead, CO_2 is directly injected from the bottom of the reactor to repressurize the chamber to 5 MPa until stable. This procedure is repeated every 14 days until the 84-day reaction period is complete.

(c) Periodic sampling and analyzing processes

Every 14 days, 10 mL brine sample and one bag of each of the 6 core samples will be collected until the reaction experiment is terminated on day 84. The core sampling process is consistent across all periods.

For brine sampling, a standard procedure is used across all sampling points. Its steps include: 1) reduce the reactor chamber's pressure to the atmosphere pressure (roughly 0.1 MPa); 2) Use a syringe to sample 10 mL brine from the chamber (the brine is turbid because of pressure disturbance effects); 4) Transfer the sampled brine to a 1000 mL sampling bottle to be diluted for 100-fold with DI water, well capped and labeled; 5) Submit the diluted brine sample to Saskatchewan Health Authority for water chemistry analysis.

It is worth noting that during the first two sampling periods (on Day 14^{th} and Day 28^{th}), an in-situ pressure sampling process was tested to avoid any pressure disturbance that might affect brine sample quality, particularly given the observation of suspended particles in the reactor chamber. Specifically, at the end of an experimental period, a 260D ISCO pump (Teledyne ISCO, Inc.), a back pressure regulator (BPR), and a sampler were installed at the reactor's exhaust port (Figure 2) for liquid collection. During this process, a pressure difference of 0.05 MPa is maintained between the reactor (5 MPa) and the BPR (4.95 MPa). This step ensures that the reacted brine flows out of the reaction system slowly, allowing CO_2 or pressure to be released from the system, thereby preventing any pressure disturbance in the test samples and reaction brine.

4 Results and Discussion

4.1 Changes in the reaction solution

Table 2 summarizes the recorded ion compositions of diluted brine in each sampling period. The original reports of water analysis in each sampling period are provided in **Appendix B**. Keep in mind all the results recorded in Table 2 and **Appendix B** are all based on sampled brine diluted by 100-fold. The values cannot reflect the actual chemical environment in the reactor, but their changing trends can represent the interactions between the brine and rocks.

In the sampling results, it is worth noting that the tests of samples 831651 and 835757 were high-pressure sampling tests. The sample 835757 involved shaking the water sample in the reactor before sampling. It can be observed that although there were significant differences between the results of high-pressure sampling and atmospheric pressure sampling at 14 days, such sampling differences were almost non-existent on the 28 days. Thus, the difference between high-pressure sampling (e.g., sample 835757) and atmospheric pressure sampling (e.g., sample 835756) can be eliminated by shaking the reactor before sampling.

Table 2. Chemical composition changes of the CO_2-saturated sythetic brine following 100-fold dilution.

Days	No.	T, ℃	P, atm	Sdate	Svol, ml	Conductivity (μS/cm)	pH	Total Alkalinity (mg/L CaCO3)	Phenol Alkalinity (mg/L CaCO3)	Bicarbonate (mg/L)	Carbonate (mg/L)	Hydroxide (mg/L)	Chloride Dissolved (mg/L)	Fluoride Dissolved (mg/L)	Nitrate Dissolved (mg/L)	Sulfate Dissolved (mg/L)	Total Hardness (Calculated) (mg/L CaCO3)	TDS (mg/L)	Fe (mg/L)	Mn (mg/L)	Ca (mg/L)	Mg (mg/L)	K (mg/L)	Na (mg/L)
						Analysis items																		
14	831650	22 ± 1	1	2024-01-30	10	391	7.5	34.6	0	42	0	0	71.3	< 0.05	0.3	36.4	29	226	0.2	0.12	5	4	4	63
14	831651	22 ± 1	49.34	2024-01-30	10	344	7	30.6	0	37	0	0	62.4	< 0.05	0.3	32.6	22	196	0.3	0.1	4	3	3	54
28	835756	22 ± 1	1	2024-02-13	10	373	6.8	34.5	0	42	0	0	67.9	< 0.05	0.2	35.9	29	225	0.1	0.14	5	4	4	66
28	835757	22 ± 1	49.34	2024-02-13	10	372	6.7	34.9	0	43	0	0	67.5	< 0.05	< 0.2	35.7	29	225	0.1	0.14	5	4	4	66
42	835755	22 ± 1	1	2024-02-28	10	373	7	34.4	0	42	0	0	67.4	< 0.05	0.3	35.7	29	223	0.2	0.14	5	4	4	65
56	838209	23 ± 1	1	2024-03-12	10	353	7	32.2	0	39	0	0	62.7	< 0.05	0.3	33.1	22	201	0.2	0.13	4	3	4	55
70	838210	24 ± 1	1	2024-03-25	10	367	7.3	32.8	0	40	0	0	65.5	< 0.05	0.3	34.2	29	211	1.1	0.15	5	4	4	58
84	839353	25 ± 1	1	2024-04-10	10	385	7.1	34.4	0	42	0	0	69.4	< 0.05	0.3	36.2	26	218	0.7	0.15	4	4	3	59

4.2 Changes in shale caprocks

The initial XRD results of core samples are presented in Table 3 and Figures C-1 to C-6 in **Appendix C**. It's evident that the quartz, albite and clay minerals are three main contents of Clearwater shales from the region of the townships 68-74, ranges 5-7, west of the fourth meridian.

Table 3. XRD results of Clearwater shale before CO_2 exposure

No.	UWI	Core box	Depth (m)	XRD test sample mass (g)	Relative content of whole rock minerals (wt.%)					
					Quartz	Potassium feldspar	Albite	Dolomite	Pyrite	Clay minerals
1	7-26-074-5w4	1-2/6	400.80	10.214	48.2	1.2	7.2	1.1	/	42.3
2	8-16-72-6w4	1-2	470.60	11.056	58.9	1.5	9.7	3.2	/	26.7
3	6-7-73-6w4	1-1/6	465.00	10.2793	45.3	1.1	4.1	4.4	/	45.1
4	10-4-73-7w4	2-2/6	455.90	11.2431	43.2	2	11.9	/	/	42.9
5	10-4-73-7w4	9-5/6	522.70	11.0018	68.3	3.6	1.9	/	/	26.2
6	11-16-68-5w4	1-2/6	461.50	12.5155	49.5	3.8	13.6	/	/	33.1

Table 4 summarizes the mineralogical changes in Clearwater shale over 84 days of exposure to CO_2-saturated brine in a potential CO_2 storage environment (22 ± 1°C and 5 MPa). The initial caprock sample consists of 33–83 wt.% quartz, 15–46 wt.% clay, 0–23 wt.% albite, 0.6–9.7 wt.% feldspar, and a small amount of dolomite. During CO_2 treatment, a decrease in clay content was observed in all samples, though at varying levels.

Table 4 also records the changes in sample mass before and after CO_2 treatment for all samples. For most wells, the mass changes were minimal, not exceeding 0.3 grams. However, samples from well 10-4-73-7W4 exhibited a significant mass change, with a maximum of 1.9 grams. This may indicate either a different degree of reaction or alteration under CO_2 exposure compared to samples from other wells, or substantial heterogeneity within the samples from well 10-4-73-7W4.

Table 4. Changes in Clearwater shale composition over time due to CO_2 exposure

Days	Collected date	Sample mass/g			Core from well 00/07-26-074-05w4/0 at 400.80 m: Relative content of whole rock minerals (wt.%)					
		Before	After	change	Quartz	Potassium feldspar	Albite	Dolomite	Pyrite	Clay minerals
0	01/15/2024				48.2	1.2	7.2	1.1	0	42.3
14	01/30/2024	5	5	0	58.8	2.1	5.5	0	0	33.6
28	02/13/2024	4.8	4.7	0.1	55.9	2.4	7.5	1	1.4	31.8
42	02/28/2024	4.1	3.8	0.3	46.2	1.3	12.7	0	0	39.8
56	03/11/2024	4.3	4.2	0.1	49	3.2	7.5	2.7	1	36.6
70	03/25/2024	4	4	0	47.2	1.2	3.6	0.7	1.5	45.8
84	04/08/2024	4.1	4.1	0	53.2	3.1	5.6	3.1	0.5	34.5

Days	Collected date	Sample mass/g			Core from well 00/08-16-072-06w4/0 at 470.60 m: Relative content of whole rock minerals (wt.%)					
		Before	After	change	Quartz	Potassium feldspar	Albite	Dolomite	Pyrite	Clay minerals
0	01/15/2024				58.9	1.5	9.7	3.2	0	26.7
14	01/30/2024	8.4	8.2	0.2	55	2.9	13.2	1.3	0.9	26.7
28	02/13/2024	7.3	7.2	0.1	56.3	1.5	12.4	3.3	0	26.5
42	02/28/2024	7	6.9	0.1	51.2	1.5	14	1.7	0	31.6
56	03/11/2024	6.7	6.8	-0.1	52.6	2.3	13.3	0.8	1.3	29.7
70	03/25/2024	6.9	7	-0.1	54	1.1	8.4	1.9	0.9	33.7
84	04/08/2024	8.7	8.8	-0.1	55.8	1.3	14.8	0	1.4	26.7

Days	Collected date	Sample mass/g			Core from well 00/06-07-073-06w4/0 at 465.00 m: Relative content of whole rock minerals (wt.%)					
		Before	After	change	Quartz	Potassium feldspar	Albite	Dolomite	Pyrite	Clay minerals
0	01/15/2024				45.3	1.1	4.1	4.4	0	45.1
14	01/30/2024	12.4	12.5	-0.1	45.2	2.1	12.8	5.4	0.6	33.9
28	02/13/2024	12.6	12.6	0	51.6	1.4	6.5	5.9	1	33.6
42	02/28/2024	13	13.1	-0.1	55.9	1.6	6.7	5.9	0.4	29.5
56	03/11/2024	10.8	10.9	-0.1	49.2	1	10.9	7.2	0	31.7
70	03/25/2024	13	13.2	-0.2	56.3	1.5	5.9	4.8	0.8	30.7
84	04/08/2024	8.7	8.8	-0.1	48.5	2.5	10	4.1	1.6	33.3

Days	Collected date	Sample mass/g			Core from well 00/10-04-073-07w4/0 at 455.90 m: Relative content of whole rock minerals (wt.%)					
		Before	After	change	Quartz	Potassium feldspar	Albite	Dolomite	Pyrite	Clay minerals
0	01/15/2024				43.2	2	11.9	0	0	42.9
14	01/30/2024	11.4	11.3	0.1	46.9	1.8	20	0	1.3	30
28	02/13/2024	11.4	11.3	0.1	49.5	1.1	13.2	0.9	0	35.3
42	02/28/2024	10.9	10.7	0.2	46.8	4.7	13.5	0	0	35
56	03/11/2024	12	12.2	-0.2	45.7	1.9	13.4	0.9	0	38.1
70	03/25/2024	13.3	13.2	0.1	53.4	1.5	14.5	1.6	0.5	28.5
84	04/08/2024	11.3	11.3	0	48.5	3.2	15.8	1.6	0	30.9

Days	Collected date	Sample mass/g			Core from well 00/10-04-073-07w4/0 at 522.70 m: Relative content of whole rock minerals (wt.%)					
		Before	After	change	Quartz	Potassium feldspar	Albite	Dolomite	Pyrite	Clay minerals
0	01/15/2024				68.3	3.6	1.9	0	0	26.2
14	01/30/2024	14.9	14.3	0.6	74.6	0.7	1.4	0.5	0.9	21.9
28	02/13/2024	14.5	13.9	0.6	83	0.6	1.3	0	0	15.1
42	02/28/2024	14.7	12.8	1.9	80.1	3.6	0	0	0	16.3
56	03/11/2024	14	12.9	1.1	74.5	4.4	1.5	0	0	19.6
70	03/25/2024	12.9	12.3	0.6	74.4	1.8	2.1	0	0.6	21.1
84	04/08/2024	11	10.8	0.2	73.1	0.8	1.1	0	0	25

Days	Collected date	Sample mass/g			Core from well 00/11-16-068-05w4/0 at 461.50 m: Relative content of whole rock minerals (wt.%)					
		Before	After	change	Quartz	Potassium feldspar	Albite	Dolomite	Pyrite	Clay minerals
0	01/15/2024				49.5	3.8	13.6	0	0	33.1
14	01/30/2024	11.6	11.7	-0.1	39.2	2.1	21.9	6.5	1.3	29
28	02/13/2024	13.1	13	0.1	51.4	4.8	19.7	0	1.4	22.7
42	02/28/2024	13.4	13.4	0	49.6	4.8	13.7	0	1.2	30.7
56	03/11/2024	12.8	12.9	-0.1	45.5	2.4	19.2	2.4	1.6	28.9
70	03/25/2024	13.9	13.9	0	48.5	4.6	21.9	0	1.1	23.9
84	04/08/2024	12.4	12.3	0.1	33.4	9.7	23.3	5.5	1.4	26.7

5 Conclusions

Six Clearwater shale caprock samples, ranging from 400.80 m to 522.7 m in depth, were collected from five wells (7-26-074-5W4, 8-16-72-6W4, 6-7-73-6W4, 10-4-73-7W4, and 11-16-68-5W4) in the Athabasca oil sands area of Alberta (townships 68-74, ranges 5-7, west of the fourth meridian). An 84-day CO_2-brine-rock interaction experiment was conducted on these samples under potential CO_2 storage conditions (22 ± 1°C and 5 MPa) typical of shallow gas reservoirs. Mineral and chemical analyses of the core and brine were performed every 14 days to monitor the changes.

This report releases the data with limited preliminary interpretation from this study to alert potential alternations in the caprock of CO_2 storage and provide a background for further research on caprock risk assessments for CO_2 storage in the Athabasca oil sands area. **Appendix A** provides the configurations of the wells from which rock samples were collected. **Appendices B and C** contain the original water chemistry data and rock XRD analyses during CO_2 treatments, respectively.

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