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Abstract

Over more than 60 years, Canadian Nuclear Laboratories (CNL) has developed expertise on hydrogen to support the development and expansion of the Canadian nuclear industry. Hydrogen research in the nuclear industry arises from hydrogen safety in nuclear reactors, hydrogen interaction with materials, and hydrogen isotopes production and management. CNL continues to grow its expertise in areas of the hydrogen value chain including production and storage technology development. Research on hydrogen safety now includes non-nuclear applications to support the development of safety technologies and improvement of codes and standards in Canada and abroad.

As part of the hydrogen materials and storage research, CNL has developed capabilities in Underground Hydrogen Storage (UHS) research since 2018. These capabilities are aimed to develop and deploy UHS as a low-cost bulk hydrogen storage option through:

- Characterization of geologic material samples with pure hydrogen at varying conditions,
- Understanding leak mechanisms and developing control strategies,
- Site specific safety and environmental risk assessment, and
- Techno-economic analysis of UHS based on location and regional energy infrastructure.

CNL strives to assess and inform site selection for cavern project developers as an independent entity and aims to advance standards such as CSA Z341 to account for the unique hazards and properties of hydrogen. CNL has also built in-house techno-economic models to assess UHS in greater detail and set up two laboratory-scale experimental systems to test and characterize different materials. The two experimental systems, both of which have been recently commissioned and tested, are the following:

1. A uniaxial microcolumn system that was designed to assess well-completion materials for hydrogen sealing characteristics under high differential hydrogen pressure.
2. A triaxial core holder designed to analyse NQ sized core specimens.

Both systems are in CNL's Hydrogen Research Laboratory Complex in Chalk River, ON. The hydrogen laboratory complex was completed in 2015 and has been designed and built to meet the Canadian Hydrogen Installation Code (CAN/BNQ 1784-000). As such, this state-of-the-art hydrogen facility enables safe laboratory-scale testing involving large volumes of pure hydrogen at high pressures and temperatures.

Detailed description of the two experimental systems will be presented in this paper with some preliminary data obtained during commissioning.

Key words: Hydrogen, Underground Hydrogen Storage, Geologic Core Analysis

Introduction

Underground Hydrogen Storage (UHS) is a storage technology that enables low-cost and safe storage of hydrogen at scales larger than current storage options and for longer durations [1][2]. There are UHS facilities in the United States (US) and the United Kingdom (UK) where pure hydrogen has been stored and handled in salt caverns for the last 15-20 years [3][4]. These sites were chosen since they have the geologic formations most suitable for hydrogen storage – i.e., homogenous and high-density halite (e.g., sodium chloride - NaCl, or other salts). Such formations are found as bedded salt deposits or within salt domes (known in geology as diapir) which form from bedded salt over thousands of years. Salt domes tend to be oriented vertically which enables the creation of taller caverns. In Canada, there are places which have such salt formations and that is why Canadian Nuclear Laboratories (CNL) is investigating the suitability for hydrogen storage in geologic formations, with focus on salt formations in Canada [5].

As each potential site varies widely in geologic composition, the characterization of the geologic subsurface for suitability as a potential site for UHS is a crucial first step in the development of a commercial UHS operation. Since 2018, CNL had developed techno-economic models to assess viability and business cases for UHS, with potential for large-scale hydrogen production using energy from nuclear power plants with integrated UHS. CNL designed and commissioned two experimental systems to characterize geologic samples for site suitability, leveraging the hydrogen laboratory complex at the Chalk River site. The two systems include:

1. A uniaxial microcolumn system that has been developed to assess well-completion materials for hydrogen sealing characteristics under high differential hydrogen pressure. The system can also be configured to assess hydrogen migration through sedimentary material. The information that can be generated in this system will be useful to simulate hydrogen behaviour downstream of a subsurface leak from a cavern and assist in determining the ideal placement of hydrogen sensors for cavern leak detection.
2. A triaxial core holder has been developed to fit NQ sized core specimens. This system is intended to assess geologic materials for hydrogen permeability under realistic geostatic pressure conditions and at high differential hydrogen pressure. Permeate gas analysis is available to assess chemical interactions of the hydrogen with the geologic material.

It is worth highlighting that there are opportunities for underground hydrogen storage projects in both bedded and domal salt deposits in Canada and CNL is positioned to provide support to those projects leveraging extensive hydrogen expertise and unique facilities.

Experimental Systems

The UHS experimental systems developed at CNL are intended to characterize various materials for their hydrogen permeation and migration properties. Materials of interest are grouped into three categories:

1. **Well-completion materials**. In particular, the cement used in casing installations is of interest, as this material will form a direct process gas interface in a UHS cavern. Samples of this type will be tested in the uniaxial core holder in a high-differential pressure configuration.
2. **Sedimentary materials**. This includes any geology which leaked hydrogen will encounter outside of a UHS cavern. Samples of this type will be tested in the uniaxial core holder in a low-differential pressure configuration.
3. **Cavern wall materials**. This includes cores of salt, as well as any interbedded materials in a location of interest for cavern development. Samples of this type will be tested in the triaxial core holder in a high-differential pressure configuration.

UNIAXIAL MICROCOLUMN SYSTEM

The uniaxial microcolumn system has a 2 L (0.5 gal) pressure vessel (Parr Instrument Company 4600 Series) fitted with a custom lid insert designed to hold NQ geologic cores up to 200 mm (7.9") in length. "NQ" is one of the standard sizes of geologic core samples and denotes a nominal outside diameter (OD) of 45 mm (1.8") [6]. In this system (see **Figure 1**), a **Sample** is held within a stainless steel **Sample Mount**, usually sealed in place using epoxy. The prepared **Sample Mount** is then placed within the **Sample Holder** such that fluid can only move from the **Annular Space (HP)** to the **LP Side** by passing through the sample.

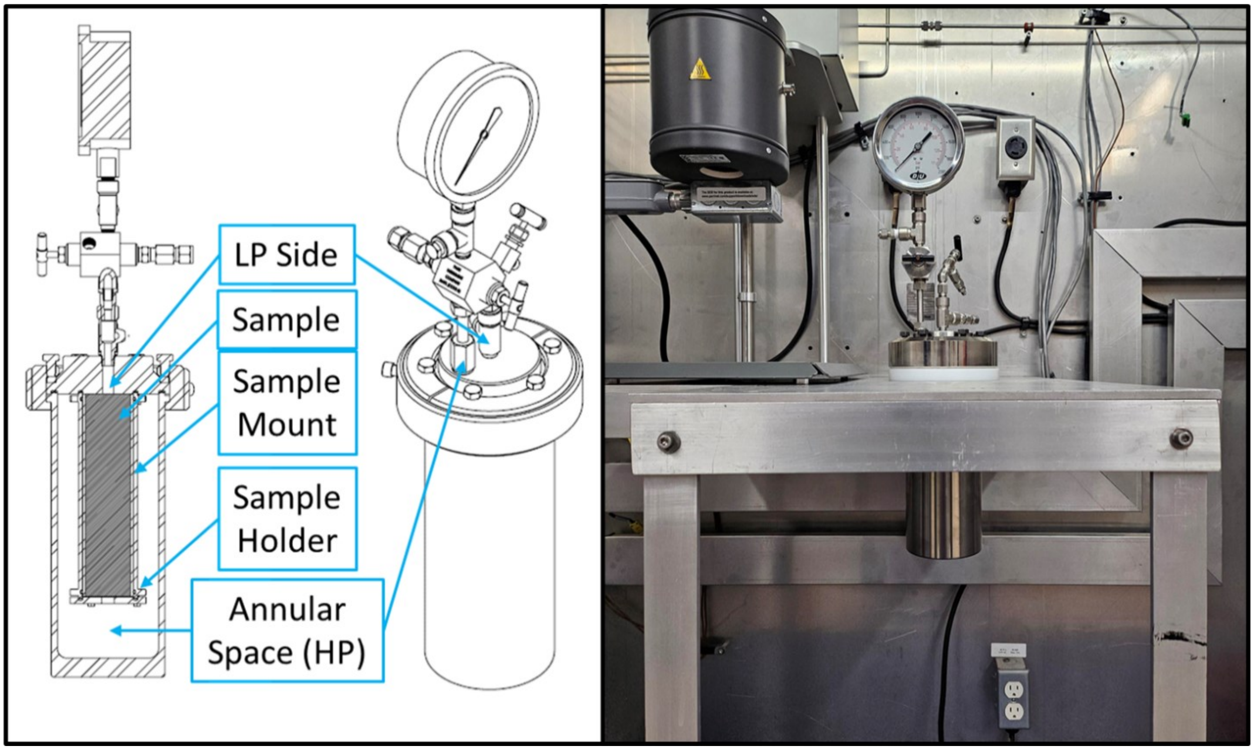

Figure 1: Diagram (left) and photo (right) of the uniaxial microcolumn system at the Hydrogen Research Laboratory Complex in Chalk River, ON.

For well-completion materials, the radial surface of a core specimen is coated with epoxy (typically West System 105 resin with 206 slow hardener), which has shown good hydrogen sealing performance during our tests. The sample is then set into a sample mount and allowed to cure fully. In the case of cements, a core can be made in the sample mount directly to ensure ideal sample geometry.

For sedimentary materials, a sample mount is modified with a stainless steel mesh screen to contain the material. Material is added in the desired state (normally fully hydrated, to mimic field conditions) and a 5 µm (0.0002") stainless steel frit is added to prevent material entering the vent lines during testing. Photos of internal components of the uniaxial microcolumn system are shown in **Figure 2.**

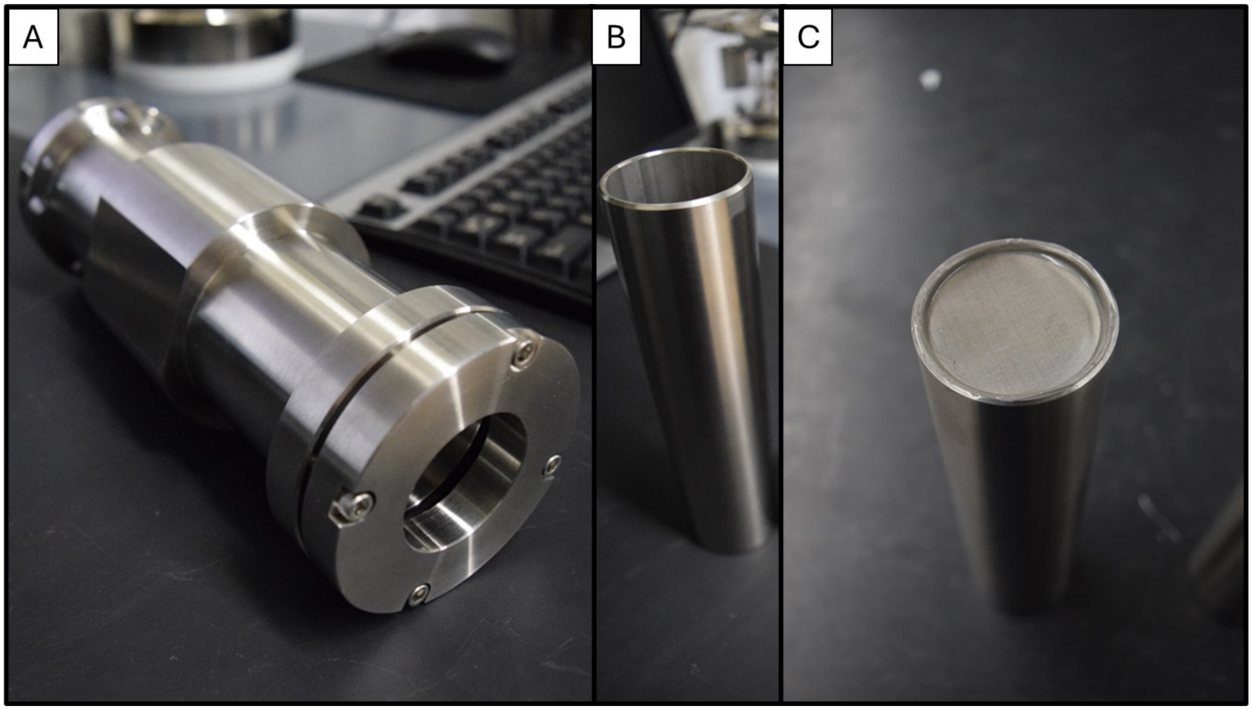

Figure 2: (A) Sample holder, (B) Empty sample mount, and (C) Modified sample mount with 200 mesh stainless steel filter element for sedimentary studies.

In both cases, the sample mount is sealed into the sample holder and installed on the underside of the vessel lid. Once sealed and leak tested, the annular space can be pressurized with hydrogen up to 12.4 MPa (1800 PSIG). Pressure data is obtained on the high-pressure (HP) and low-pressure (LP) sides of the vessel by pressure transmitters. The LP transmitter is a 0-100 kPa (0-15 PSIG) transmitter to maximise sensitivity. Permeation data is obtained from analysis of pressure data with respect to time.

For well-completion material, hydrogen gas is applied to the HP side of the sample holder. Once a target pressure of approximately 7 MPa (1,000 PSIG) is achieved, the hydrogen supply is closed. The pressure of the LP side is measured as a function of time. For low-permeability materials, the test is left for several days. For higher-permeability materials, the HP side pressure is measured as a function of time instead.

For sedimentary material, hydrogen gas pressure of 35 kPa (5 PSIG) is applied to the HP side of the sample holder and the LP side pressure is measured as a function of time.

TRIAXIAL CORE HOLDER

The triaxial core holder is designed for non-destructive tests of core samples. It was developed for this work in conjunction with the engineering team at Parr Instrument Company. The system is based on an existing Parr system - model A1000G triaxial core holder - which was designed to test 100 mm (4”) lengths of core specimens having 25 mm (1”) nominal OD. The adapted system was then modified to test the NQ core size [6], which is most commonly available for testing at CNL and can accommodate cores 25-100 mm (1-4”) in length and 45-50 mm (1-1.77”) in diameter. This design is now available from Parr Instrument Company as the model A1300G Tri-Axial Core Holder [7].

The triaxial system mimics geologic stresses on core samples using a confining gas while a test gas is applied to the specimen. The test gas (e.g., hydrogen) is applied upstream of the core sample and pressure is monitored downstream, similarly to HP and LP sides of the uniaxial microcolumn system. A schematic of the instrument is shown in **Figure 3**, where system components are labeled in white boxes and gas pathways are identified in black boxes. This system is designed in such a way that confining pressure can be independently applied axially and radially up to 50 MPa (7,500 PSIG), in addition to the test gas. In the current configuration of the system, the maximum allowable working pressure (MWAP) of the system is 20 MPa (3,000 PSIG). All gases for these experiments are supplied by standard gas cylinders with a MAWP of 20 MPa (3,000 PSIG) and the ancillary components (fittings, gauges, transmitters, etc.) were selected accordingly. Axial and radial confining pressure is generally applied using argon gas. The axial pressure is applied in the head space, forcing the end faces of the core sample flush against the upstream and downstream gas distributors. The radial pressure is applied in the annular space around the core sample and deforms the polytetrafluoroethylene (PTFE) sleeve to seal around the OD of the core sample. With axial and radial confining pressure applied to the core sample, the test gas can be applied upstream (HP side) of the core sample. The test gas then travels through the core sample to the LP side.

Sealing the core specimens required additional effort; the PTFE sleeves used to provide the OD seal were more sensitive to specimen diameter than expected. With careful assembly, the system sealed reliably for specimens 47.5-50 mm (1.87-1.97”) in diameter, but below this, a reliable seal was not achieved. Application of heavy-wall heat-shrink tubing to increase the OD of undersized specimens was successful in some cases, but this was also inconsistent. To resolve the issue, more compliant sleeves composed of Viton™ rubber have been manufactured and will be used in the next phase of system testing.

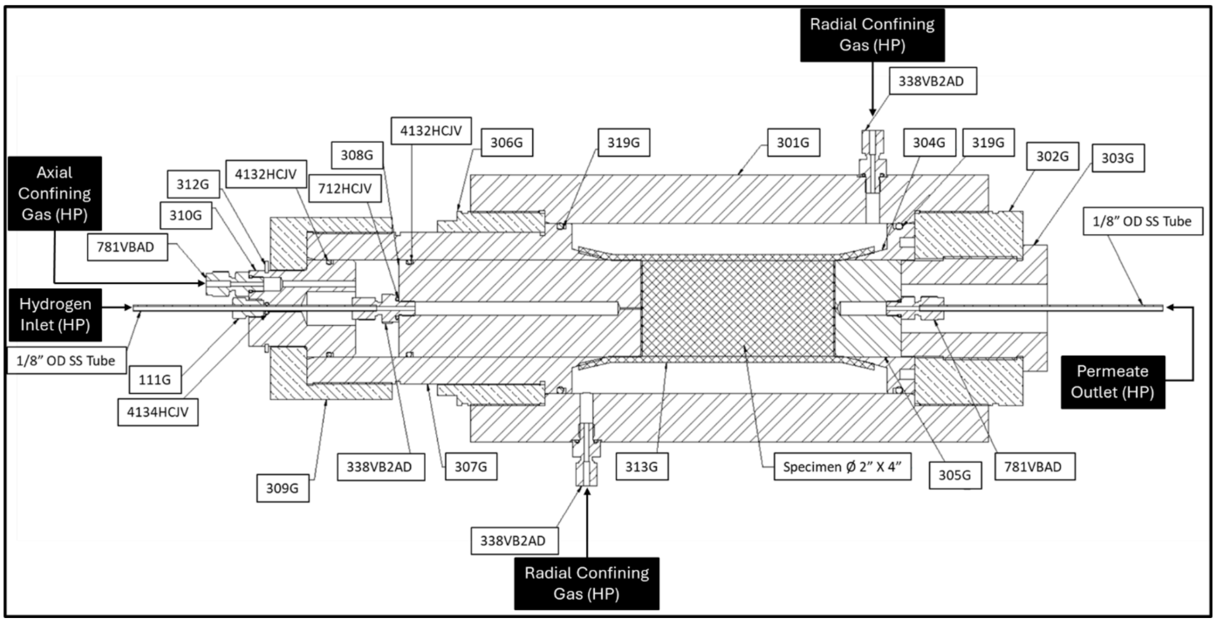

Figure 3: Cut-Away Schematic of the Parr Instrument Company A1300G Tri-Axial Core Holder. <u>Note</u>: Part numbers identified in the figure are described in the documentation provided by Parr Instrument Company.

A photo of the triaxial core holder with gas distribution lines is shown in Error! Reference source not f ound. Hydrogen gas is introduced to the upstream side of the core sample through 3.175 mm (1/8”) stainless steel tubing and its pressure is monitored using a 0-20 MPa (0-3,000 PSIG) pressure transmitter. Hydrogen gas is evenly distributed to the end face of the core sample through the spiderweb-style gas distributor. The hydrogen gas, if any, that passes through the core sample will flow into the gas distribution flow path on the downstream end face of the core sample and be detected using a 0-100 kPa (0-15 PSIG) pressure transmitter.

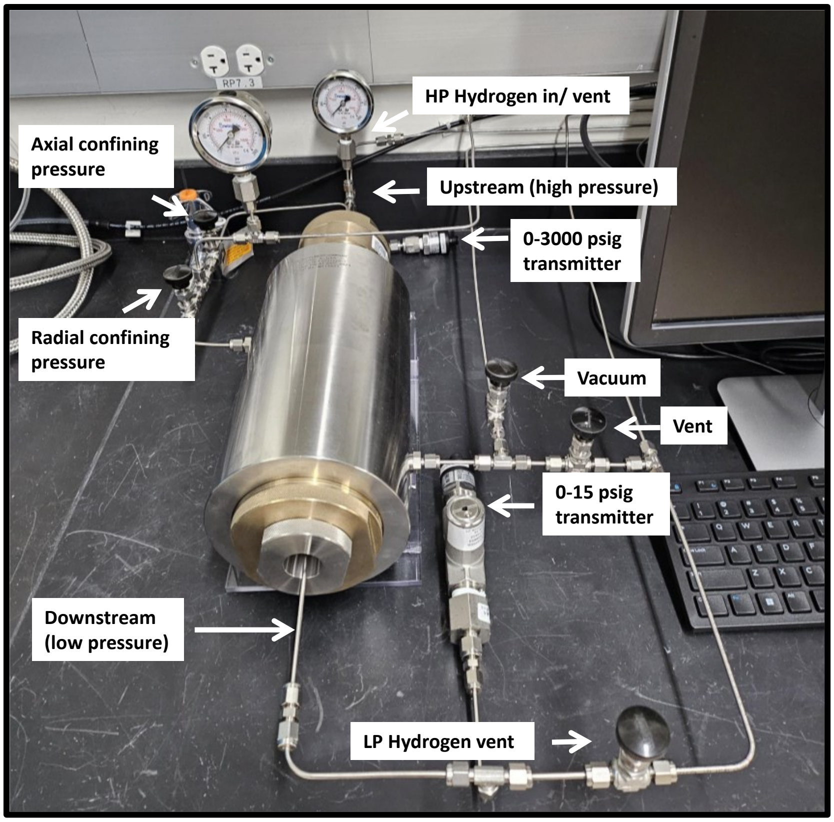

Figure 4: Gas distribution lines for the A1300G triaxial core holder from Parr Instrument Company.

SPECIMEN PREPARATION

WELL-COMPLETION MATERIALS

The well-completion materials are the components used to prepare a drilled underground well for production or injection of compressed gases. These materials include a variety of tools and equipment, such as tubing, packers, valves, and safety devices. In the current case, this refers to the cement-based material that is needed to seal the well casing with the geologic substrate. The material chosen for this work was type-10 portland cement with an additive (BARAD-658™). The additive is designed to improve cement setting characteristics to form effective seals for well completions and abandonments. It primarily achieves the desired characteristics by retaining additional water in the cement during the curing process [8].

Three samples were prepared containing only type-10 portland cement, and three samples were prepared containing cement and the BARAD-658™ additive. The samples were all prepared as per the supplier instructions and left to cure directly in sample mounts for one week. Once cured, the samples were removed from the mounts, coated with West System 105 epoxy resin (with West System 206 slow hardener) and transferred into clean sample mounts making sure that the end faces of the cylindrical samples remained free of epoxy. Once the epoxy was fully cured, the mounted cement samples were ready to be tested in the experimental apparatus described above. Photos of the raw material as well as prepared cement samples are shown in **Figure 6**.

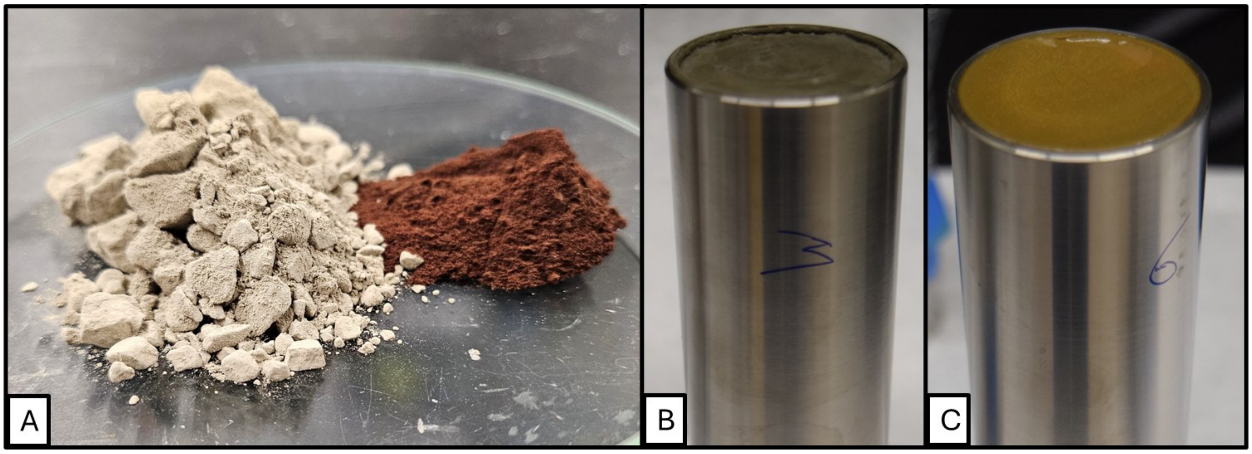

Figure 5: (A) Type 10 portland cement and BARAD-658™ additive; (B) Sample mount containing cement only (no additive); (C) Sample mount containing cement with BARAD-658™ additive.

SEDIMENTARY MATERIALS

The sedimentary material chosen for this work was the local sediment from a gas-exploration field in Alberta, Canada. A representative sample was collected from the field which was washed and dried before being sent for characterization. The sample composition is approximately 70% silt and 30% clay. The sedimentary material was combined with high purity silica sand on a weight basis, starting with 0:100 sediment and sand and increasing the sediment content in increments of 20 wt. % to a final mixture of 100:0 sediment and sand. All mixtures were tested in the approximately fully hydrated state. For consistency, 30 vol. % deionised water was added to each sediment and sand mixture immediately prior to testing.

The prepared samples were added to modified sample mounts which had been fitted with 200 mesh stainless steel screens. The material was compacted manually during filling using a balance to achieve consistent packing from sample to sample. Finally, a top filter element composed of 5 μm sintered stainless steel was added to prevent solids from entering the low-pressure vent lines during testing.

Due to the tendency of water to drain from the material if left for prolonged periods of time (this effect was faster for higher sand-content samples), sedimentary material samples were tested immediately following sample preparation. Photos of the sedimentary material as well as the sample preparation process are shown in **Figure 6**.

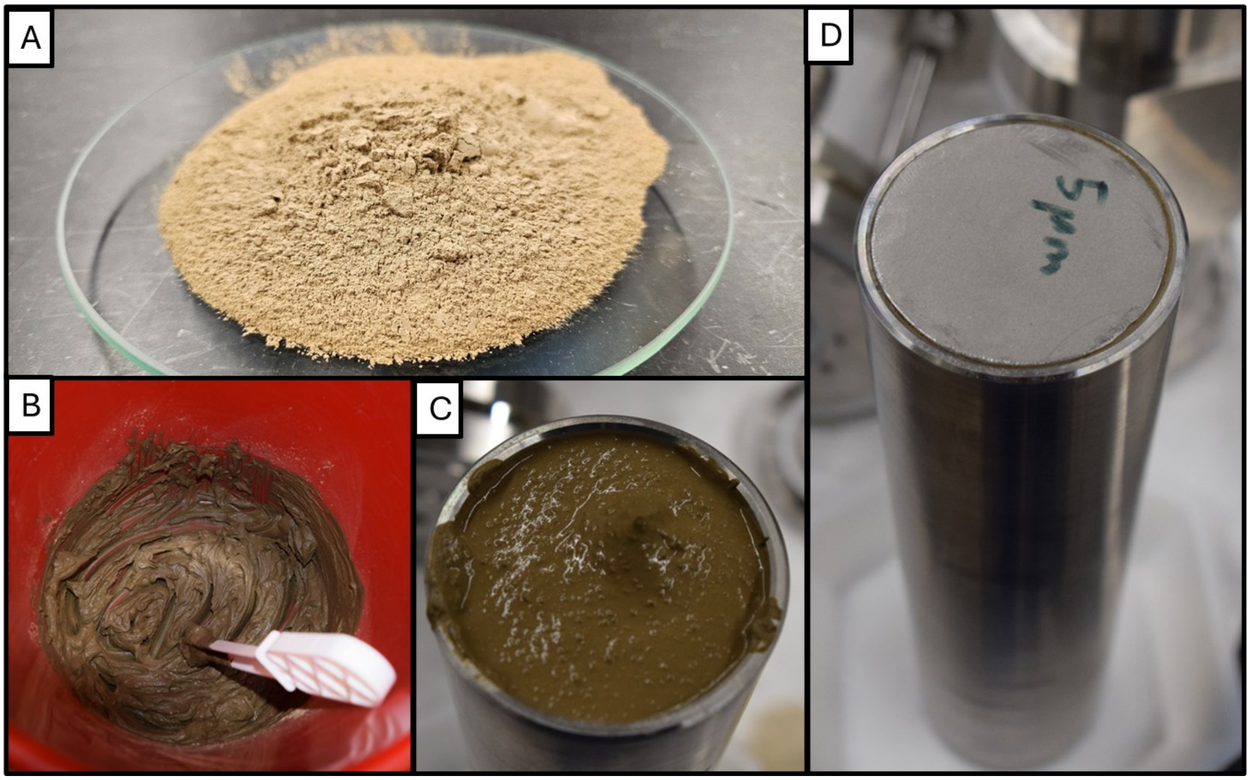

Figure 6:. (A) Sedimentary material obtained from a gas exploration field in Alberta, Canada; (B) Sedimentary material mixed with sand and hydrated; (C) Material loaded into modified sample mount; (D) Assembled sample mount containing sedimentary material and topped with 5 μm filter element.

SALT CORES

Salt cores have been provided by different geologic research groups across Canada. The source sites and suppliers include:

- Cores from the A-2 salt in southern Ontario have been obtained from the salt mine in Goderich, ON and provided to CNL by a representative of the Ontario Geologic Survey (OGS). Samples were drilled as NQ sized cores and used directly.
- Salt from the Prairie Evaporite have been provided through a collaboration with the Geologic Survey of Canada (GSC). Samples were sourced from the Nutrien mines located in Lanigan, Saskatchewan, and Allen, Saskatchewan. Prairie Evaporite cores were larger HQ size and were prepared for analysis by CNC milling. Milling was intended to enable careful dimensional control of the specimens without the usual abrasive chipping normally experienced using coring bits.

A summary of the salt cores and photos are given in **Table 1** and **Figure 7** respectively.

Table 1: Salt cores prepared for system commissioning.

ID	God-01-01	God-04-01	Lan-02-06	All-01-03	Bottom of 4th Lift	A-2 Salt Lower Contact
Source	Goderich mine (ON)	Goderich mine (ON)	Lanigan mine (SK)	Allan mine (SK)	Goderich Mine (ON)	Goderich Mine (ON)
Description	Clean halite	Inclusion - bearing halite	Inclusion - bearing halite	Clean halite	Inclusion - bearing halite	Inclusion-bearing halite

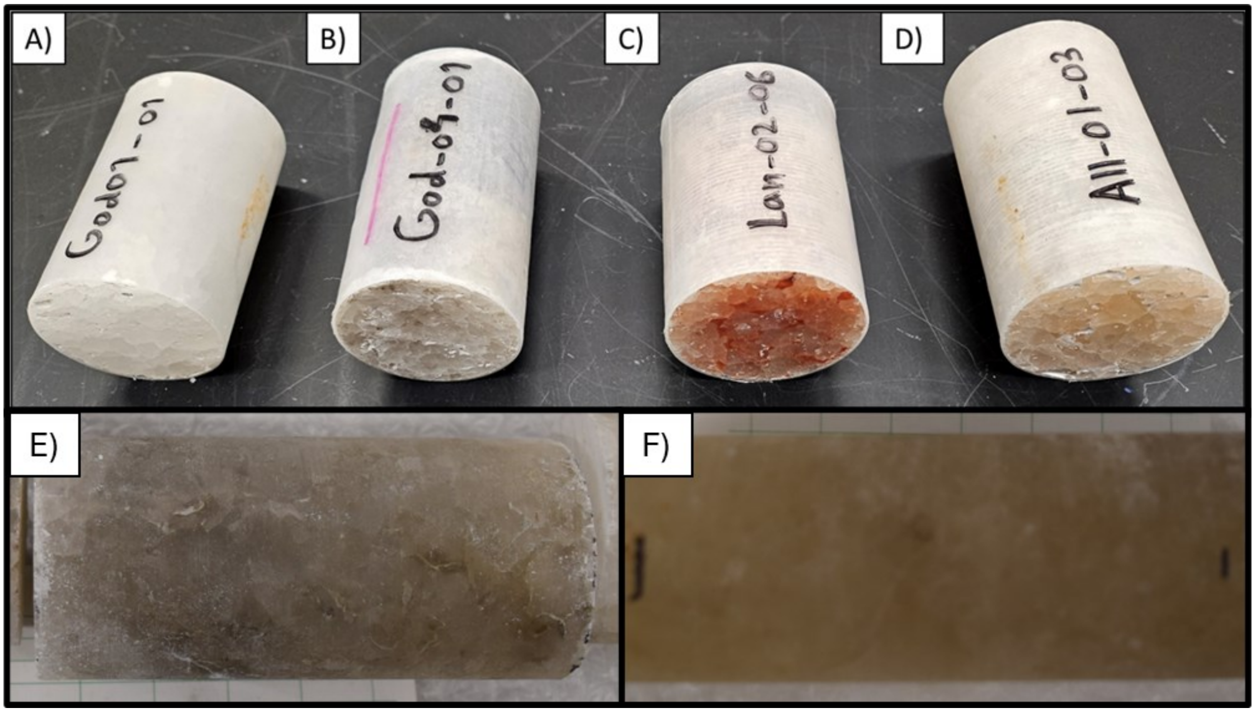

Figure 7: Core specimens prepared for system commissioning. (A) - God-01-01, (B) God-04-01, (C) Lan-02-06, (D) All-01-03, (E) Goderich mine bottom of 4th lift, and (F) A-2 salt lower contact (230 m/755 ft depth).

Preliminary Results & Discussion

WELL-COMPLETION MATERIALS WITH HYDROGEN

Graphs of two cement samples are shown without and with the BARAD-658™ additive in **Figure 8** and **Figure 9**, respectively. Tests were run overnight and the first hour of data is shown. As fluid leakage is a pressure-dependant process, the rate of pressure decay decreases with time as the differential pressure decreases. During the first hour, the rate is approximately linear and represents the differential pressure of interest for a real cavern scenario. The slope of the curves is proportional to the rate of hydrogen pressure decay on the HP side. This corresponds to the permeation rate of hydrogen through the cement samples.

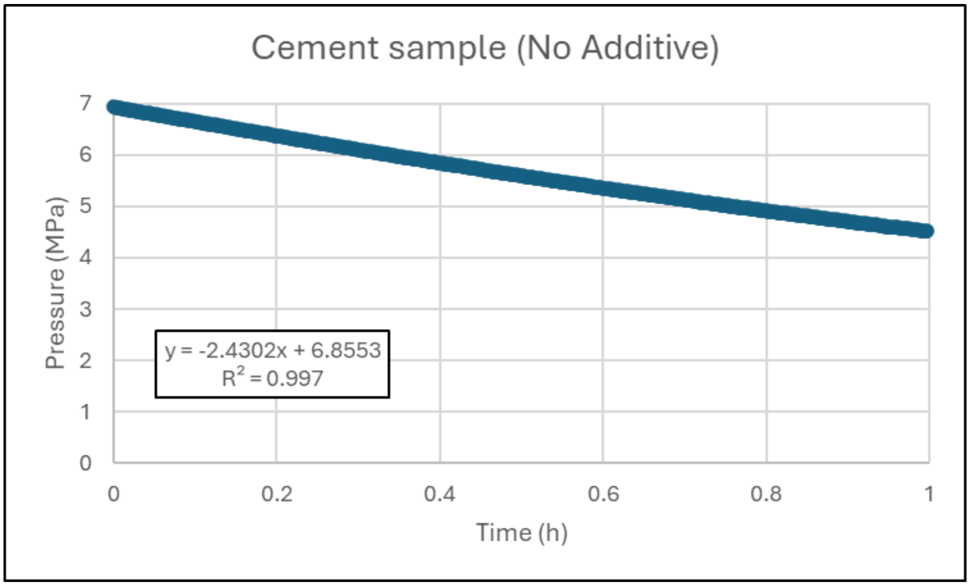

Figure 8: Pressure measured as a function of time at the HP side for a cement sample without additive obtained using the uniaxial microcolumn system.

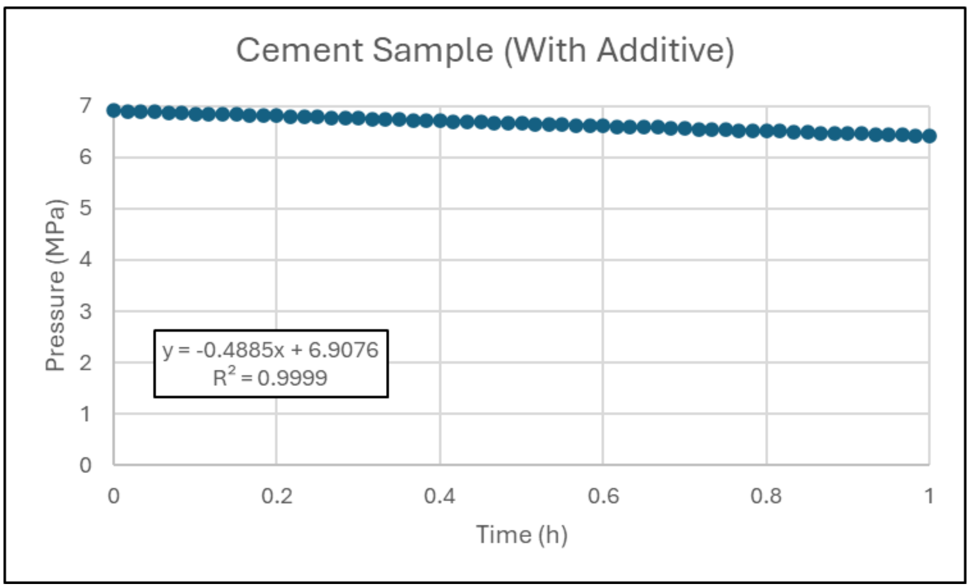

Figure 9: Pressure measured as a function of time at the HP side for a cement sample with additive obtained using the uniaxial microcolumn system.

The data shown in Figure 8 and Figure 9 can be used to model the leak behaviour which can be expected for a given cavern based on the cavern-wellbore interface geometry. Additional tests need to be performed to confirm this result, but it seems likely that the increased moisture-retention resulting from the use of the BARAD-658™ additive improves the hydrogen barrier characteristics of the cement used in well completions.

SEDIMENTARY MATERIALS WITH HYDROGEN

Figure 10 shows graphs from the sediment and sand mixtures. At 100 % sand, hydrogen migration was not slowed down in any measurable way. As the sediment (clay) content increased, an equilibrium differential pressure across the sample became evident in the data. With increasing sediment content, the rate of migration decreases, which is seen as a decreasing initial slope in downstream pressure measurement (orange dashed lines in the below graphs). This effect is most prominent above 60 % sediment, meaning that subsurface strata surrounding gas storage wellbores high in clay could be expected to resist hydrogen migration if there is a low-clay pathway available in the event of a leak from a cavern. Such experiments can be easily adapted to study a wide variety of sedimentary materials to aid in site characterization and leak detection and mitigation planning.

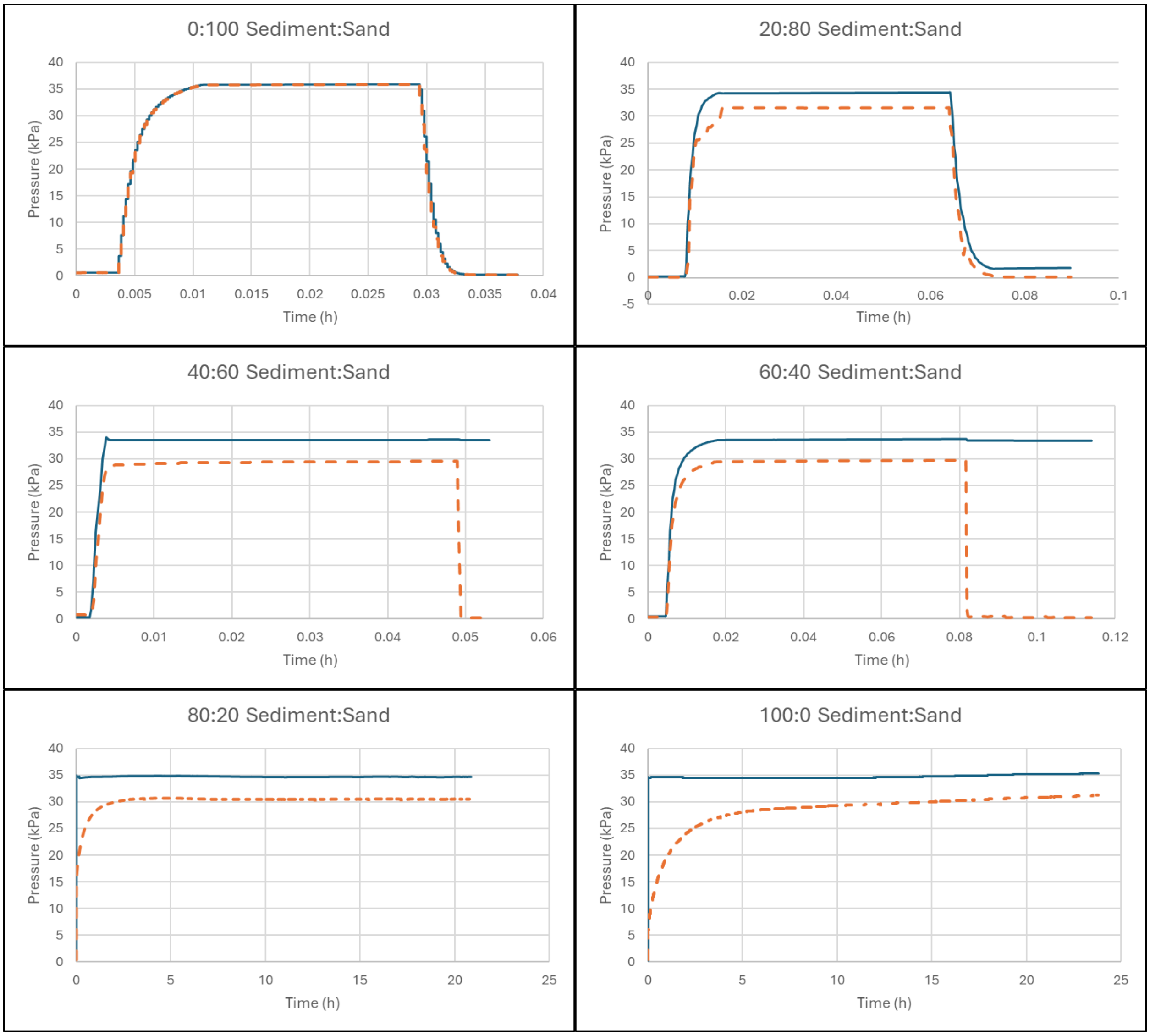

Figure 10: Pressure measured at the LP side for various sediment:sand mixtures as indicated in the figures obtained using the uniaxial microcolumn system. The blue lines represent the pressure upstream of the sample, whereas the orange dashed lines represent the pressure downstream of the sample. Note: The sedimentary material is identified as "Clay" in the above figure, as this is the primary constituent of the sediment.

SALT CORES WITH HYDROGEN

Permeation experiments were carried out on four of the six specimens listed in Table 1. The two specimens from Saskatchewan mines exceeded the maximum diameter for triaxial core holder samples. In every case, it was difficult to seal the core in the core holder as the samples were between 45-47.5 mm. Even when all components of the system passed leak checks and a seal around the core was achieved, still the pressure data was not repeatable from test to test (even for the same sample).

For the cases where a seal was obtained and hydrogen permeation was observed, a time-dependence of permeability was detected based on the amount of time the specimen was exposed to confining pressure. **Figure 11** shows the trend observed in two different days. The following observations should be highlighted:

- The Day 1 (data with blue dots) was taken immediately following sample installation:
    - Application of 8.25 MPa (1,200 PSIG) argon confining pressure, and system leak checking.
    - 3.5 MPa (500 PSIG) hydrogen pressure was applied to the HP side of the sample and the LP side was monitored for pressure increase.
    - The initial increase to ~35 kPa (5 PSIG) can be attributed to the specimen shifting in the apparatus, but thereafter, the pressure increases steadily over the next hour.
- The Day 2 (data with orange dots) is an identical experiment run approximately 24 hours later:
    - The specimen had been left under confining pressure overnight.
    - The behaviour is significantly different, showing only a small passage of hydrogen over a two-hour period.

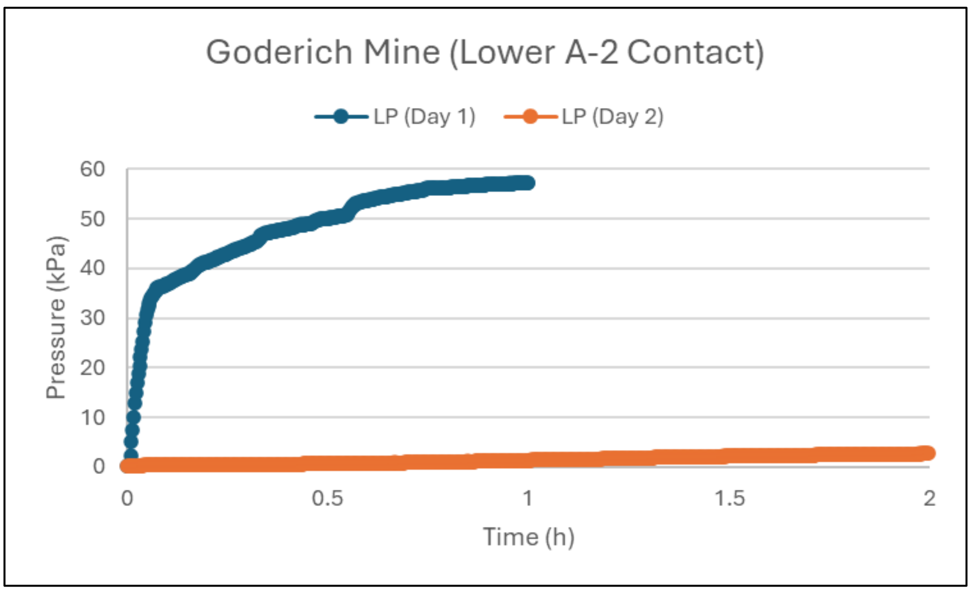

Figure 11: LP side pressure reading on Day 1 vs. Day 2 of confining pressure being applied to the Goderich Mine A-2 salt sample obtained using the triaxial core holder.

The trends shown in **Figure 11** are likely related to the ability of salt to flow, or "self-heal" under applied loads. This effect is well-known and must be accounted for during salt cavern operation, as geologic stresses are constantly working to close the cavern in on itself, and some minimum pressure of stored fluid is required to counteract this.

Additional efforts are in progress to assess other sealing methodologies to improve the reliability of the triaxial core holder for a wider range of sample diameters.

Conclusions

The uniaxial microcolumn system and triaxial core holder were able to provide useful hydrogen migration and permeation data on well-completion, sedimentary, and cavern wall materials.

The BARAD-658™ additive reduced the hydrogen permeability of type-10 portland cement by retaining additional water in the cement matrix and reducing the presence of void space. Leaks from a properly cased wellbore will likely be too small to measure given the large cavern volumes. Nonetheless, hydrogen leaks need to be avoided from a safety and economic aspect and further testing and analysis is required.

The composition of the surrounding sediment will inform the most likely leak pathway of potential cavern leaks and where to position hydrogen leak detectors. Each potential gas storage site will need to be characterised separately when attempting to model the possible leak pathways. Additionally, there are other aspects which are not well understood yet, such as sub-surface dwell time and potential hydrogen interactions (biological and chemical), which would impact the effect of a leak on the environment.

CNL, with its hydrogen expertise and research facilities, is open to collaborate with groups interested in salt cavern development for hydrogen storage. CNL has already contributed to the creation of a supplement to the CSA Z341 standard for underground hydrocarbon storage to address the unique properties of hydrogen [9]. Additional research will help to identify any remaining gaps in the standard in terms of testing and characterization requirements.

Acknowledgements

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