Feasibility of using distributed chemical sensing for CO2 leakage monitoring

DigiMon

Digital monitoring of CO_2 storage projects

Prepared by Dr. Tiziana C. Bond

Lawrence Livermore National Laboratory

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Scope

The feasibility study focused on the development of new fiber for distributed chemical sensing (DCS) that will allow direct detection of CO_2 leakages in the environment. This is particularly important for monitoring well integrity for carbon capture and storage (CCS), to provide early warning for an incoming well failure and potential CO_2 leaking through it. We proposed using optical spectroscopy in optical fiber for direct detection of CO_2. The main approach is based on Raman interrogation within gas-filled Holey Fibers (HoFs), so that the location and concentration of the gases would be provided simultaneously via backscattering. Additionally, Infrared (IR) Absorption Spectroscopy could also be used, and the architecture would be more discrete since interleaving with standard solid core fibers and Fiber Bragg Grating (FBG) sections is required to enable reflection to the I/O controls. The possible Raman length or the IR numbers of sections would be defined based on signal to noise ratio. The optical spectroscopy methodology would overcome current roadblocks to CCS, as fiber optics will allow for CO2 (and other gases) detection in wells with direct in-situ measurements of concentration along with other important parameters such as temperature and pressure as the baseline of environment background.

We were able to assess commercially available IR/Raman hollow core fiber and demonstrated detection of CO_2 through them in our controlled environment setups. We have also established the ability of drilling precisely with pulsed femtoseconds (fs)-lasers side holes to enable penetration of CO_2 into the hollow core fiber and reduce diffusion rates. Open joint collars were also explored having a double functionality: splicing to solid core fiber critical for field deployment and creating gas ingress locations. Both diffusion-only and pressurized fiber systems have been constructed following numerical simulations in COMSOL based semi-hybrid optical /fluido-dynamics models. FBGs have been identified, procured, and characterized but not yet integrated. Along the work we leveraged internal modeling/design, photonics/laser characterization, optical fiber fabrication, and AM lab capabilities to design, develop and test in-house components or assemblies. Our results indicate the critical potential that the HoF would have in the direct detection of CO_2 downhole.

Revision

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1 Background and Research Objectives

Commercialization of carbon capture and storage is limited by the perceived risks of long-term CO_2 storage. Wellbore leakage tops the list of these perceived risks. Wells are a direct, engineered path from the CO_2 storage reservoir to the surface, piercing the intervening sub-surface layers. Properly constructed wells provide a virtually impervious barrier to any unintended subsurface transmission. However, damage during well construction or CO_2 injection can allow CO_2 to escape from the reservoir into drinking water aquifers and the atmosphere. The ability to ensure the long-term integrity of such wells is vital to ensuring the success of any CCS operation. Therefore, needs exist for robust monitoring at a carbon storage site to detect, locate, and quantify the migration of CO_2 and native storage formation fluids in the subsurface within and above the storage complex main seal. Distributed fiber optic sensor technology has surfaced as an innovative means of monitoring physical parameters in wellbores and other subsurface environments including temperature, pressure, and acoustics. Fiber optic-based monitoring technology is currently at a high Technology Readiness Level (TRL) as it has been successfully commercialized and field-deployed for a broad range of subsurface measurements including Distributed Temperature Sensing (DTS), Distributed Strain Sensing (DSS), and Distributed Acoustic Sensing (DAS)[i,ii,iii,iv,v] . Such techniques provide indirect information about subsurface chemistry through integration with advanced physics-based models and machine learning / artificial intelligence methods, but the highest value information can only be obtained through direct chemical sensing technologies. Distributed chemical sensing is an alternative fiber optic-based technology holding tremendous promise for subsurface application, but also one that is relatively immature. Current leading DCS approaches integrate functional sensor layers with optical fibers to generate measurable responses to chemical analytes of interest[vi]. Despite promising results in a controlled laboratory environment, inherent technical challenges to field deployment have recently emerged including: (1) Insufficient selectivity to analytes of interest (e.g. CO_2) given complex chemistries of the subsurface. (2) Instability of sensing layer functionalized optical fibers in direct contact with subsurface analytes. (3) Difficulties with scaled manufacturing of sensing layer functionalization techniques with optical fibers. Accelerated progress of DCS to true field deployment requires alternative sensing modalities which overcome the challenges associated with engineered functional sensor layers via their elimination.

Our vision is a system that will enable simultaneous, real-time detection of concentrations, pressure, and temperature along the entire well by intermittently embedding segments of slotted hollow-core fiber and Bragg gratings within an optical fiber. The system would allow gas properties to be measured by infrared or Raman spectroscopy, wavelength shift on return signal from the sensor segments, or optical time-domain reflectometry (OTDR). The basics of the concept are drawn in Figure 1. With Raman interrogation, the Stokes shifted signal is backscattered in all direction and is reflected with a very specific wavelength shift that can be temporally mapped to location. With IR combined with OTDR, the blue and red pulses tuned at the absorption and slightly off the absorption line of CO2, so after passing a section with CO2 the blue pulse is reduced and bounced by FBGs providing time-space mapping at the external well controls.

With a different FBGs (tuned to the red pulse wavelength) the reflection can be used to evaluate environmental conditions and remove any confounding effect.

Enabled by recent patented advances in photonics-based advanced manufacturing and novel optical fibers, we proposed to address this important need within the area of subsurface chemical sensing by demonstrating an alternative DCS technology. More specifically, ultrafast fs laser processing combined with engineered hollow-core (photonic crystal) optical fibers would allow periodic gas phase access to the hollow core for a direct and distributed spectroscopic analysis via near-IR and Raman techniques. The limitations of sensing layer based DCS techniques would also be overcome as follows: (1) Near-IR and Raman spectroscopy methods provide selective signals to specific chemical analytes. (2) Enhanced stability in the subsurface by eliminating the requirement for thin film-based sensing layers. (3) Reel-to-reel laser processing techniques readily scaled to multi-km length scales of optical fibers. Advantages of the proposed technique include the ability to use fs laser processing to optimize the periodicity, physical configuration, and optical characteristics of gas access channels to the optical fiber central air region. In addition, the same processing technique can produce additional in-fiber devices (Fiber Bragg Gratings, Fabry Perot Interferometers, etc.) as needed to optimize sensitivity and spatial characteristics of distributed measurements (range, resolution, etc.). The flexible method can even produce multi-parameter functionality such as temperature, strain, and acoustic measurements. Multi-parameter functionality is of unique value in cases where subsurface chemistry is highly sensitive to temperature and pressure, and in cases where both chemical and physical parameters are of interest to inform subsurface modeling and real-time monitoring practices.

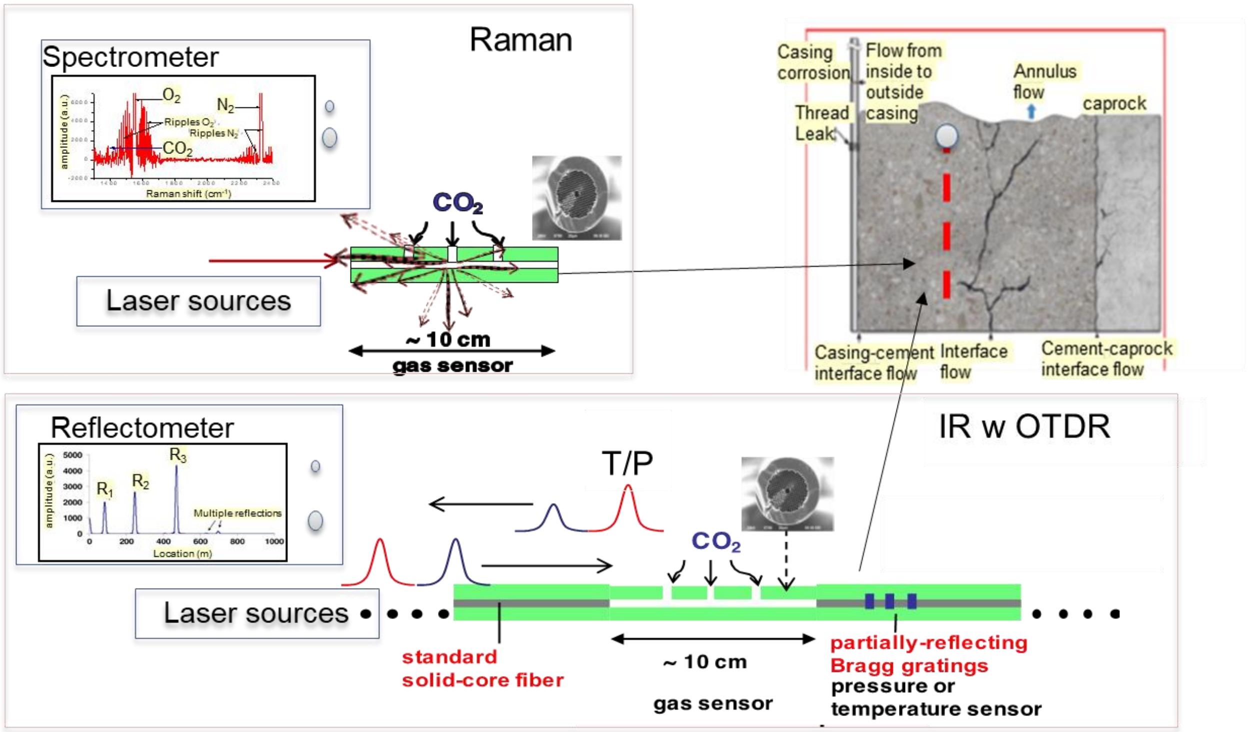
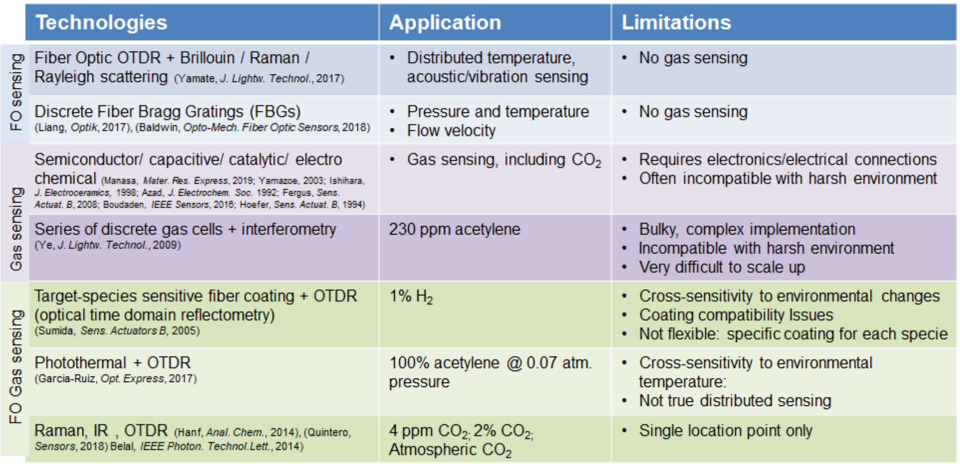


Figure 1. CO2 detection by spectroscopy in slotted HoF aided by FBGs for multipoint detection.

Necessary survey of literature and state-of-art revealed limited body of work on fiber optic gas sensing either using Raman spectroscopy with photonic crystal fiber to detect CO_2 or distributed gas sensing via sensitive coatings or indirect environmental effects measurement (Table I)

Table I. Summary of current work on fiber optic gas sensing



	Technologies	Application	Limitations
FO sensing	Fiber Optic OTDR + Brillouin / Raman / Rayleigh scattering (Yamate, *J. Lightw. Technol.*, 2017)	• Distributed temperature, acoustic/vibration sensing	• No gas sensing
	Discrete Fiber Bragg Gratings (FBGs) (Liang, *Optik*, 2017), (Baldwin, *Opto-Mech. Fiber Optic Sensors*, 2018)	• Pressure and temperature • Flow velocity	• No gas sensing
Gas sensing	Semiconductor/ capacitive/ catalytic/ electro chemical (Manasa, *Mater. Res. Express*, 2019; Yamazoe, 2003; Ishihara, *J. Electroceramics*, 1998; Azad, *J. Electrochem. Soc.* 1992; Fergus, *Sens. Actuat. B*, 2008; Boudaden, *IEEE Sensors*, 2016; Hoefer, *Sens. Actuat. B*, 1994)	• Gas sensing, including CO_2	• Requires electronics/electrical connections • Often incompatible with harsh environment
	Series of discrete gas cells + interferometry (Ye, *J. Lightw. Technol.*, 2009)	230 ppm acetylene	• Bulky, complex implementation • Incompatible with harsh environment • Very difficult to scale up
FO Gas sensing	Target-species sensitive fiber coating + OTDR (optical time domain reflectometry) (Sumida, *Sens. Actuators B*, 2005)	1% H_2	• Cross-sensitivity to environmental changes • Coating compatibility Issues • Not flexible: specific coating for each specie
	Photothermal + OTDR (Garcia-Ruiz, *Opt. Express*, 2017)	100% acetylene @ 0.07 atm. pressure	• Cross-sensitivity to environmental temperature: • Not true distributed sensing
	Raman, IR , OTDR (Hanf, *Anal. Chem.*, 2014), (Quintero, *Sensors*, 2018) Belal, *IEEE Photon. Technol.Lett.*, 2014)	4 ppm CO_2; 2% CO_2; Atmospheric CO_2	• Single location point only

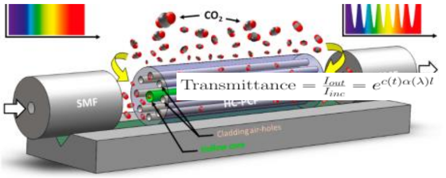
Our critical target has been combining sensitivity/selectivity of Raman/IR with true distributed sensing for multipoint optical detection and scale-up capability. We have previously demonstrated the capability of IR and/or Raman interrogation in HoFs of several volatiles in small volumes on short fiber stocks[vii]. As light and chemicals overlap along the same hollow core, the increased light-matter interaction is increased by several order of magnitude enabling ppm level of detection[vii]. We have also developed compact NIR systems which setup can be extended to HoFs gas detection as complimentary technique[viii]. FBGs have also been used for stress/pressure (S/P) and temperature (T) sensor quite frequently by the oil industry: the reflected signal is specific to a wavelength and any S/P/T change will be reported in the wavelength shifts at the reflected output. Herein, they can be used for a complementary function, as S/P/T sensor but also as temporal detector as the arrival time of the reflected signal is correlated to the distance travelled. Within this proposal, our research objectives were: to demonstrate CO_2 detection with commercially available HoF via diffusion or pressurization; enable rapid detection via optimized positioning of slotted apertures along the fiber side or at their edges; interleave with FBGs for spatial location and environmental monitoring when used with IR; optimize splicing with solid core fiber if needed for deployment along wellbore 100s-1000s meter stretches.

2 Scientific Approach and Accomplishments

As our main concept relies on intermittently embedding segments of Slotted Holey Fibers (S-HoFs) to allow for IR/Raman spectroscopy (chemical fingerprinting) with solid core fiber and FBGs, we proceeded with design, fabrication, and characterization of commercially available hollow core fiber HC-800-2/HC-1550-2 (NKT Photonics) for Raman and IR respectively.

2.1 Modeling & design

We used COMSOL to determine the optical modal properties. 2D optical mode distribution validated single mode operation with optical parameters was confirmed with the fiber providing company (Figure 2a). Due to the periodic structure of the cladding, light is confined nearly entirely to the hollow core (filling factor f=99%), allowing the gas and light active volumes to fully overlap, making the fiber an extremely sensitive sensor. The light can interact with the gas as if it were propagating through free space. The relative sensitivity defined as r = f n_{gas}/n_{eff} which compares the light propagating through an optical fiber (n_{eff} is the mode effective index) with light propagating through free space is ~1[ix]. This relaxes the absorption analysis to a 1D Lambert-Beer law where the transmittance T for a length l of the fiber is expressed as $T(\lambda) = e^{-rlCr(t)\alpha(\lambda)}$ where C(t) the average relative gas concentration and $\alpha(\lambda)$ absorption coefficient derived from HiTRAN database[x]. When paired with diffusion models (Figure 2b), this 1D model sufficed to validate literature experimental results (Figure 2c) and helped tremendously in accelerating any following configuration optimization[xi], given a lighter COMSOL hybrid model with reduced memory requirements. The advanced design included slotted holey fibers; we derived that an optimal ratio of number of sections to total exists. This factors in greatly as our fabrication guidance, i.e. 3 holes or separation of ~3 cm for over 10cm allow reduction of filling rates from minutes to seconds length (Figure 2d). A larger number of holes or reduced separation does not improve diffusion rate significantly. Optical 2D propagation models were fully investigated but the memory requirements to properly mesh the fiber even by exploiting symmetries were practically insurmountable.



$$\text{Transmittance} = \frac{I_{out}}{I_{inc}} = e^{c(t)\alpha(\lambda)l}$$

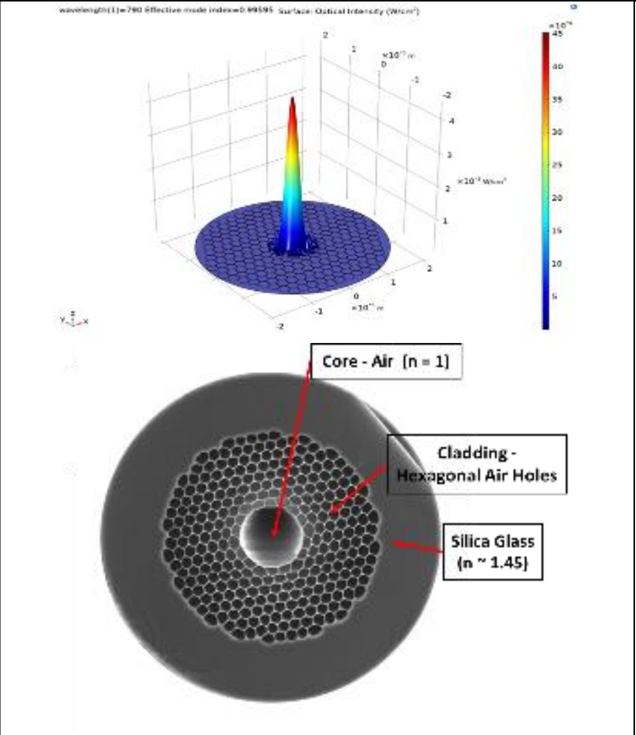


Figure2a: Optical model: mesh and optical mode for HC-800-2; SEM image of fiber cross section

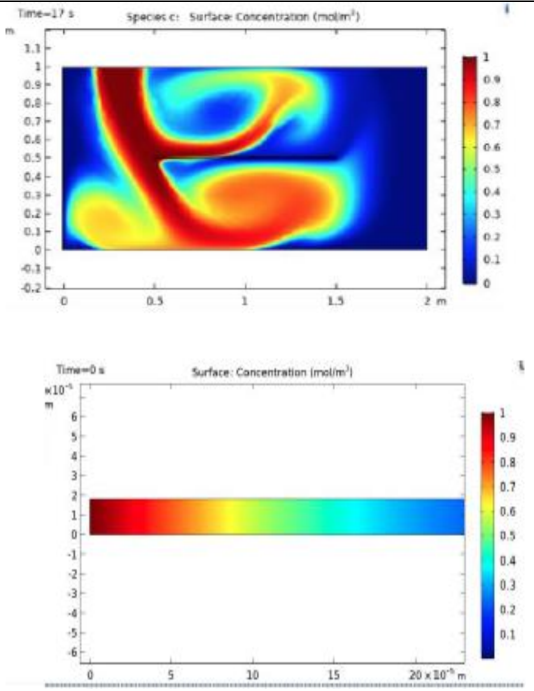


Figure 2b: Concentration model: fiber in chamber and zoom in over fiber at 0sec (2ports like in reference [Hoo])

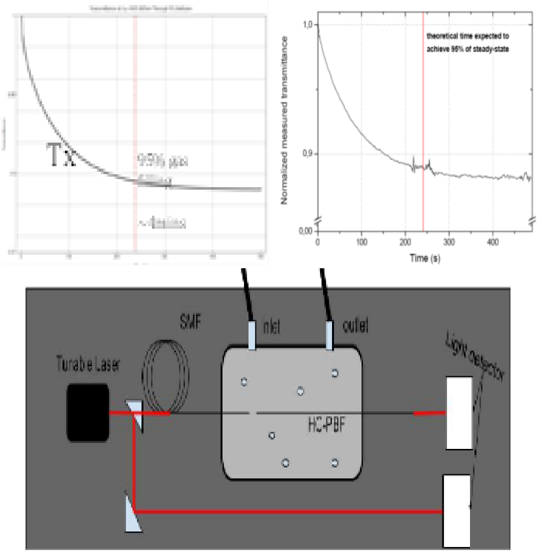


Figure2c. Combo model: simulated T(l) compared to data in diffusion experiment[ix]

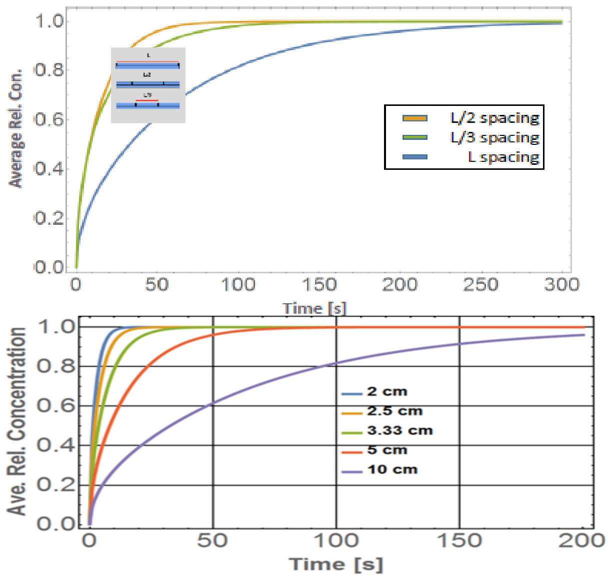


Figure 2d. Model of 2 slots in HoF (r=1um); reduced spacing decrease fill up time from minutes to seconds

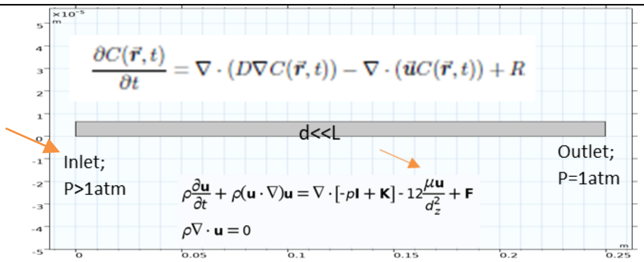


Figure 2e. Convection-diffusion and Navier Stokes equations fpor pressurized system in the schematic

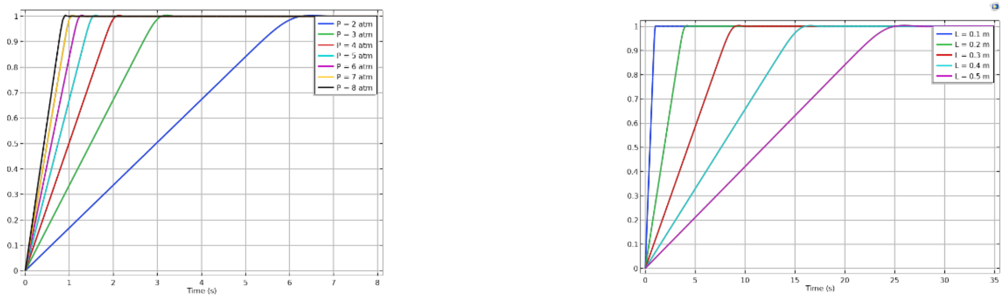


Figure2f. Gas filling dynamic: 1) varying ΔP for L=25cm. 2) varying L for ΔP= 1 atm; filling time is a few seconds

We have also started investigating pressurized conditions to estimate the improvement of fiber gas filling rates (Figure 2e). The theory underlying diffusion-based gas flow versus pressure driven gas flow is similar. Both types of simulations require the Navier-Stokes equations describing fluid flow be coupled to the Convection-Diffusion equations describing a diffusing species. However, the momentum term in Navier-Stokes equation is modified to include the shallow channel approximation term if its height is much smaller than its length like the ones of interest to us. In these micro-scale systems, there is significantly more drag due to the walls (Figure 2e). In the exemplary model, the fiber has a core diameter of 7.5 um and a length of 25 cm. Carbon dioxide flows throughout the fiber from the inlet, and the outlet is maintained at atmospheric pressure. The fiber is initially filled with nitrogen. As seen in the graph (Figure 2f), the filling time appears to decay exponentially as a function of inlet pressure. Regardless, even for a moderate pressure difference of 1 atm (inlet pressure of 2 atm), the fiber fills in about 6 seconds. That is a very fast filling time for a sizeable sensor. As expected, the filling time increases with length, though the filling time remains rather short even at a modest pressure difference of 1 atm (and inlet pressure 2 atm). This model is very useful to investigate the pressure differences that could be achieved in the lab that can relate to any fielding condition. We started looking at the combination pressurized system with slotted configuration, but it does not seem to improve significantly as we are already in the seconds filling time at least for this fiber lengths up to 50cm. Longer stretched might experience some improvement.

2.2 Fabrication and characterization

We have built several setups for Raman investigation at 785nm dedicated to diffusion processes studies (Figure 3a), and at 532nm where we focus on pressure driven flows (Figure 3b). The 785nm system has the advantage to reduce the fluorescence from background but it is weaker than Raman at 5332nm which instead suffers from fluorescence. Having excitation wavelength available will give as a way of downselecting the best performer. We also have an existing setup for IR absorption spectropsy approach at ~1570nm at which the CO2 is absorbing that we can combine with the FBGs we procured and were delivered only at end of the proposal term. The setup was used for measurements on independent projects. (Figure 3d) [xii]. Furthermore, FBGs at 870nm (CO2 Raman Stokes line) were procured as well to test them with Raman to boost back reflection as well and increase SNR).

The hollow core fiber HC-800-2, PMC-C-Green-26 HoF, and HC-1550-2 were procured and tested for throughput, but also cleaved into segment to validate the length dependent models and allow various novel fabrication tests. We were able to flow CO_2 in a controlled box at several pressures and measure CO_2 Raman signal dependence (Figure 3c). This is a very promising result since the Raman signal is very weak and we are far from optimized as we are limited in power and integration time: the power coupled into the HoF via the portable DeltaNu Inspector Raman gun is << 1mW, ~10cm^{-1} resolution, and we were limited to 1 minute integration only. Furthermore, additional filters have been installed to help cleaning up the from the signal and increase SNR of the CO_2 doublet (1276 cm^{-1} and 1293 cm^{-1})[x] that is right on the shoulder of the Raman emitted by the silica of the fiber itself. Currently dynamic testing is ongoing on 10cm long holey fiber to validate the COMSOL diffusion/convection model results. We have been modifying the system to collect data continuously while introducing CO_2 rather than measuring just at end of flow. The first data on uptake of CO_2 were taken for 2” and 6” hollow core fibers and are aligned with the simulation data (Figure 3c). The 532nm system is made in-house and compact, composed of OEMLaser DPSS laser at 532nm with very low jitter, high power up to 200mW, a compact Ocean Optics QE Pro spectrometer with ~10cm^{-1} resolution and a GloPhotonics micro cell made of a 532nm HoF and two compact gas cell at both ends for controlled pressurized experiments (Figure 3b). This setup that required specialized optics for the specific wavelength was just completed and checked for alignment and throughput operation.

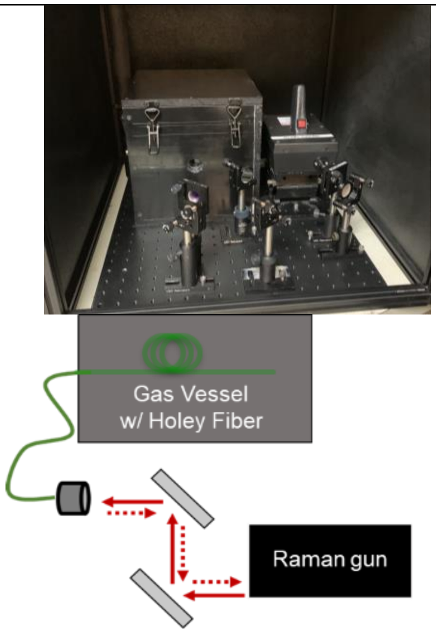


Figure 3a. Current Raman system at 785nm with a Portable Raman gun and schematic.

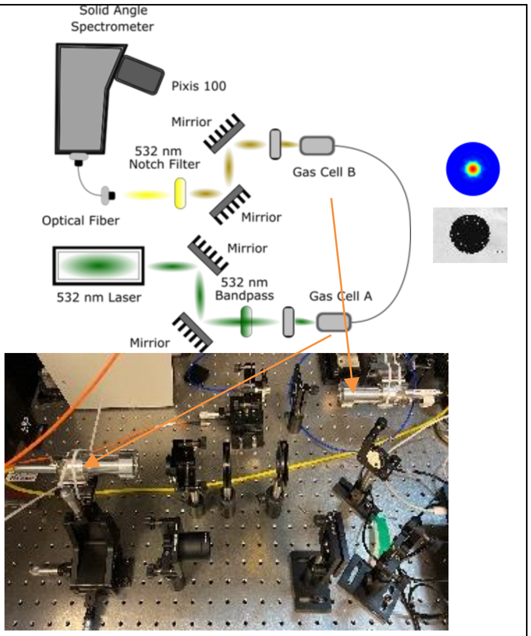


Figure 3b. 532nm Raman setup with Glophotonics pressurized system with PMC-C-Green-26 HoF.

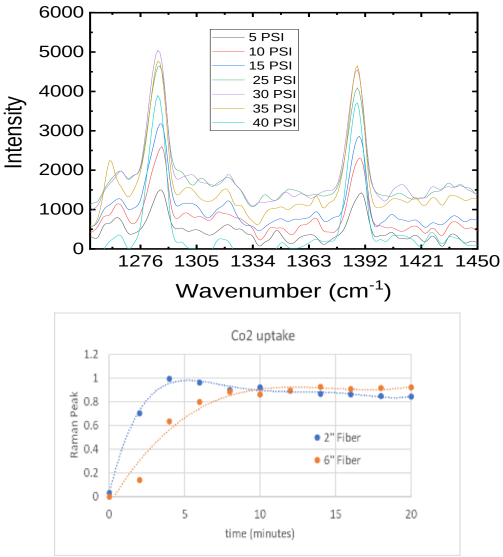


Figure 3c. Raman spectra for CO2 at different pressure and Co2 uptake for 2' and 6" fibers

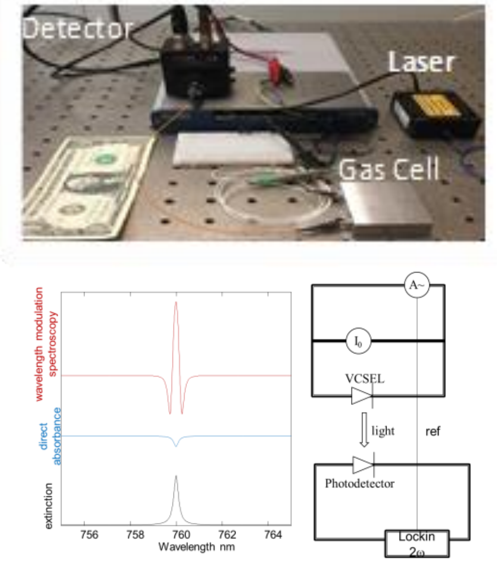


Figure 3d. Example of absorption spectroscopy setup and schematic of operation

We selected and procured Fiber Bragg Gratings to investigating enhanced back reflection of the Stokes shifted signal (~830nm). For their use will be critical to enable splicing of the fibers to HoF fibers. In general, this will be a necessary capability for economically deploy HoFs for Kms downhole or subsurface, as they are currently quite more expensive than standard telecommunication fibers.

Splicing or cleaving HoF is delicate since given the holey nature and the lattice morphology they could not be stressed otherwise the fiber collapses on itself and there is no significant throughput. Nevertheless, we were able to achieve splicing with the expertise of the fiber group at LLNL and manage losses of ~10%.

What is even more challenging is to create a join collar of only few 10s of um that could hold the ends of both the HoF and solid core fiber together and maintain a gap to enable gas ingress. First attempt: 2% power throughput measured at 800nm via the C-shaped collar splicing of 780nm transmitting solid core fiber to hollow core fiber (Figure 4a left). Although we successfully made the mechanical joint, it seems fragile and not able to sustain too much handling (easily breaks). We resorted to another simpler approach by just sliding the two ends into a mating sleeve that already comes with a large opening as an off-the-shelf item (Figure 4a right) – this setup guaranteed 50% throughput, but diffusion results are not yet clear as it seems the HoF might got occluded**.**

In parallel we have been working on fabrication of holes in the HoFs by using a femtosecond laser that would drill multiple holes along the fiber length on reel-to-reel setup. First tests are promising for continuing tuning the laser power, focal length, and positioning. With a 40fs pulsed Ti-Sa 800nm laser 100 µJ, 20x @ 0.40 NA Objective, 1 µm/s speed we were able to drill an array of 1-2um holes along 10cm fiber length (Figure 4b).

We are also experimenting another alternative by making in our fiber lab a C-shaped collar that not only would connect the solid core and hollow core fibers but would have a larger opening for gas ingress. This step will also help with integration with FBGs. We are about to put these technologies to the test.

Finally, for sake of completeness, we started investigating the effect of supercritical CO2 environment on the hollow core fiber morphology, resiliency by submitting them to the relevant high temperature and pressures and in presence of brine reach with supercritical CO2, exploiting our internal Hydrolab. Currently we are testing fibers by ramping to typical cap rock temperature (80C) and pressures (150Psi). IF the outcome is positive, we will pursue the investigation of CO2 spectroscopic detection in brine conditions.

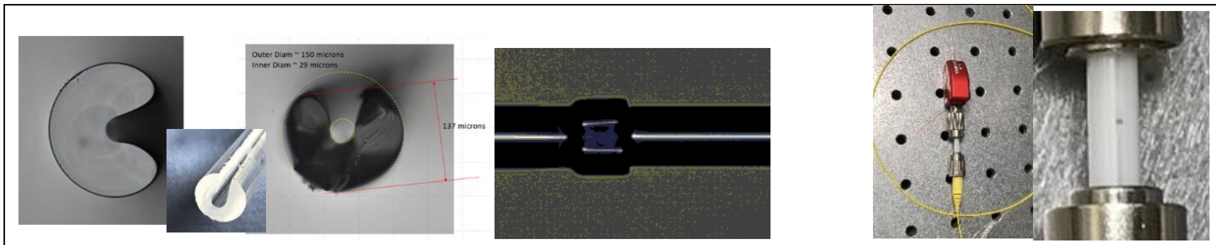
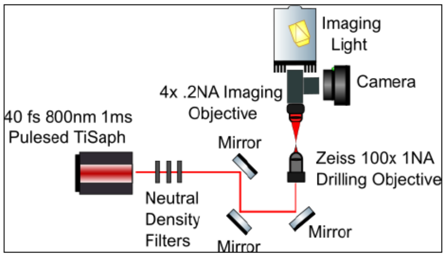
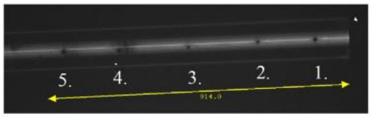


Figure 4a. (left) C-shaped preform out of a silica tube then drawn into a C shaped collar of ~60um that got spliced to the solid core and hollow ore fiber ends; (right) Images of splices with mating sleeves.





Hole #	Position from Edge	Drilling Speed	Power
1	100µm	1µm/s	81mW
2	275µm	1µm/s	81mW
3	485µm	1µm/s	81mW
4	700µm	1µm/s	220mW
5	900µm	10µm/s	220mW

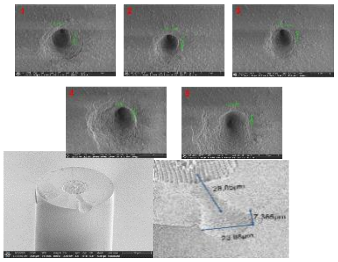


Figure 4b. (left) Setup for fs drilling; (center) Image of 5 drilled holes in HoF; (right) SEMs images of holes on the side and at cleaved facet showing etch depth control.

3 Conclusions

We have experimental evidence on the ability of using HoFs for the spectroscopic detection of CO2 and more understanding about their arrangement with solid core fiber and fiber gratings. We have been able to measure the CO2 at various pressures and built models demonstrating fill rates down to order of seconds to minutes, depending on section lengths, that we are currently validating. We have demonstrated fs-laser side-hole drilling for faster ingress of the gas in the HoF and the splicing of solid fiber core to HoF maintaining the integrity of the HoF. The loss overall throughput vs. HoF lengths and number of splices is a critical result for deployment and even if we measured a 3dB loss there is large room for improvement, in a proper engineering setup. We believe we demonstrated most of the key elements to prove this technology has potential and worthwhile pursuing increasing its TRL levels to make it deployable and economically sound. HoF are commercially available but still quite expensive per unit length, longer lengths being prohibitive because of lack of market consequently inhibiting the overcome of fabrication and technological limitations. At this stage it is unequivocable that we need hybrid solutions, interleaving longer length of solid core fiber with shorter HoF sections at specifc locations along the wells. If leak detection of CO2 in CCS could be proved valid, then a larger market would boost the HoF industry, reduce cost and making our system conop more cost-effective. We are conducting work on the area of environmental effects (temperature, pressure) on the fiber and eventually such as optical and mechanical property changes.

Some of the key challenges that will need to be resolved, include (1) possible spectral interferences and environmental noise that could mask the signal; (2) low signals and (3) microchannel impairments, (4) greying (ordarkening) of the fibers due to environmental effects such as radiation, temperature, pressure, optical dose, etc.; (5) physical robustness . We will address these challenges by (1) isolating the noise with laser modulation, multivariate analysis methods to help reveal complicated signatures, and spectral filters; (2) evaluate amplification schemes that might be easier with the complimentary NIR technique; (3) replace microchannels with open volume gaps at end of HoF sections to capture signal in transmission and reflection mode (FBG or mirror), (4) additional protection shielding around the opening and periodical photobleaching that is demonstrated to restore the fiber performances,(5) testing in our HydroLabs will provide the information we are seeking – furthermore special armoring will be required like done for other fiber deployment .

Longer term plans seek to enable highly sensitive CO2 detection in the deep subsurface with high selectivity, stability, and sensitivity. More specifically, the proposed program targets to demonstrate the following by the end of the project conclusion: (1) Successful detection of <1% CO2 in a complex gas mixture representative of subsurface environmental conditions at a length of >1km from the source and detector in a lab environment. (2) Successful demonstration of multi-point measurements of CO2, consisting of at least 5 individual sensor elements spaced no more than 5m apart on a single optical fiber. (3) Successful field validation in a shallow monitoring well for controlled CO2 gas release in the well. Ultimately, the proposed DCS technology is anticipated to be capable of selective monitoring of CO2 in complex subsurface conditions at levels <100ppm and multi-km depths with at least 1m spatial resolution. Because near-IR and Raman spectroscopic techniques are being utilized, future research can also enable multi-component speciation, detection, and quantification of additional analytes of interest in subsurface environments through wavelength division multiplexing with multivariate data analytics methods. The proposed technology can be applied to detect, locate, and quantify migration of CO2 and formation fluids within and above the storage complex main seal through surface deployment within monitoring wells.

Finally, and very importantly there is the delicate issue of the effective deployment of the fibers down the hole: for instance, if for offshore settings the fiber systems could be either attached outside the casing with centralizers or within the annulus, strapped to tubing, always providing the appropriate armoring protection, that could be either in metal or ceramic [xii]. Similar procedure that exists for other fiber based monitoring system will be followed and adapted to Among the questions to address are: 1. Isolation (protect form corrosive environment and sustain high pressure – high temperature).2. Avoiding any backflow (compartmentalize into segmented sealed sections; single point detection with end cap could be more feasible). 3) Onshore vs Offshore (CO2 escaping could cause catastrophic effect). 4) Certification of connections (completion engineer would need to verify even before the centralizer is implemented)

The results obtained in this work have been critical to continue our interaction within NORCE, and develop relationships with companies like Equinor, and Silixa at the hearth of the Digimon Monitoring Consortium leading the revolutionary effort for subsea CO2 sequestration.

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5 Publications, Presentations, and Patents

M. Heyrich et al., Internal LLNL SULI internships reports, 01/2021 and 08/2021

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