Development of hollow-core fiber optic gas sensors for CO_2 containment assurance

2024 FECM / NETL Carbon Management Research Project Review Meeting

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LLNL-AR-867410

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Lawrence Livermore National Laboratory

FECM FEW087 project overview (2nd year)

Task 1: Development of high-resolution induced seismicity hazard modeling tools

Budget: 300k (Y1) - **500k (Y2)** - 500k (Y3)

Research team:

- Matteo Cusini
- Kayla Kroll
- Nicola Castelletto
- Randy Settgast
- Joshua White

Collaborators (unfunded):

- Vidar Stiernström (Stanford)
- Matteo Frigo (Stanford)
- Eric Dunham (Stanford)

Task 2: Development of hollow-core fiber optic gas sensors for CO_2 containment assurance

Budget: 300k (Y1) - **500k (Y2)** - 500k (Y3)

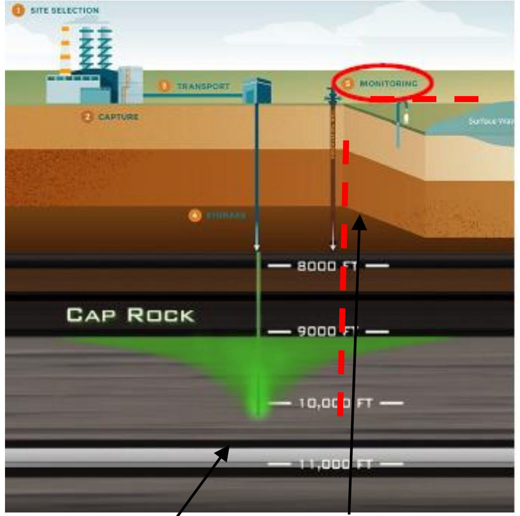
Research team:

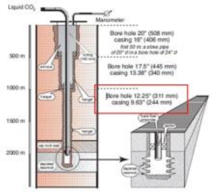
- Allan Chang
- Brandon Demory
- Sarah Sahota
- Jorge Arteaga (UCM PhD student)

Collaborators (unfunded):

- Ruishu Wright (NETL)
- Alex Shumski (NETL)
- Sayanti Ghosh (UCM)

Task 2: Hollow-core fibers for enhanced Raman detection of CO2 leakages in CCS

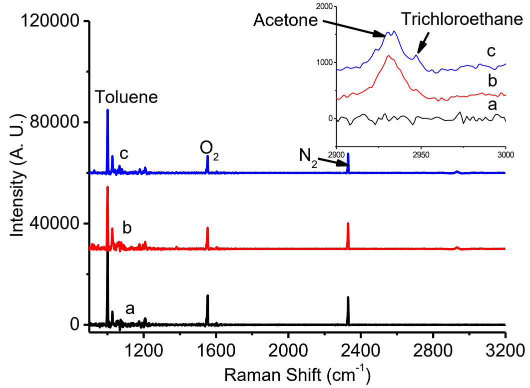




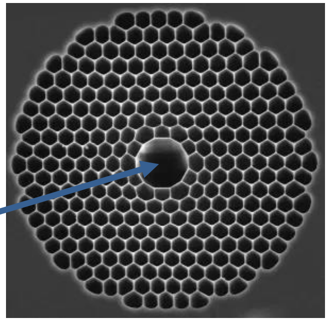
Deploy Holey Fiber with Raman or IR spectroscopy for Distributed Chemical Sensing

- Fiber-based distributed chemical sensing (DCS) is recognized as an alternative for in-situ monitoring of CO2 in storage reservoir and subsurface barrier system

- Raman and IR spectroscopy are standard **lab-based** techniques for measuring composition
  - Exploit our expertise and experience
  - Can detect multiple gases simultaneously (impurities)



- <u>Specialized</u> hollow-core photonic crystal fiber or **"Holey Fiber" (HoF)** to enhance detection
  - Exploit existing fiber industry and LLNL's fiber know-how



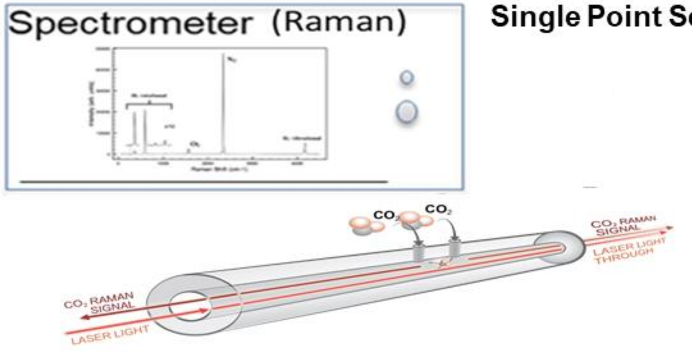
Light+Matter

Raman(/IR)+ HoF: broadband spectroscopy that fingerprints & quantifies gas species + no need for coatings that are selective and easily degrade with time and environmental factors

Gas detection by laser vibrational spectroscopy using slotted HoF aided by FBGs for multipoint detection

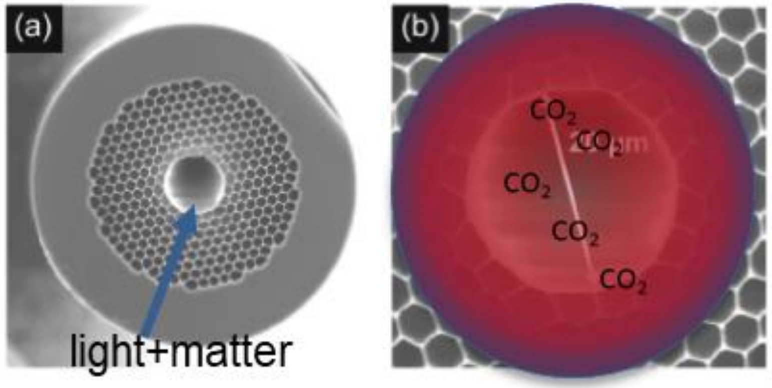
A quasi-distributed system integrating

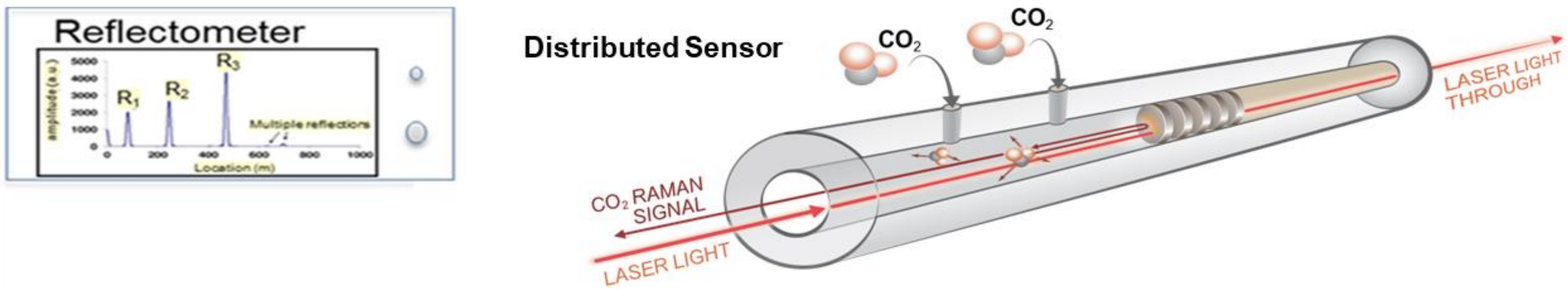
- Slotted Holey Fibers (S-HoFs) to allow for IR/Raman spectroscopy (chemical fingerprinting)
- Fiber Bragg Gratings (FBGs) for optical time-domain or frequency-domain reflectometry on return signal from the sensor segments; FBGs can also be exploited as P/T sensors



Single Point Sensor

Hollow core fiber where light-gas interaction is enhanced





Patent application
ROI IL-13495

Task 2: objectives and subtasks

Objective: develop distributed fiber-optic chemical sensing for direct detection of CO_2 gas leakages in the environment using holey fibers (HoFs)

Subtask 1 – Optimization of HoF slotted designs

- **Milestone 1.1a:** *CO2 limit of detection & uptake time for slotted HoF* *2^{nd} yr*
- **Milestone 1.1b:** Establish operation for additional gases (H2/CH4) 3^{rd} yr
- **Milestone 1.2a:** *Demonstration of fs-laser drilling over 1-3m HoF* *2^{nd} yr*
- **Milestone 1.2b:** *Environmental factor evaluation (mechanical/optical)* *$2^{nd}/3^{rd}$ yr*

Subtask 2 – Integration of HoFs with solid core fibers and FBGs

- **Milestone 2.1:** *Demonstration of HoF-solid core splicing* *2^{nd} yr*
- **Milestone 2.2:** *Production of splices with FBGs* *2^{nd} yr*

Task 2: objectives and subtasks (cont'd)

Objective: develop distributed fiber-optic chemical sensing for direct detection of CO2 gas leakages in the environment using holey fibers (HoFs).

Subtask 3 – System throughput analysis and light budget optimization

- **Milestone 3:** Analysis of HoF sensor system throughput in shallow wells 3rd yr

Subtask 4 – Harsh condition survivability determination and armoring

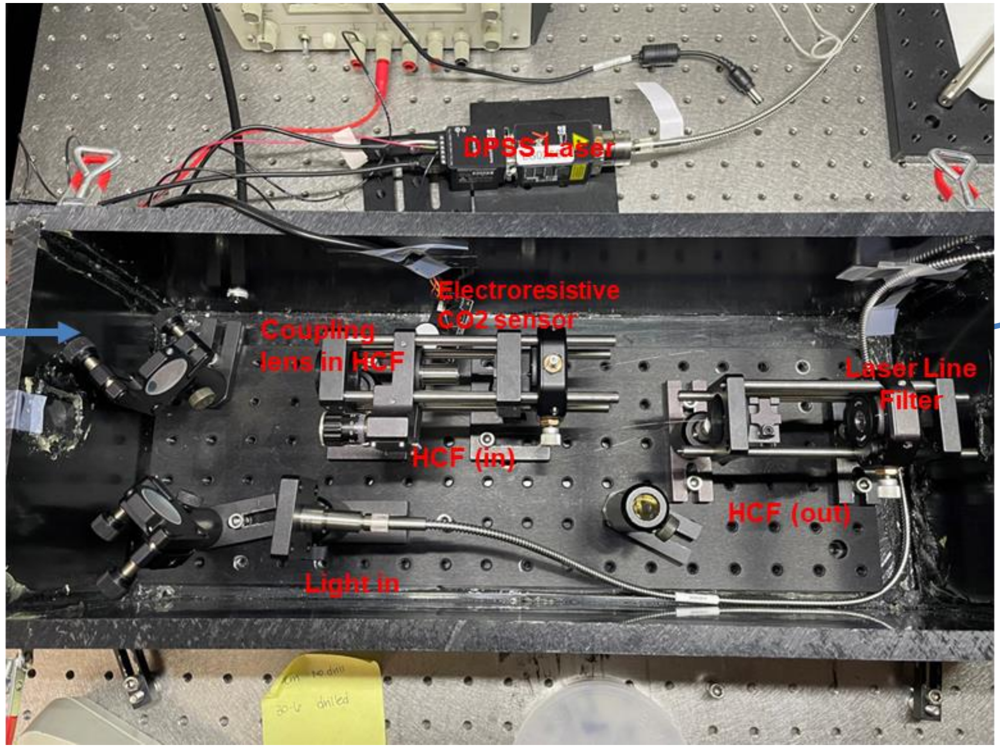
- **Milestone 4:** Integrity test and validation results at brine conditions 3rd yr

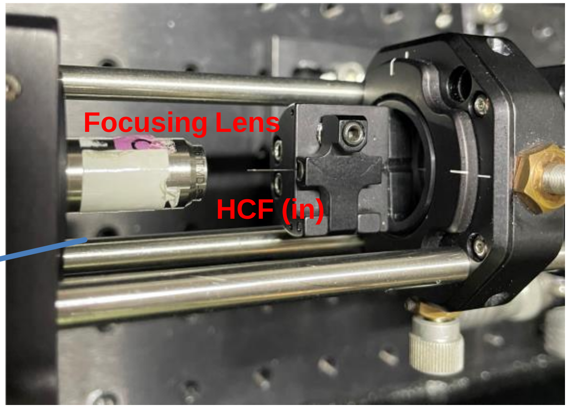
Subtask 1 – Optimization of HoF Slotted Designs

1.1a Raman setups for experimental evaluation of the technology

Diffusion-based gas Raman system at 785nm was significantly improved with better laser, spectrometer, alignment, and fiber coupling controls

CO_2
0.5% - 100%
mixed in N_2





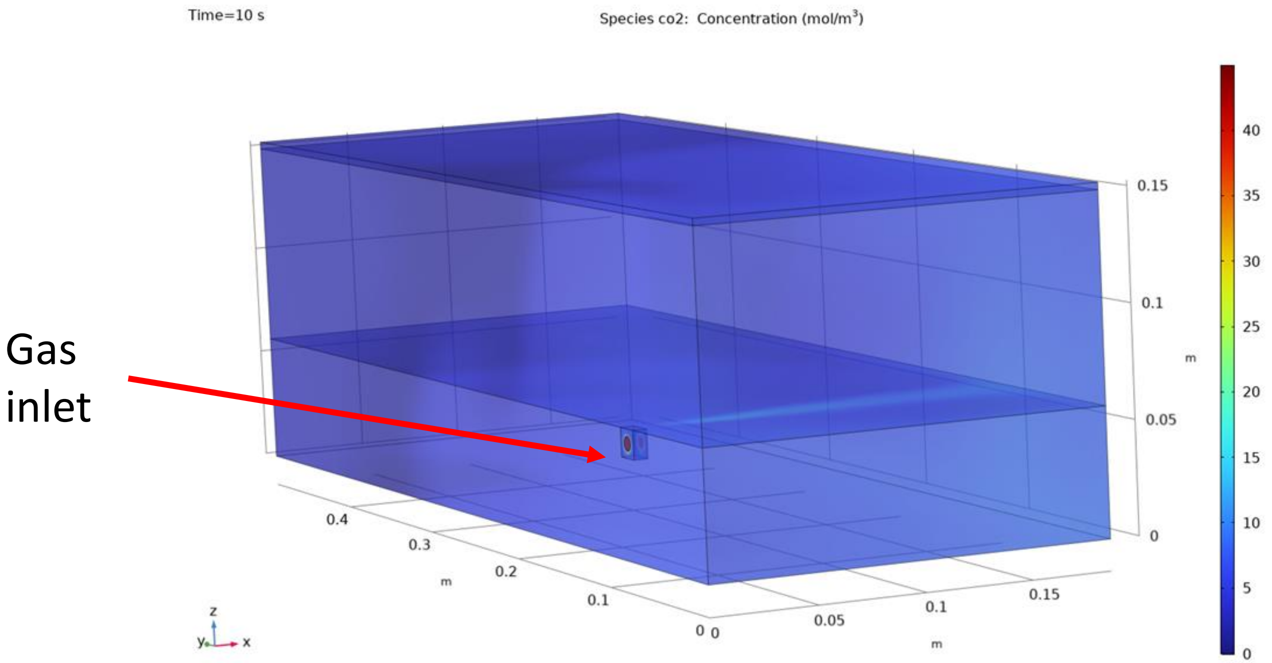
Light out
into

Spectrometer

We have setup an infrastructure with controlled environment that will enable us to characterize the hollow-core fiber-based gas detection.

1.1a COMSOL Multiphysics: simulation of CO_2 concentration intake in setup box

- *Convection-diffusion model coupled with Navier-Stokes model (laminar flow conditions)*
- CO_2 mass flow rate of 5 L/min within a ¼" tube corresponds to a velocity of ~2.3m/s (or 1.65x10^{-4}Kg/sec)
- Take gas fill information and feed back into fiber diffusion model



Set up COMSOL models to evaluate the effect of experimental box. The results are used to set up the boundary conditions for the hollow core fiber simulations.

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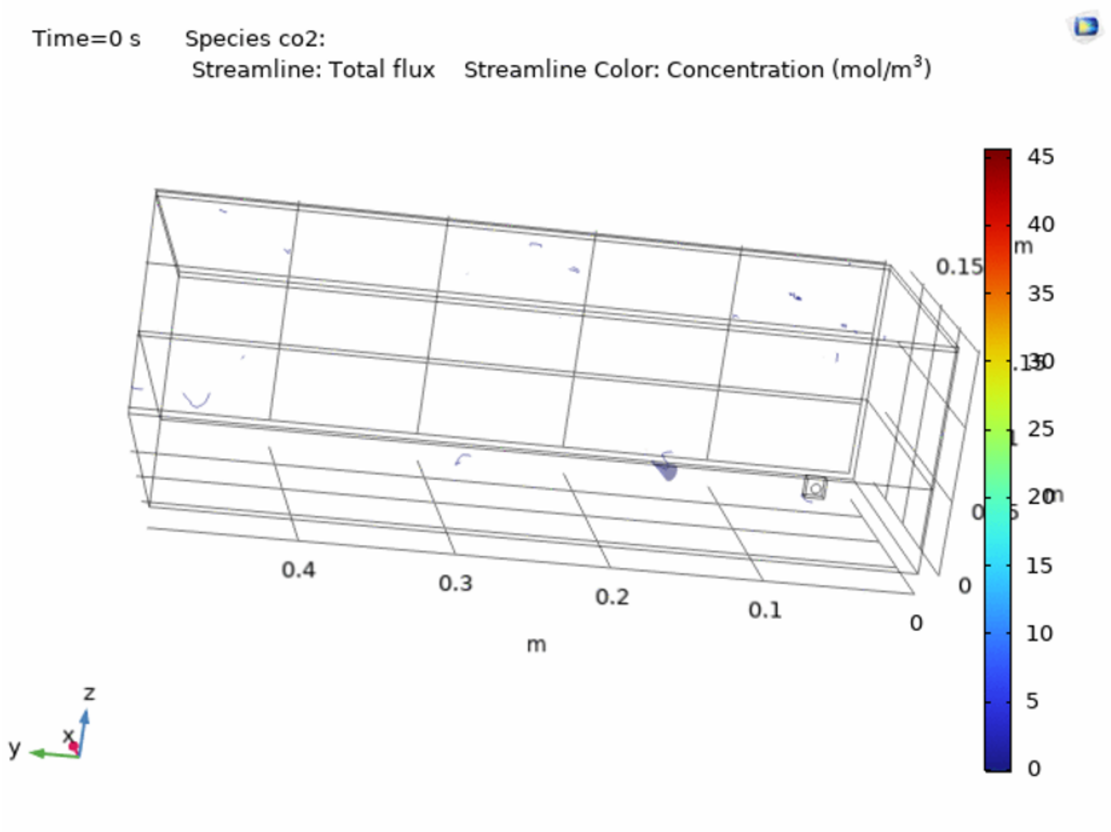

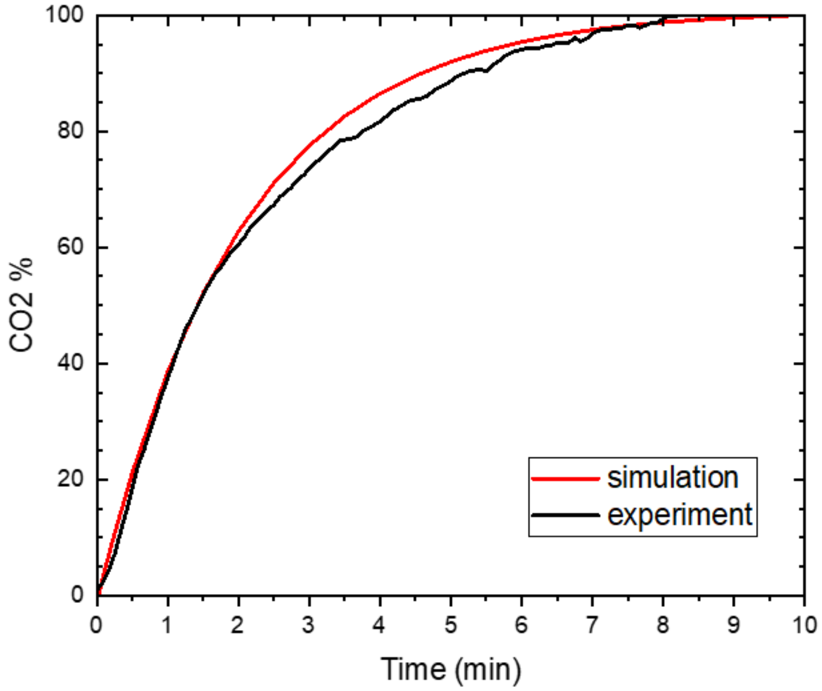

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Experiment 95%: 6.5mins

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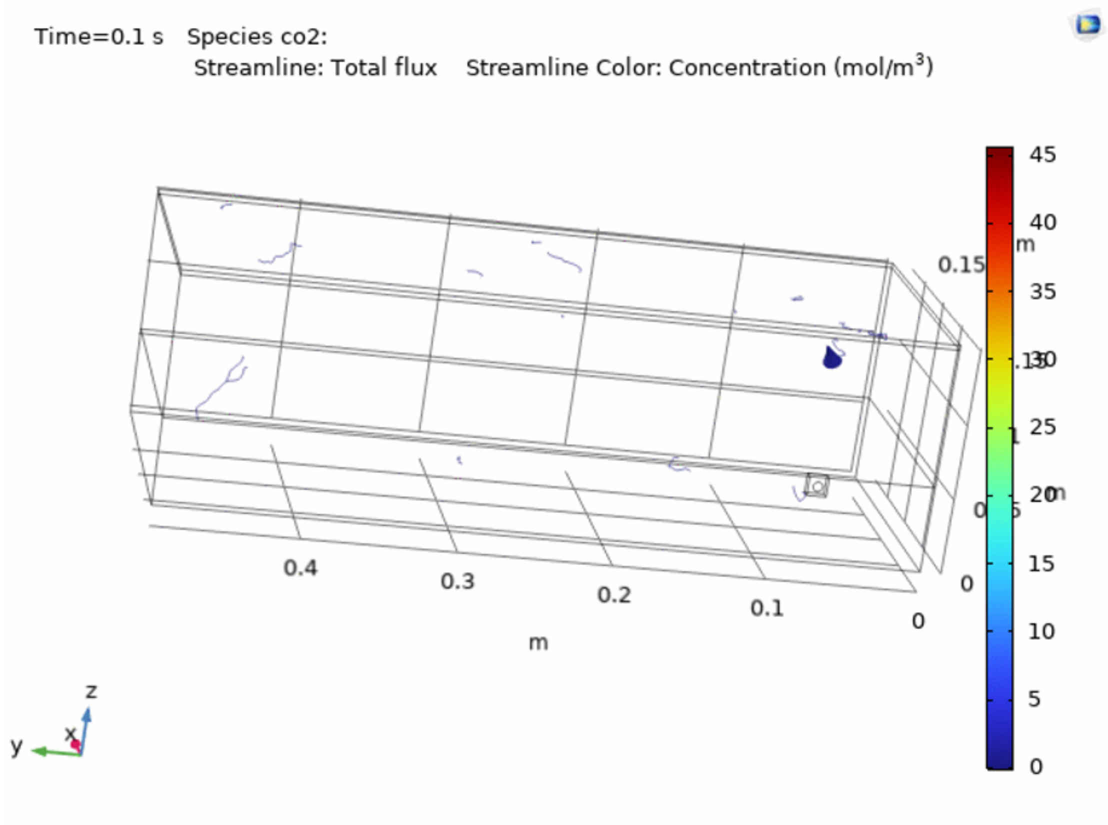

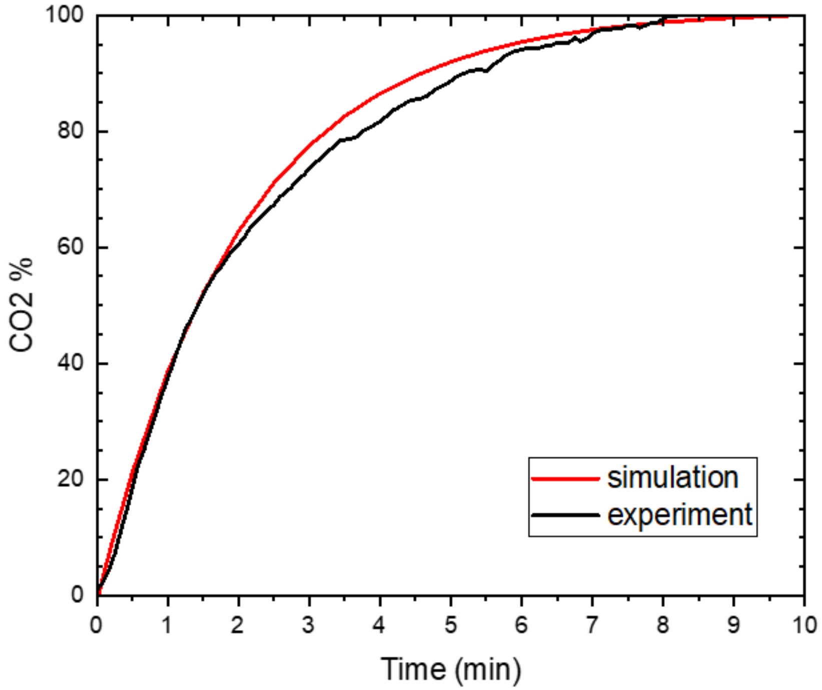

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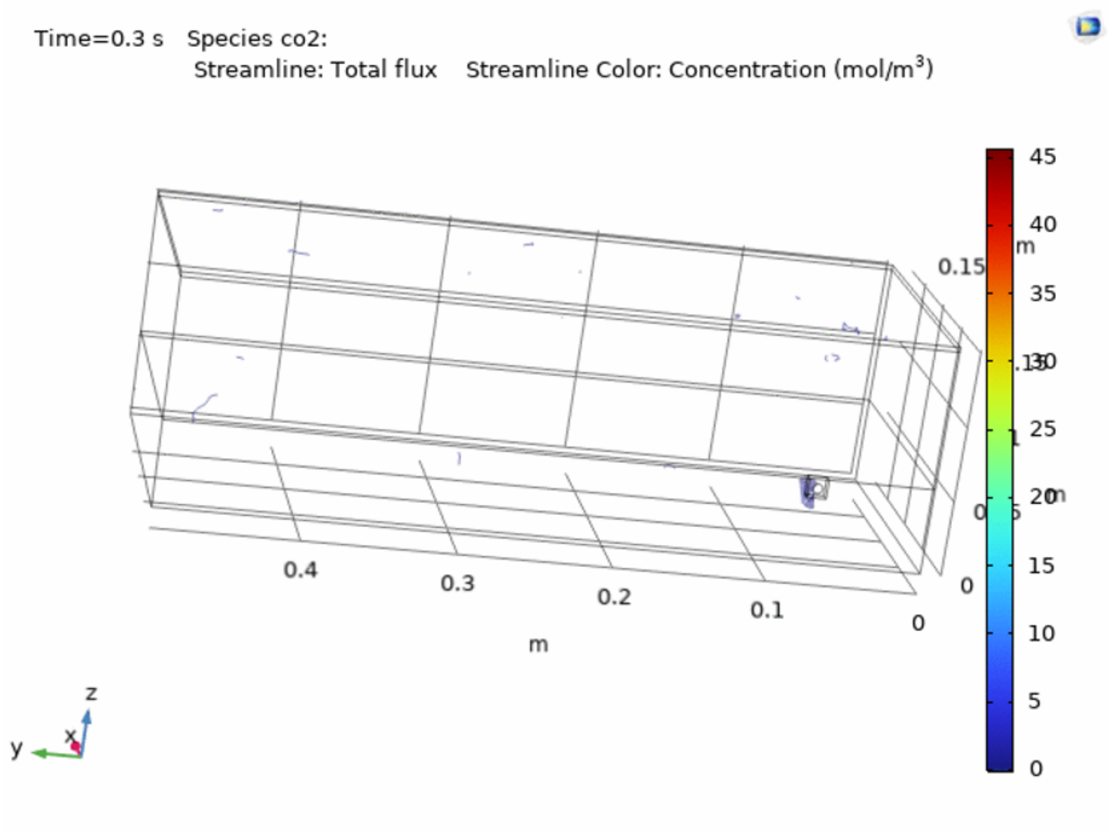

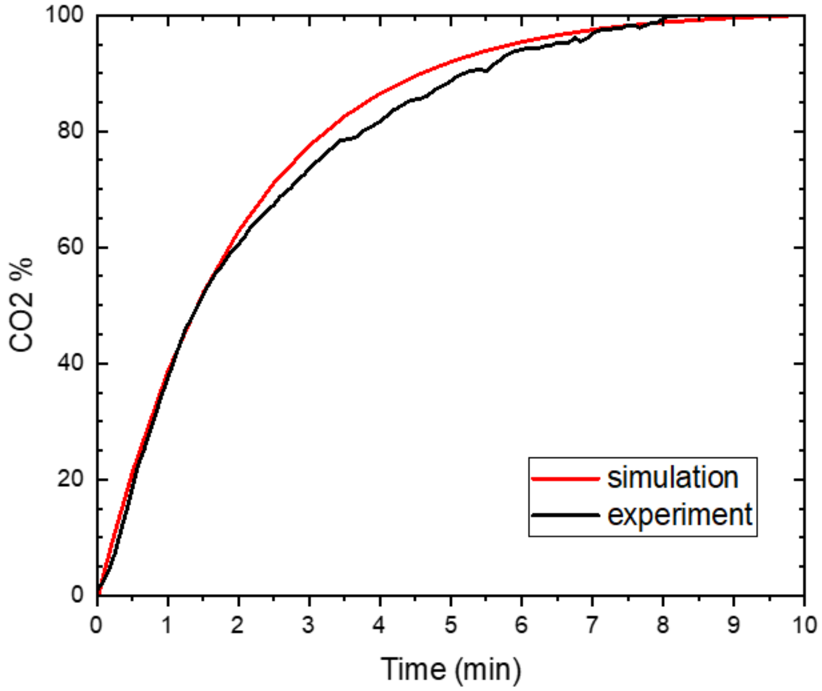

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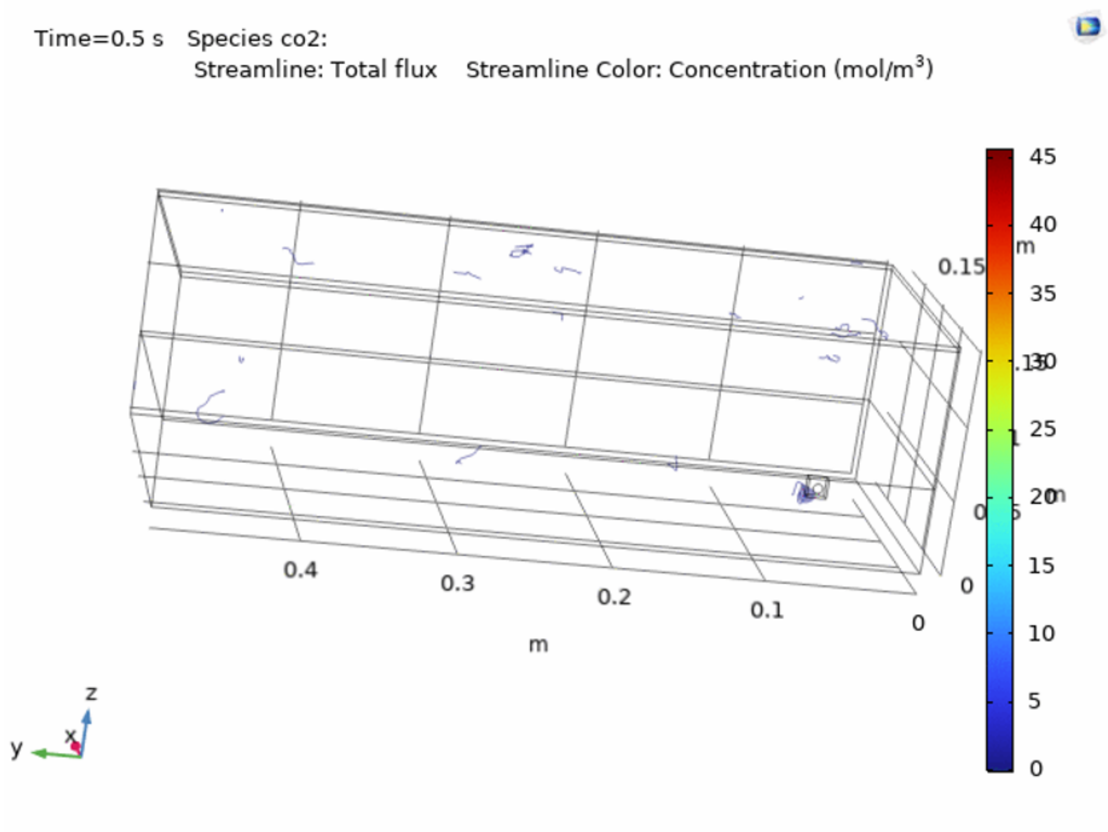

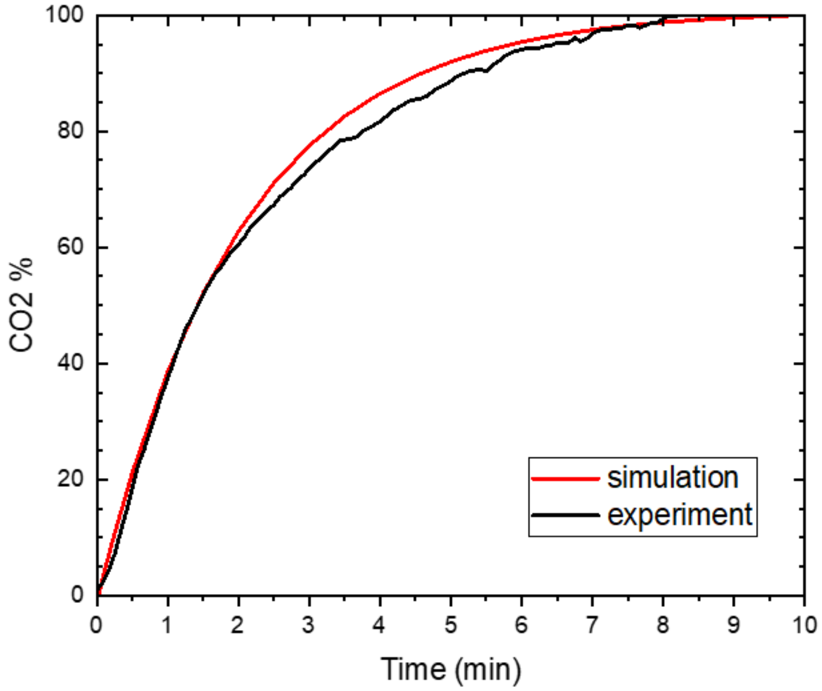

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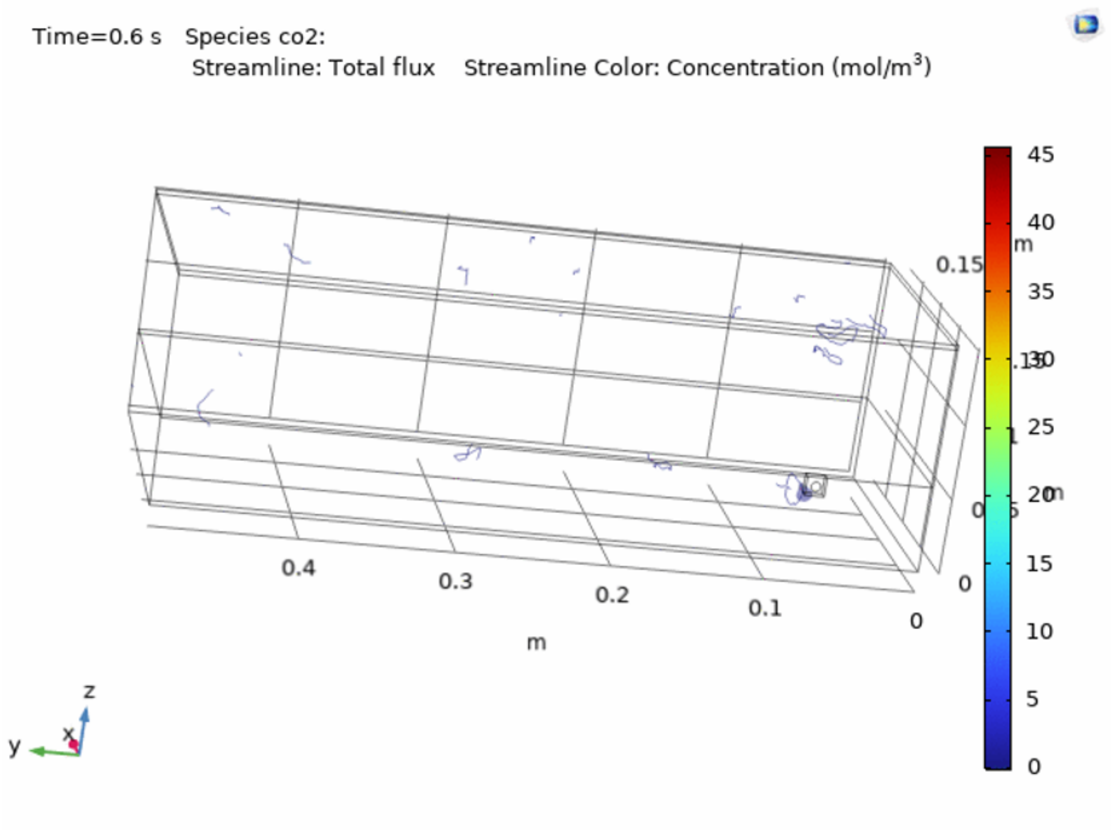

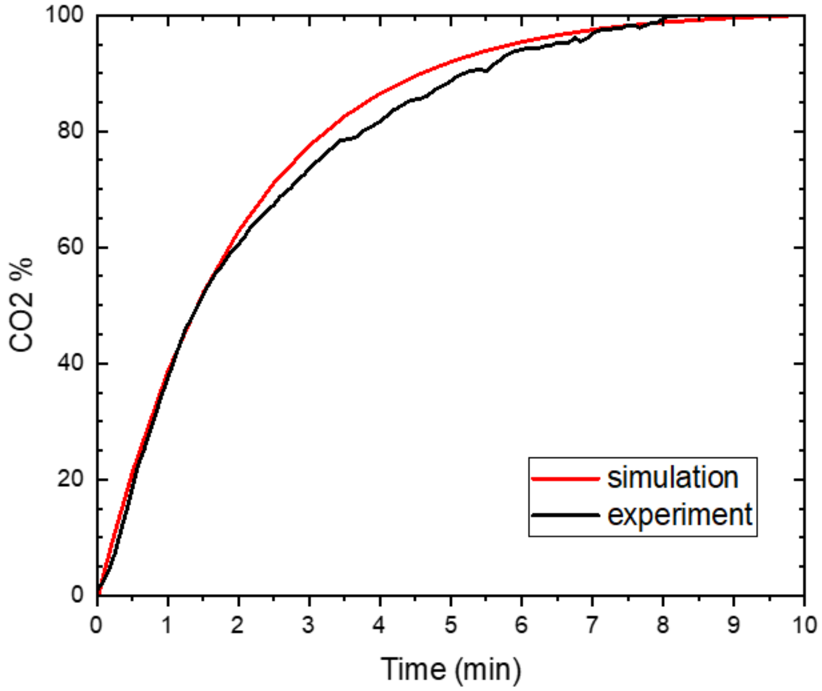

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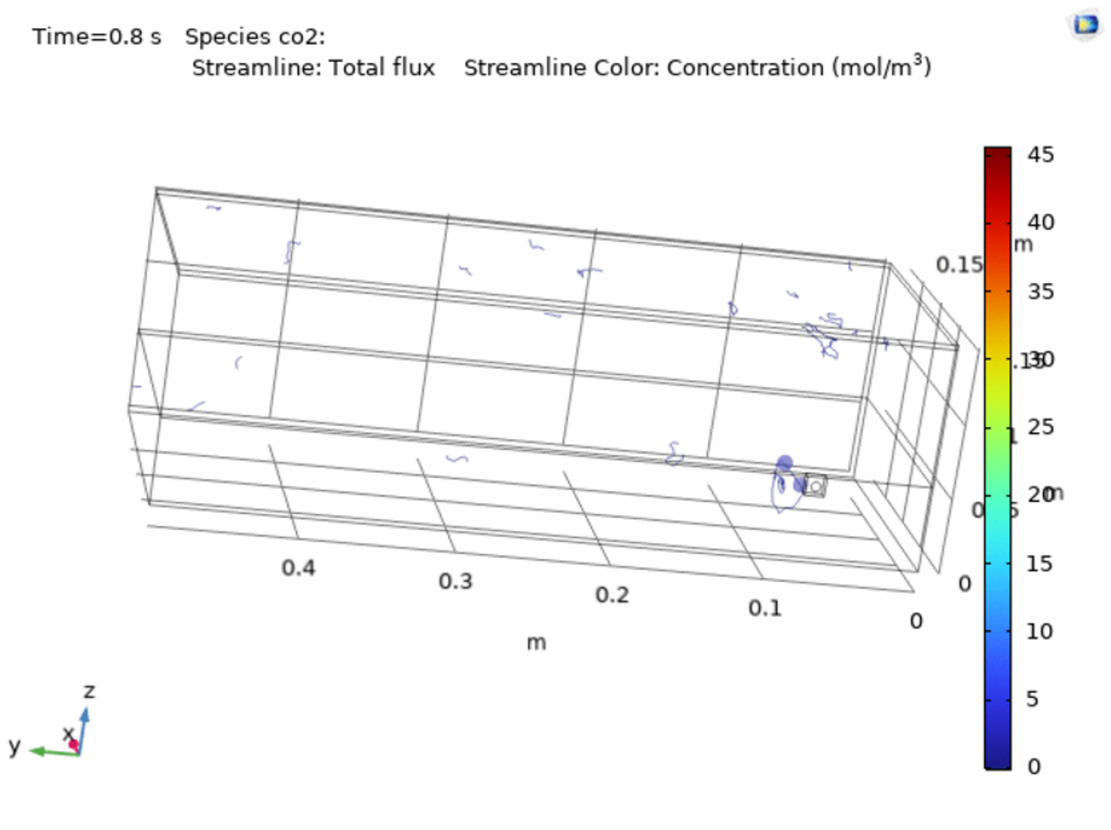

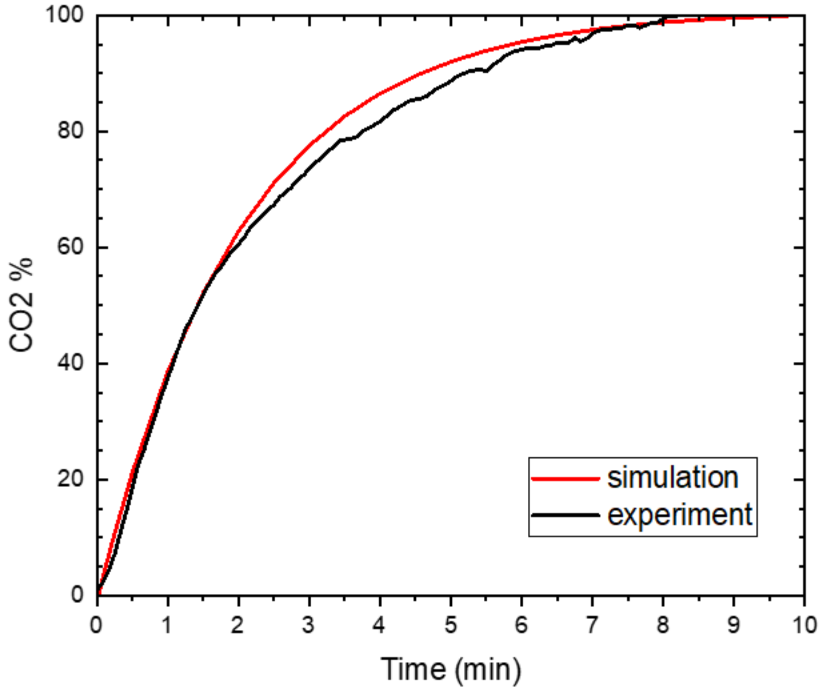

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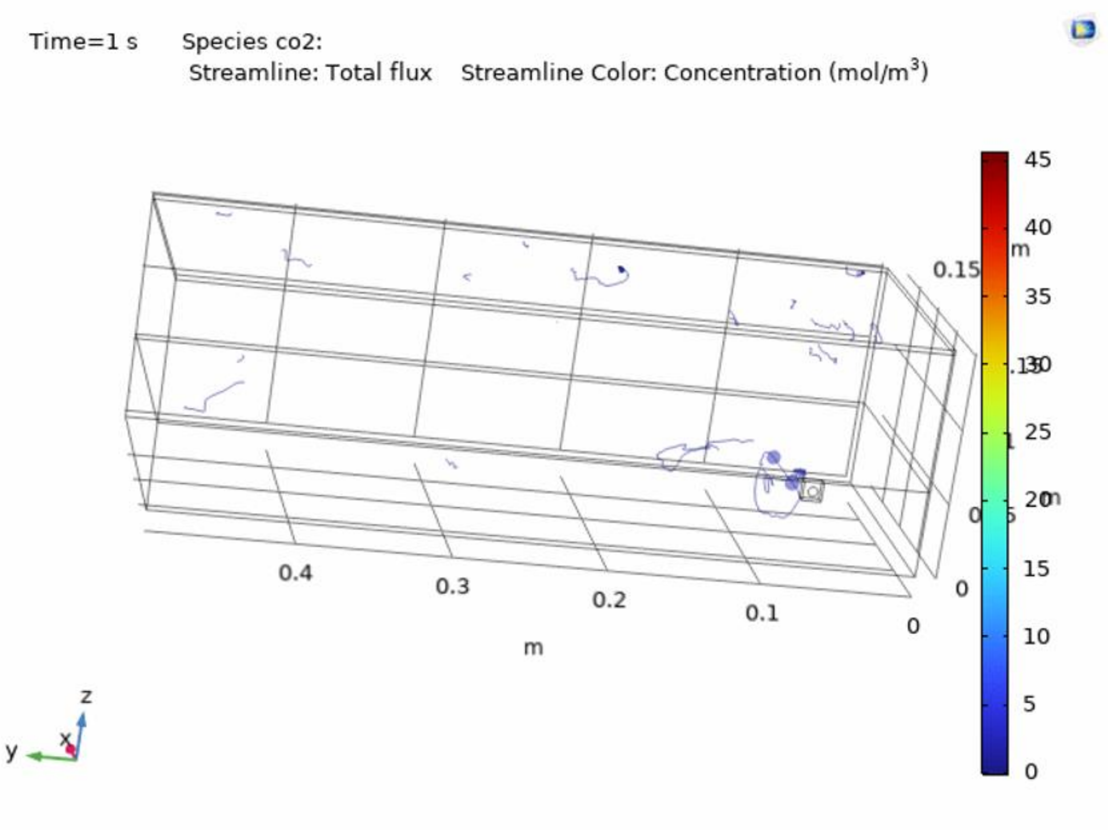

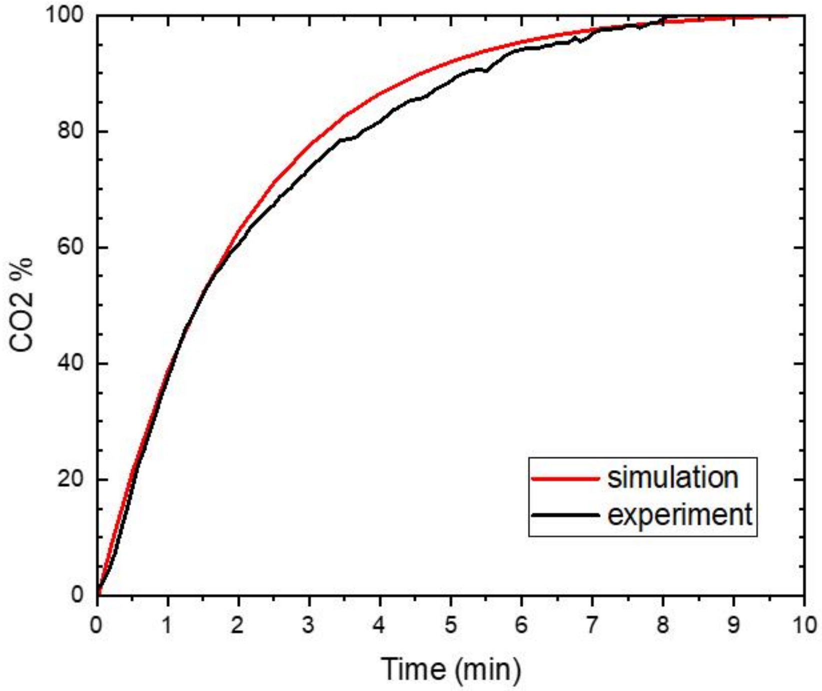

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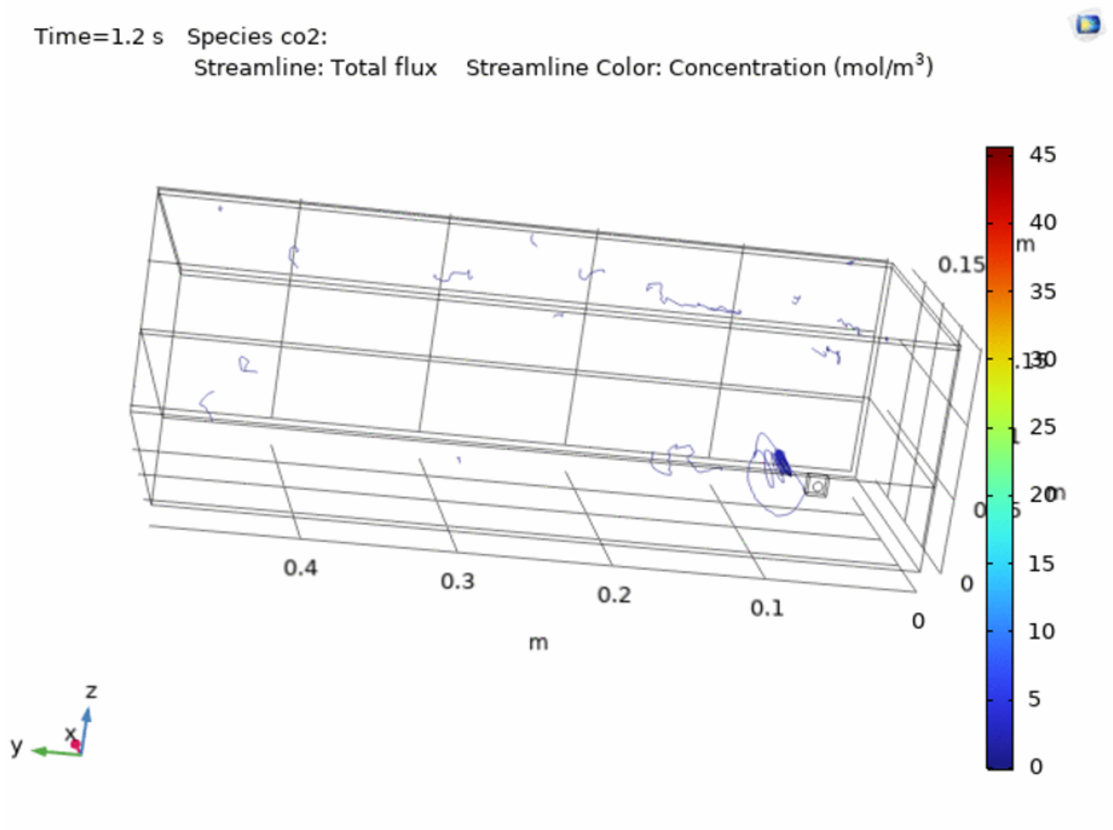

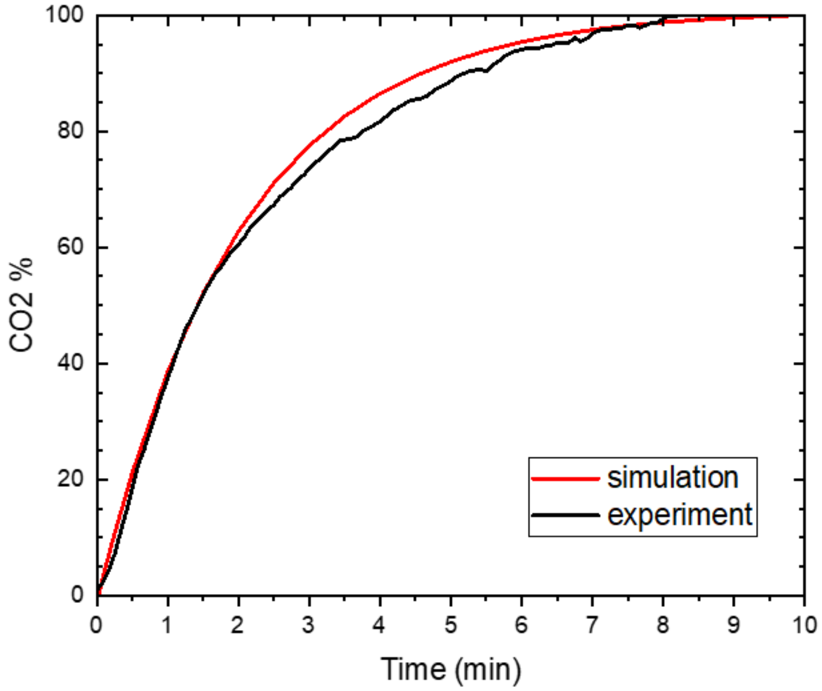

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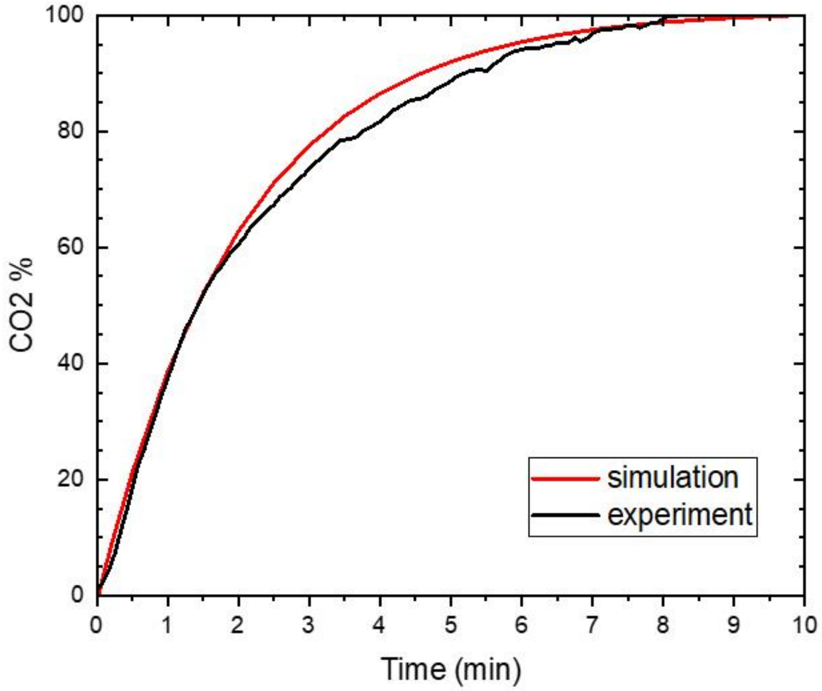

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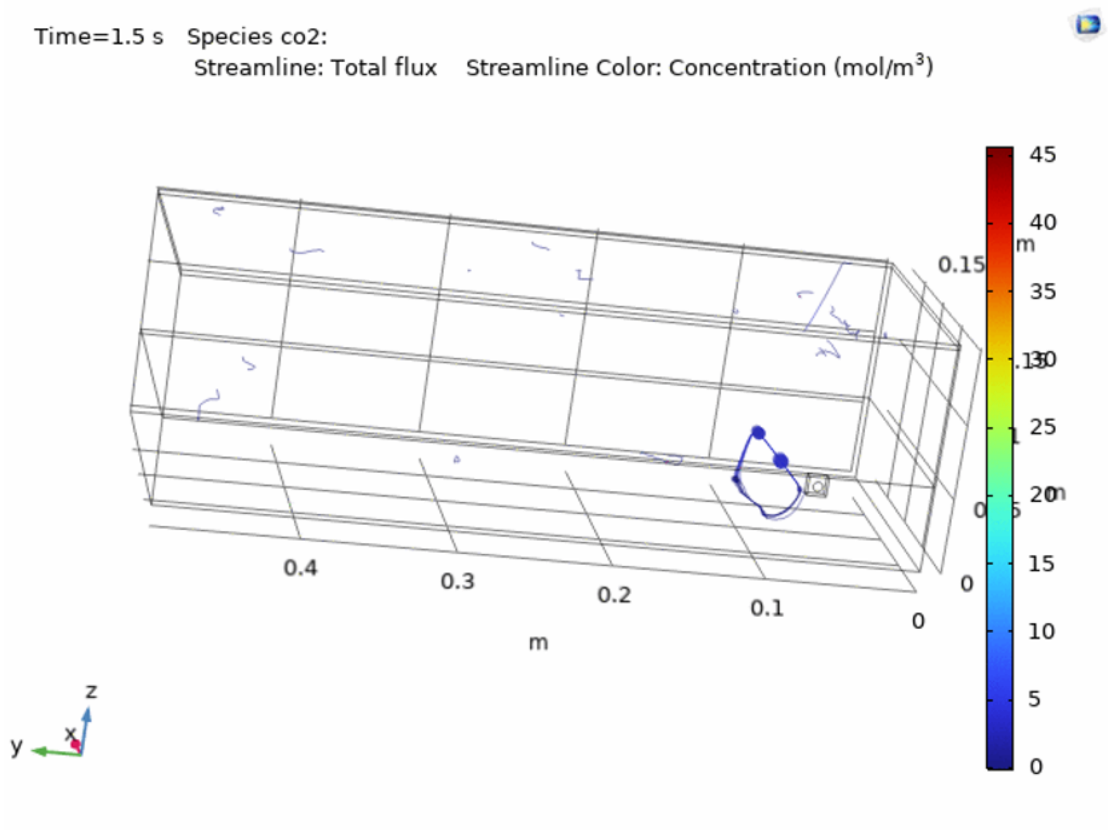

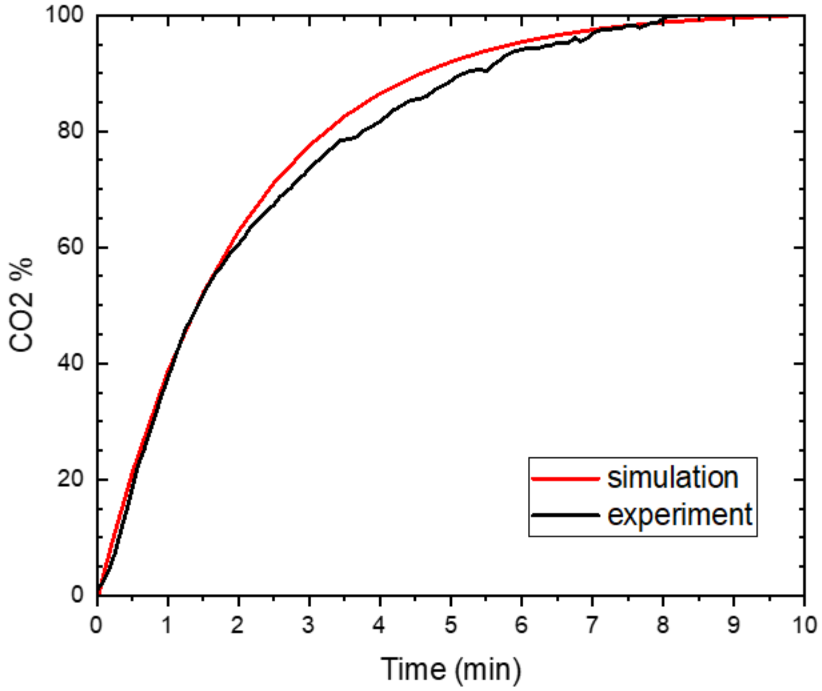

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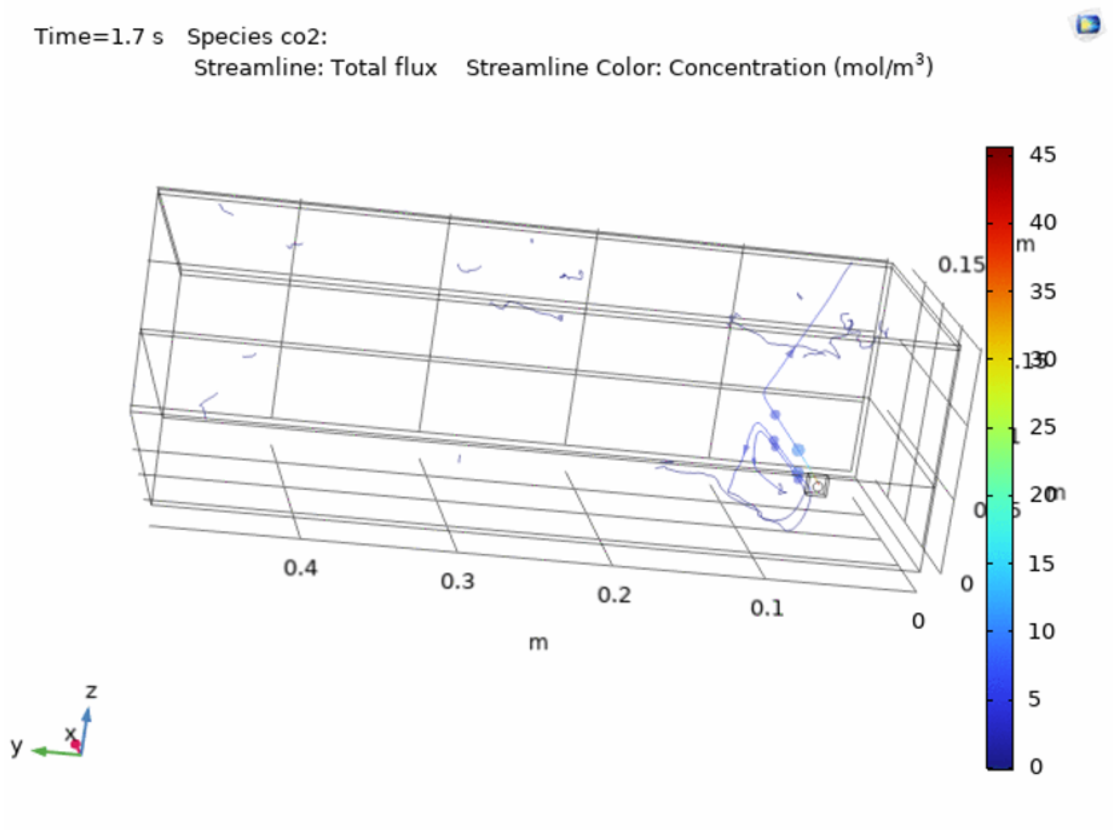

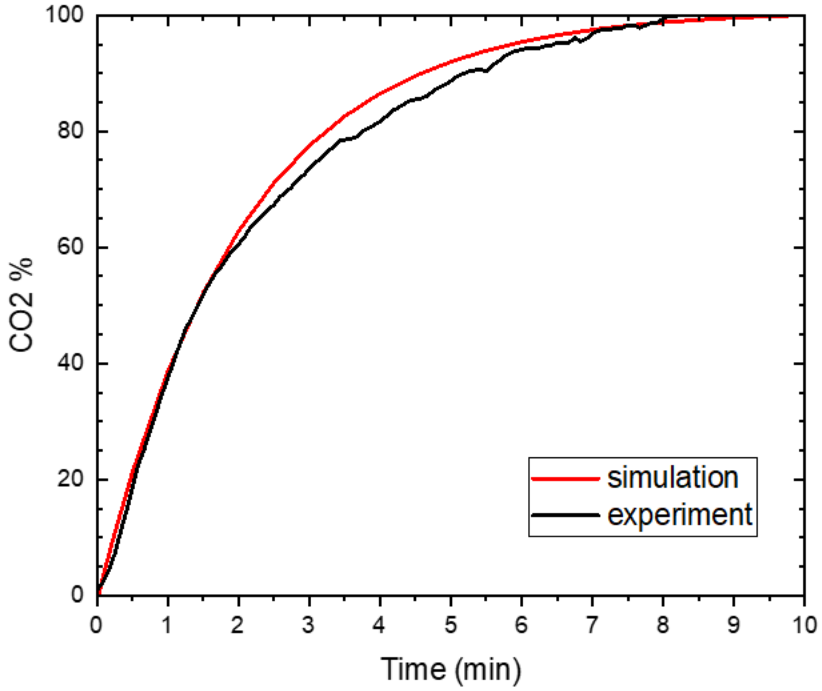

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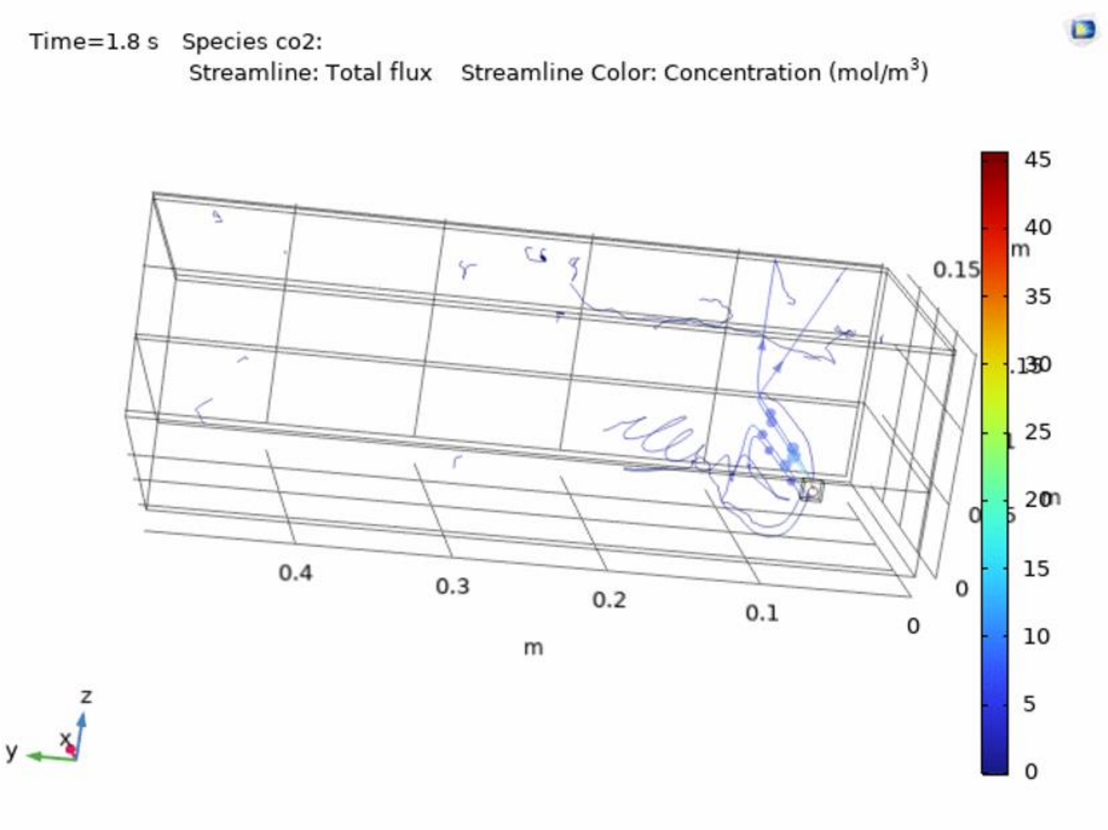

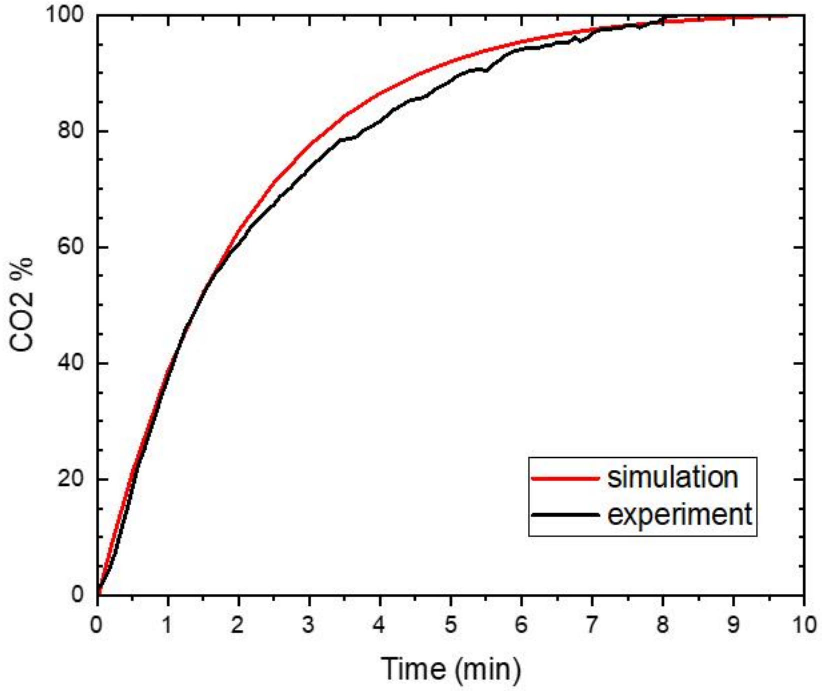

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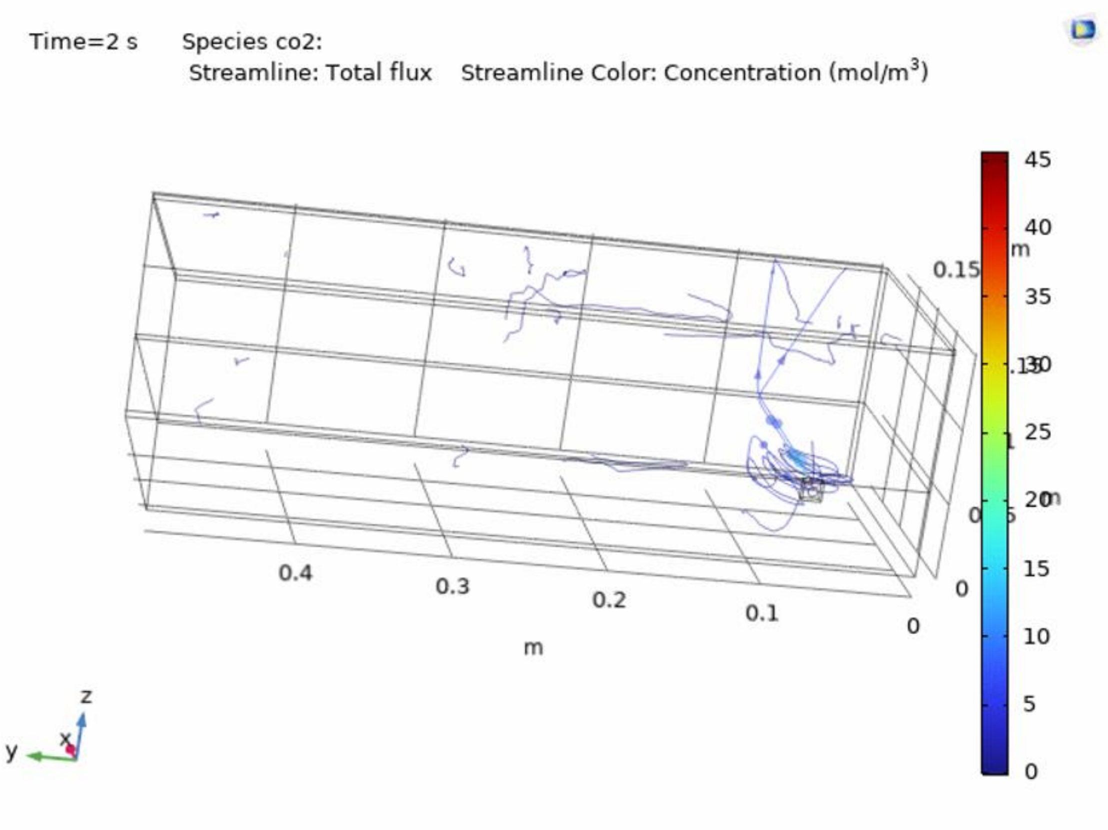

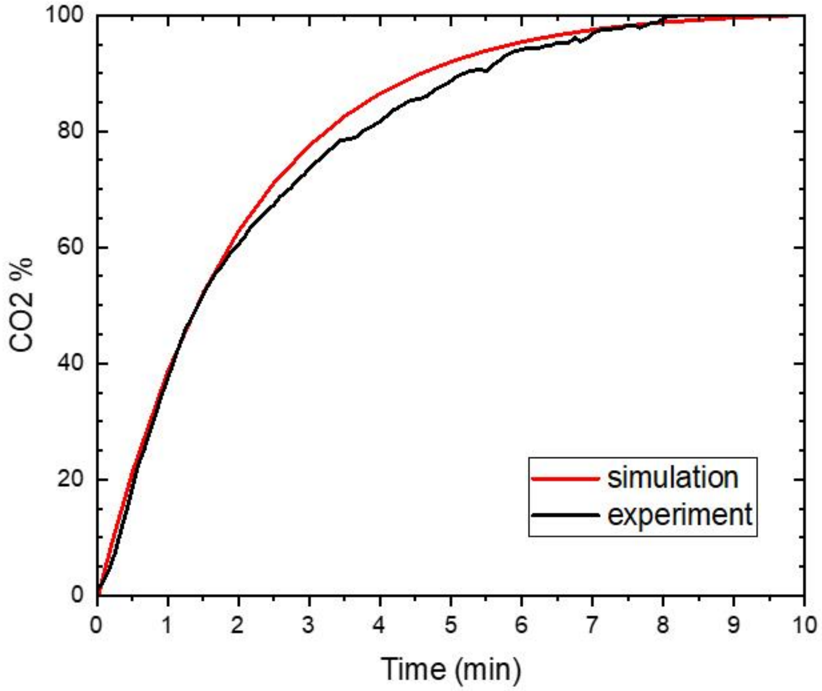

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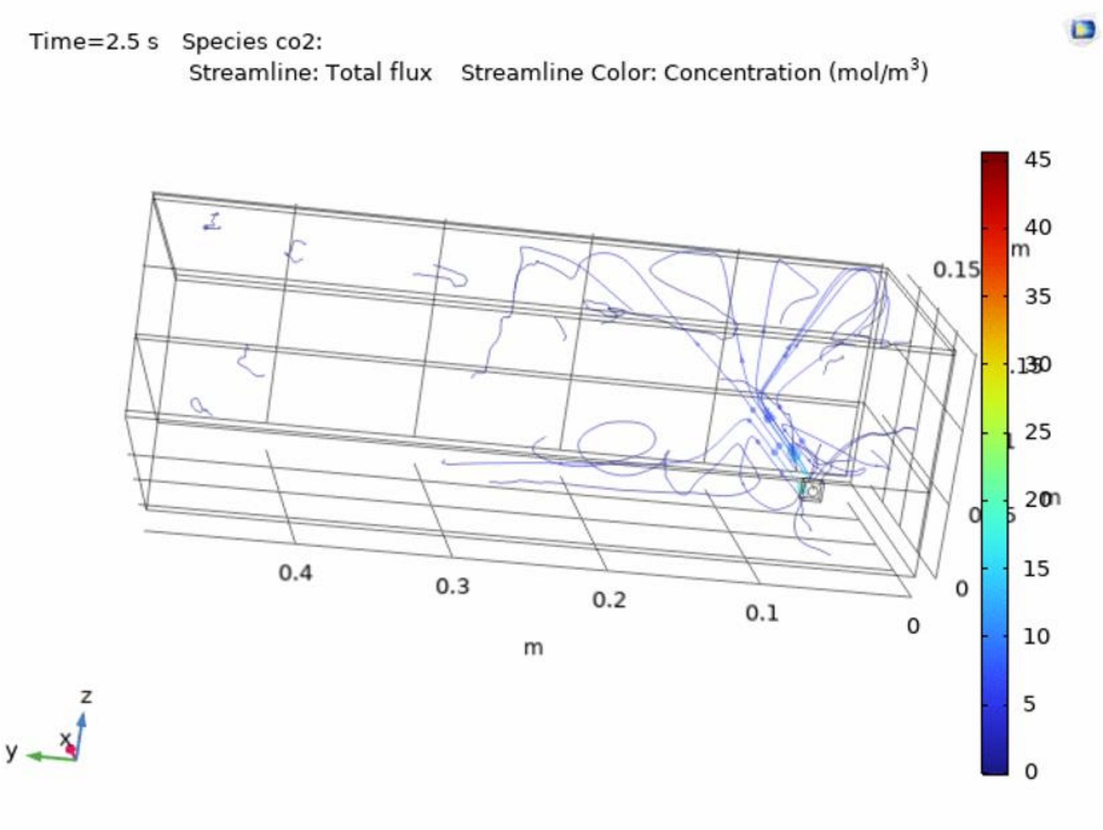

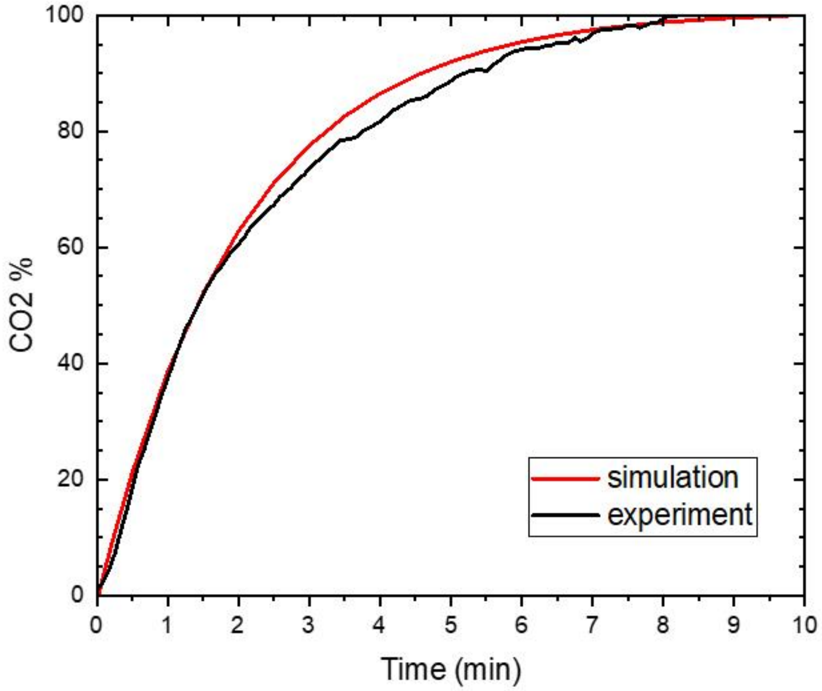

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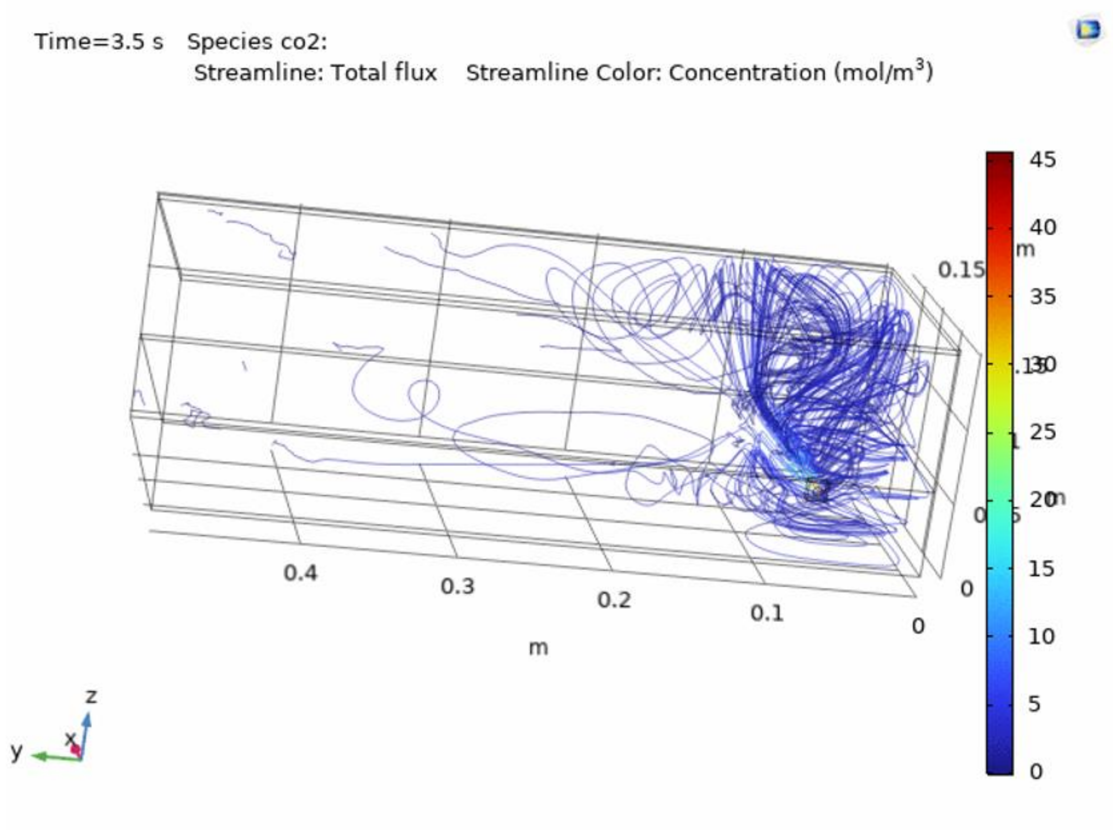

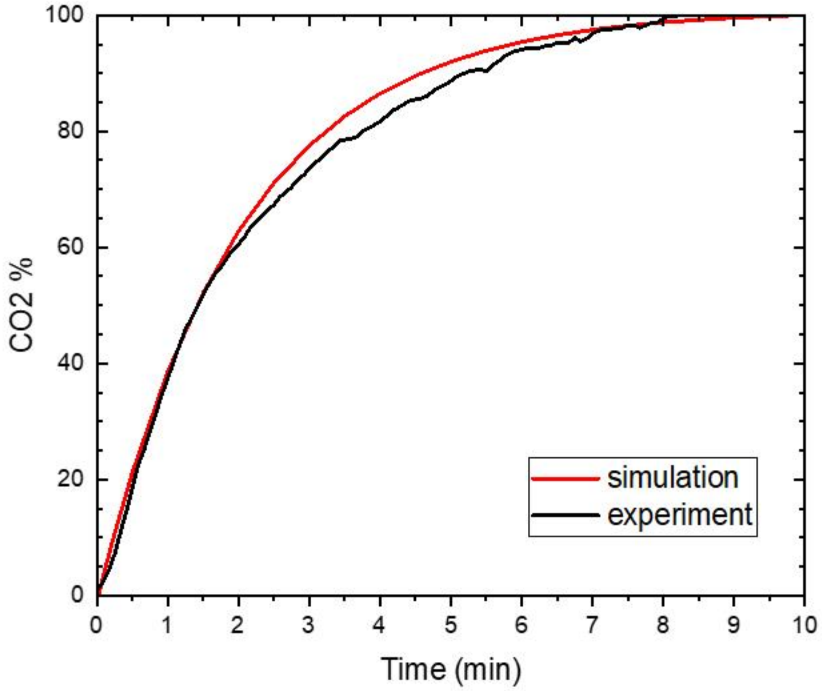

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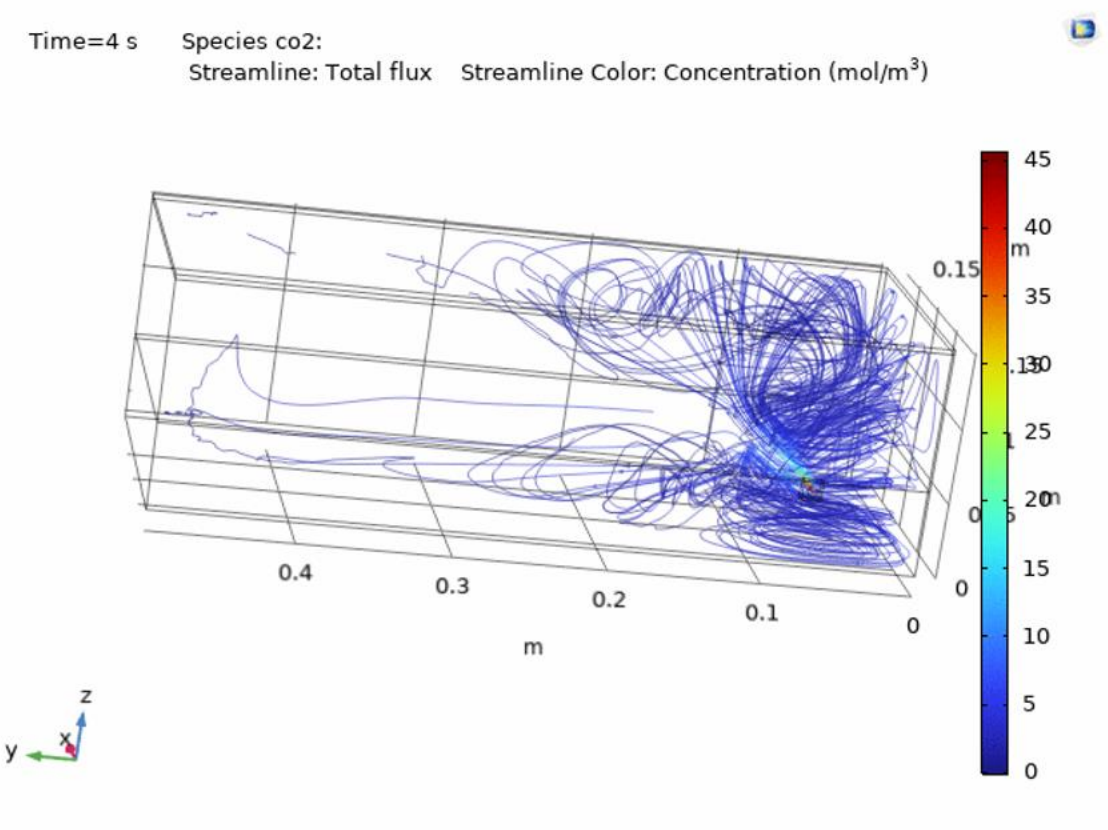

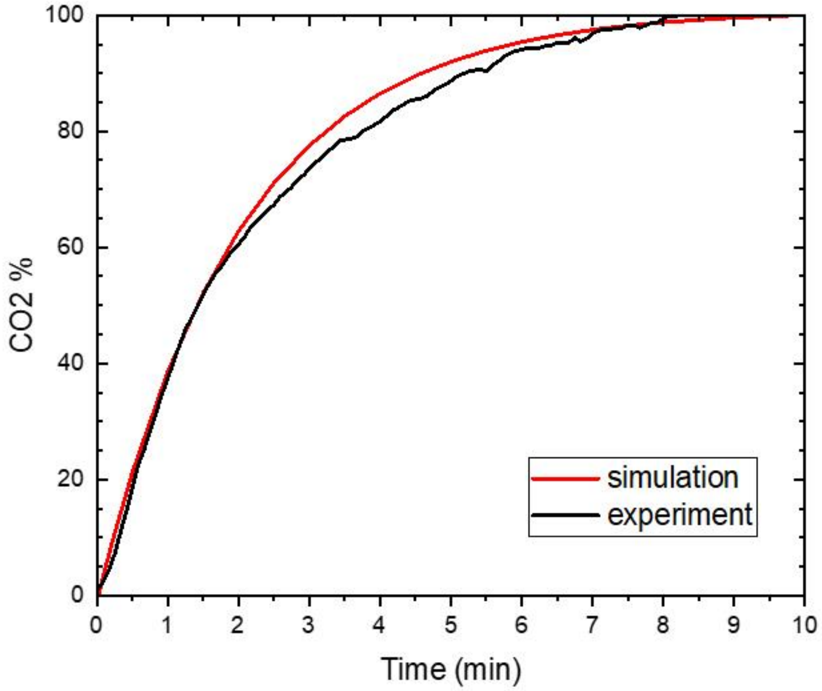

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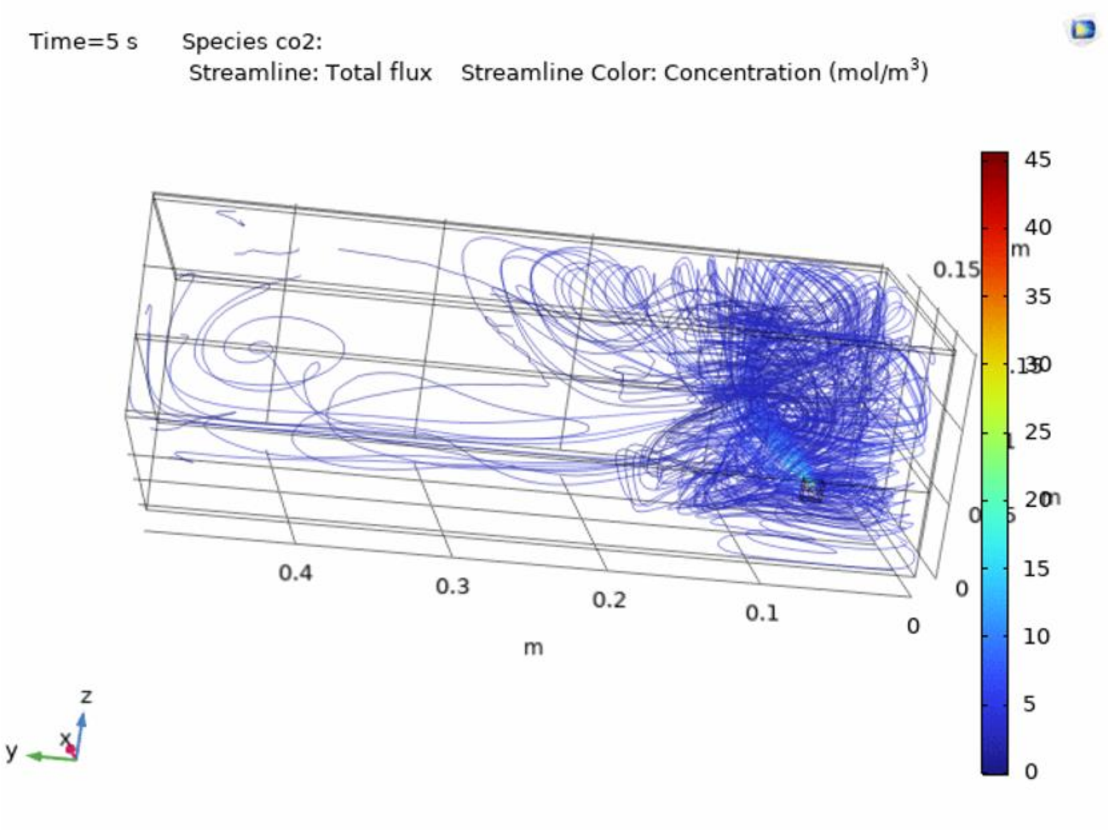

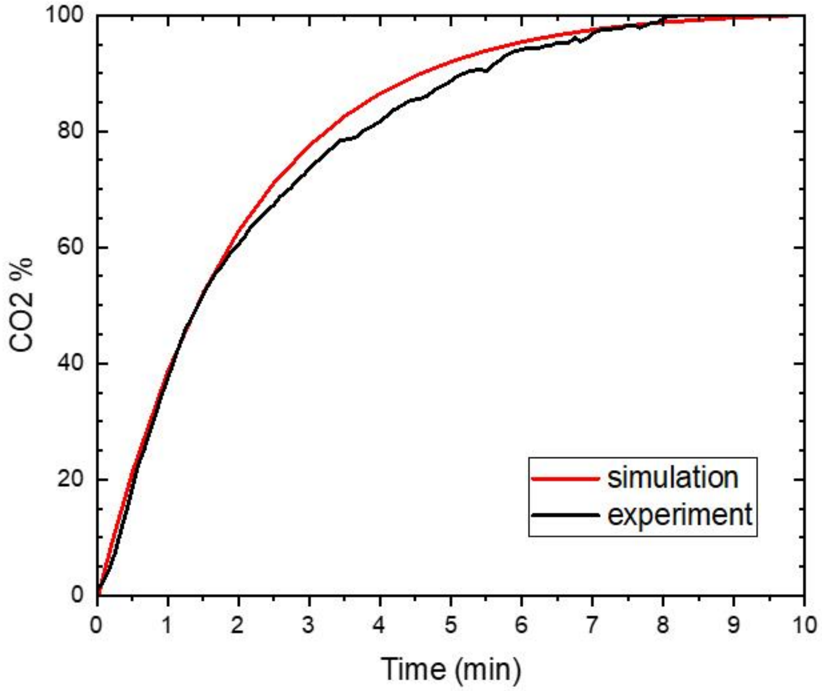

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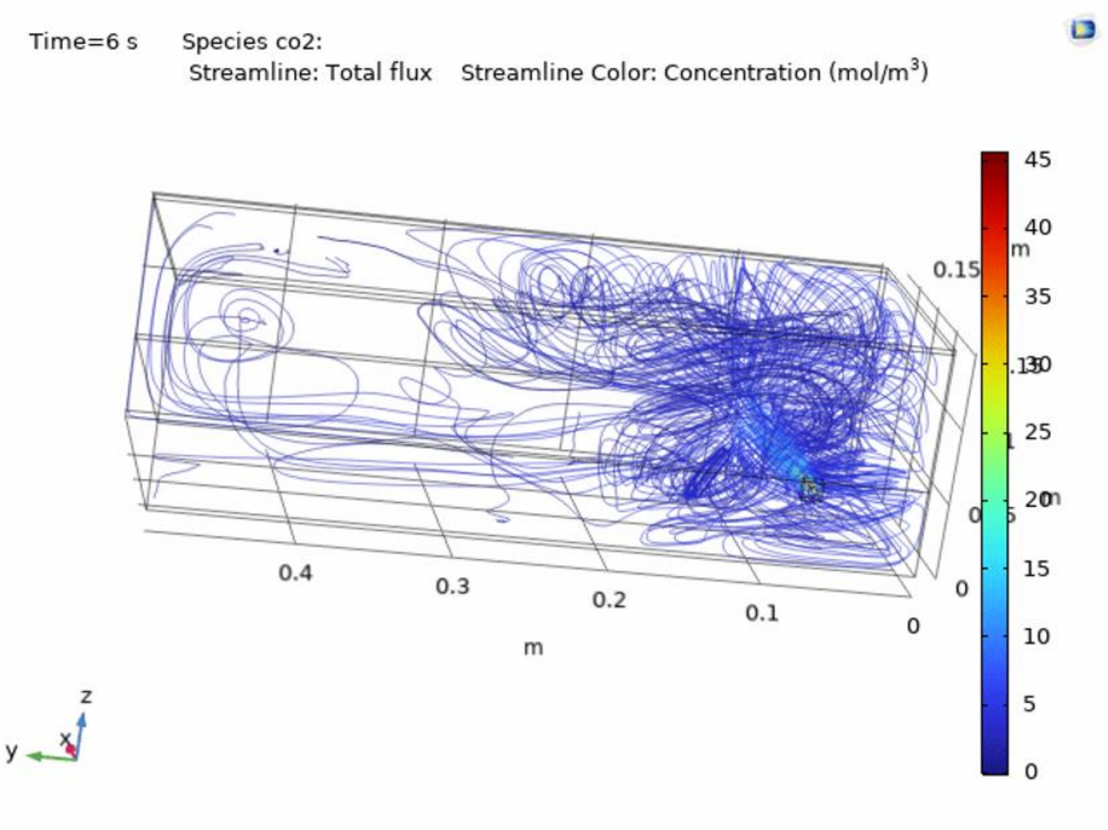

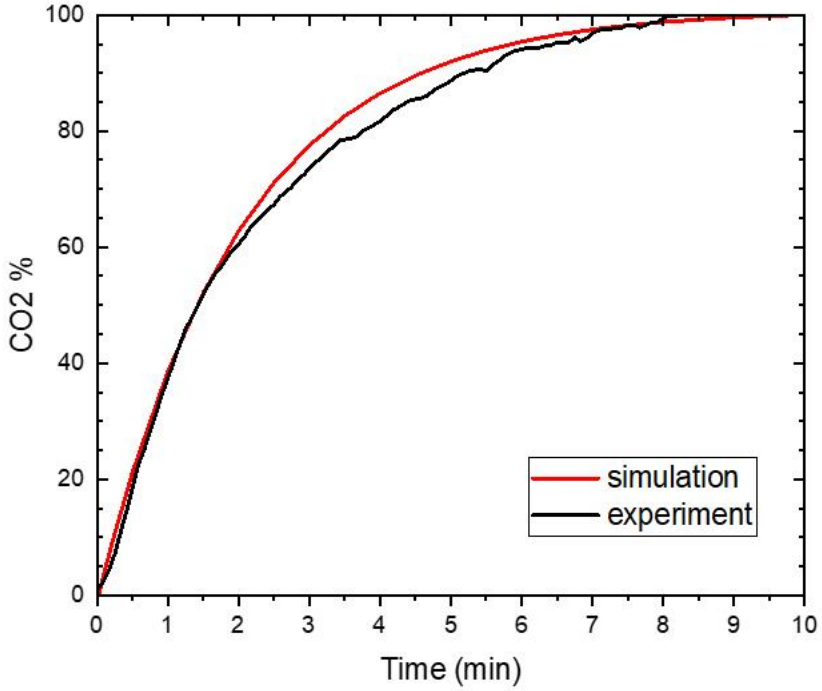

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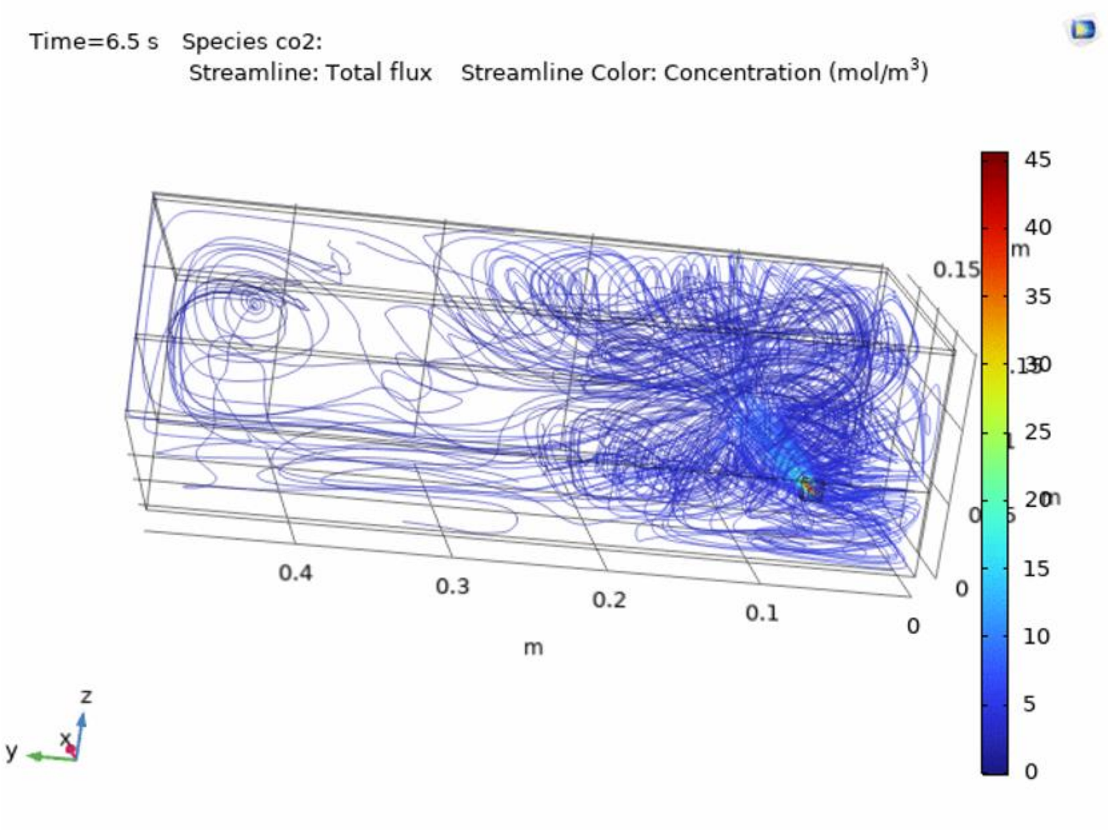

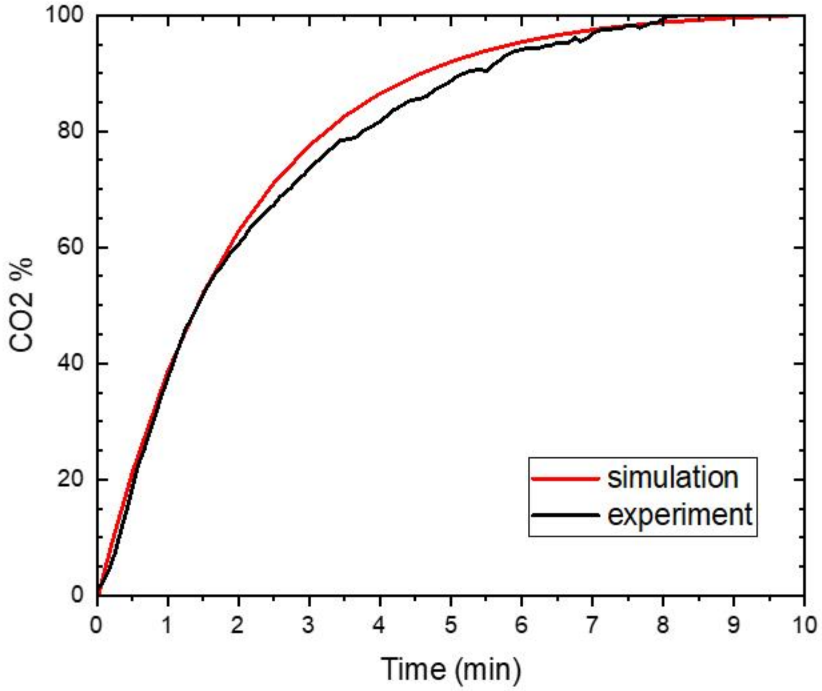

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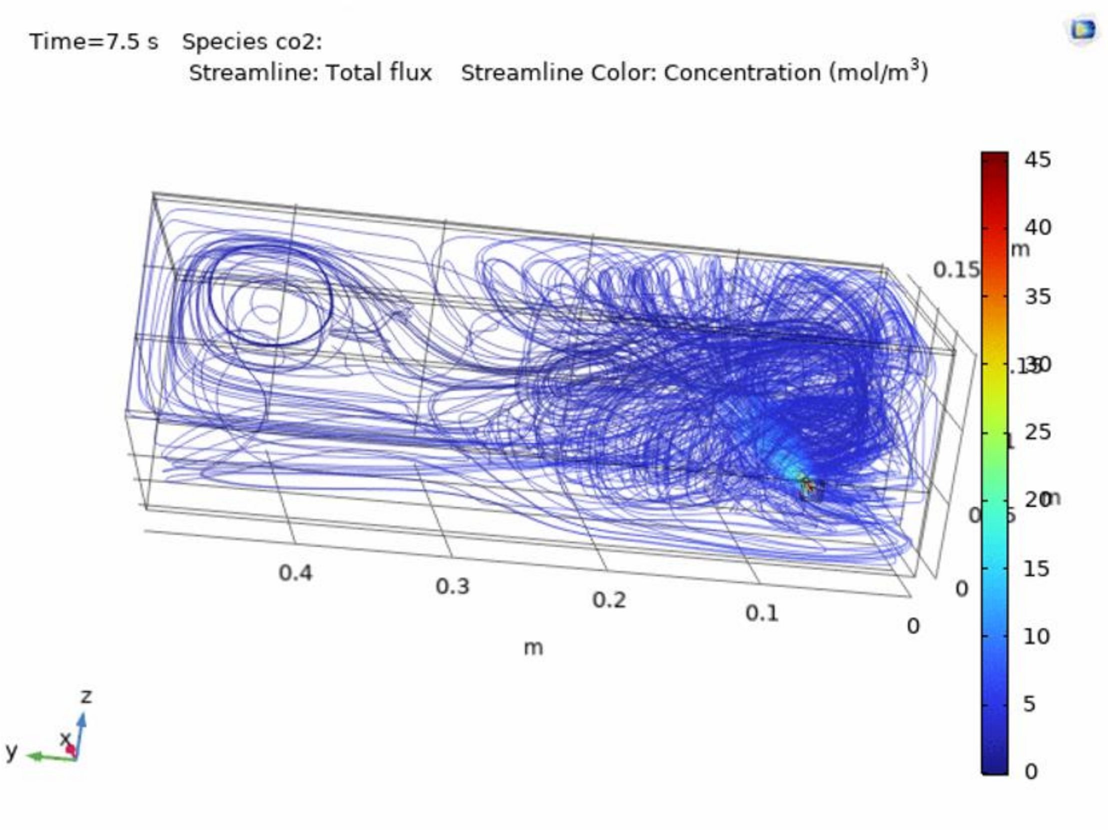

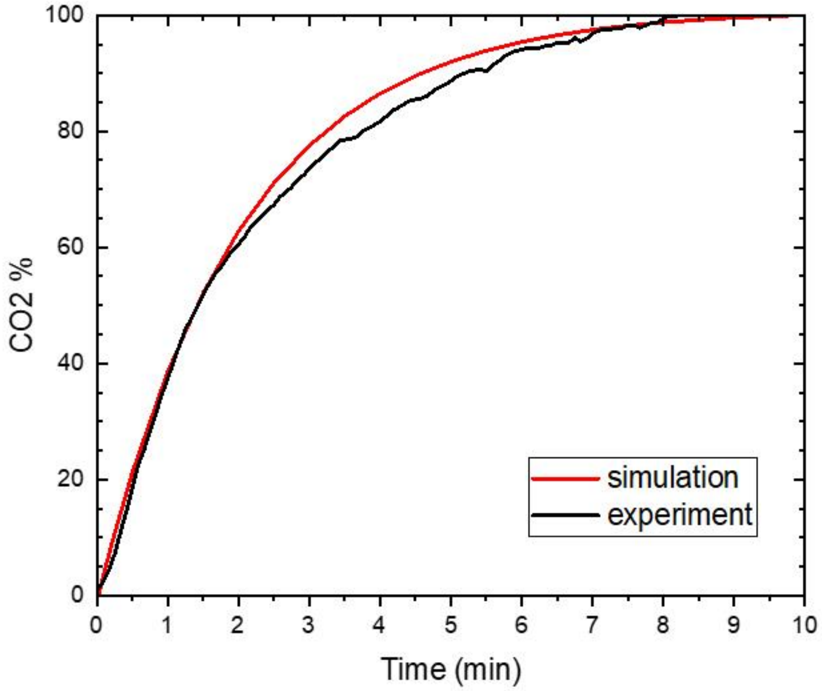

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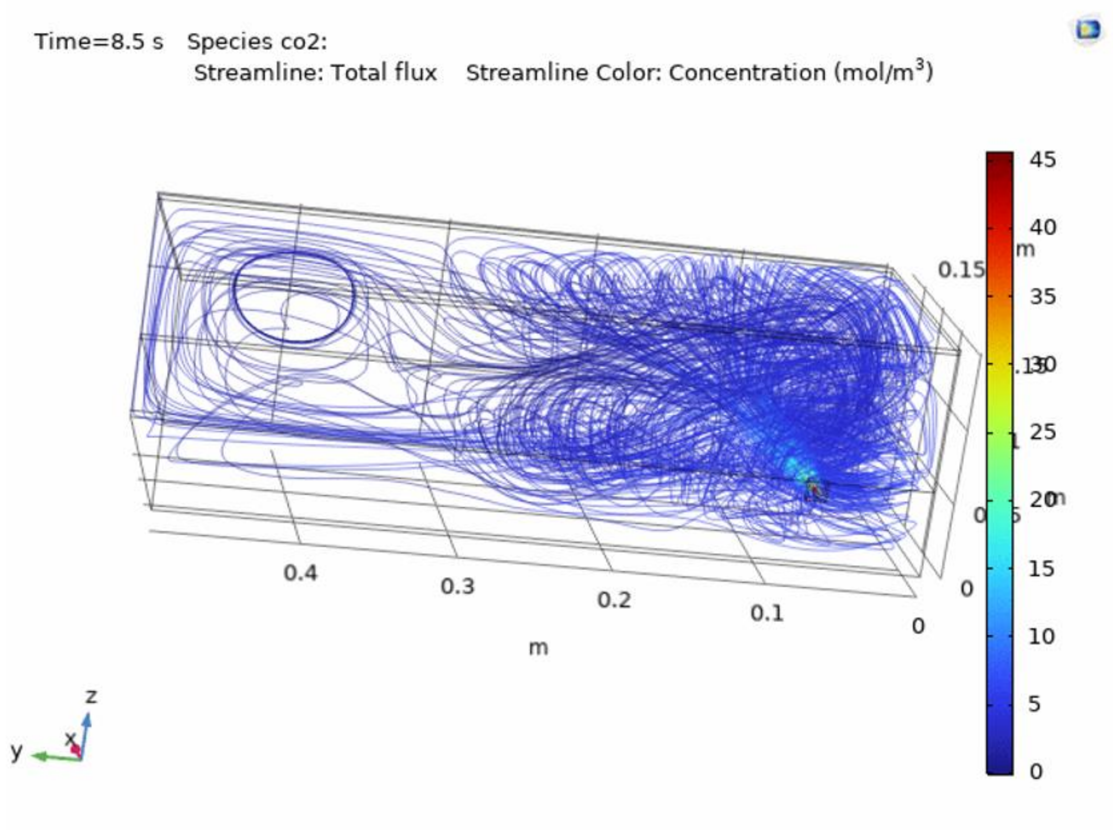

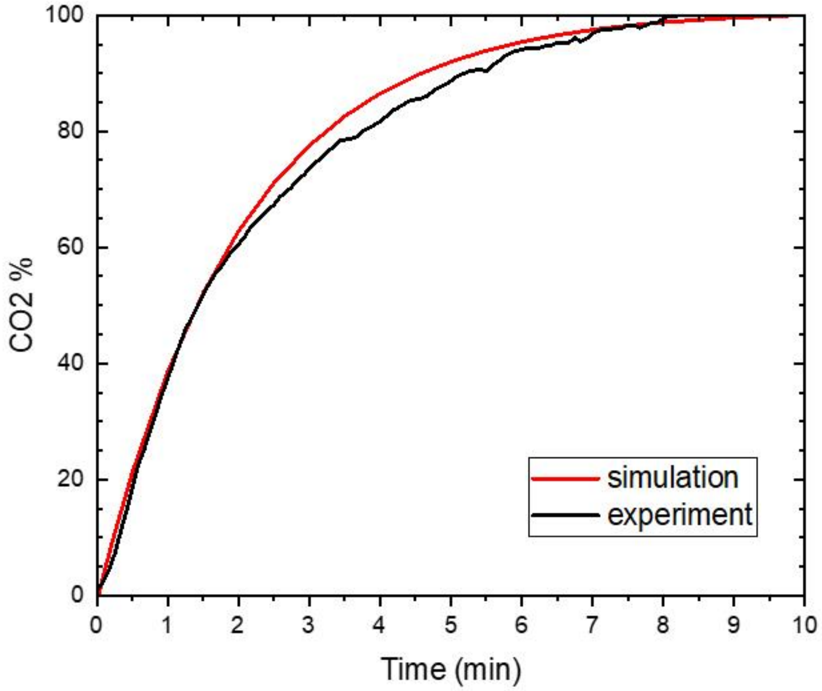

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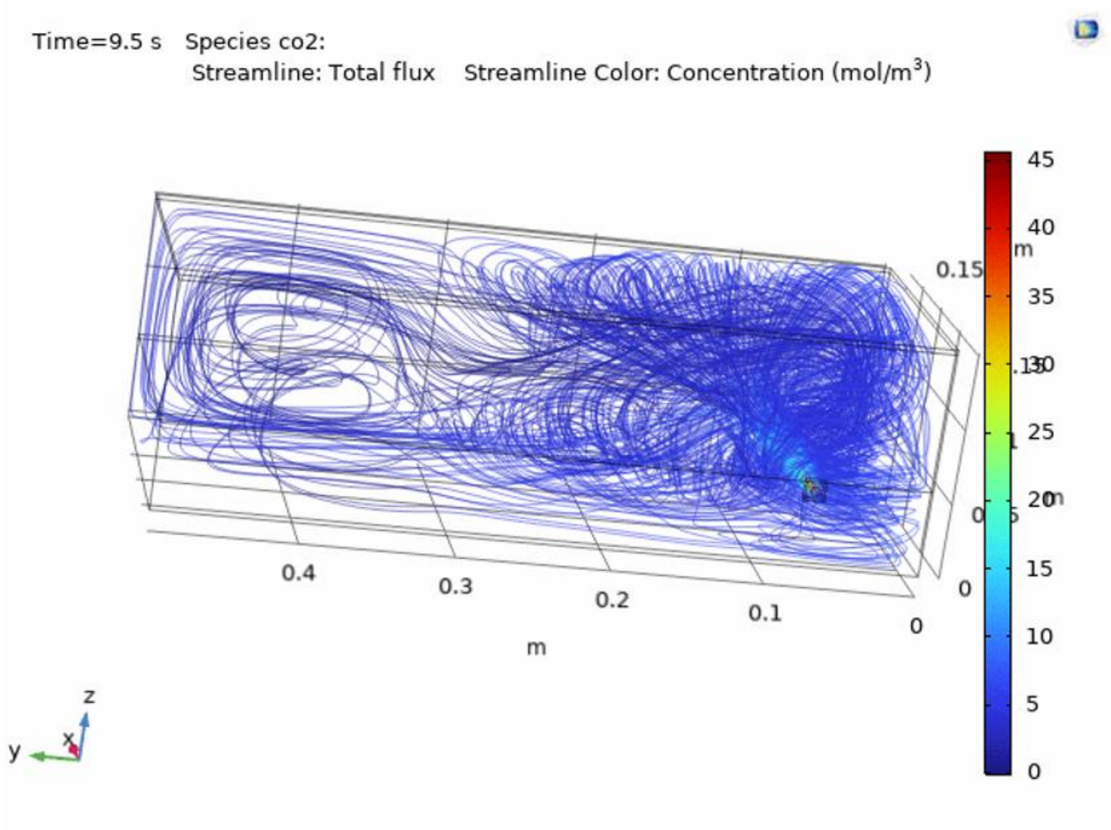

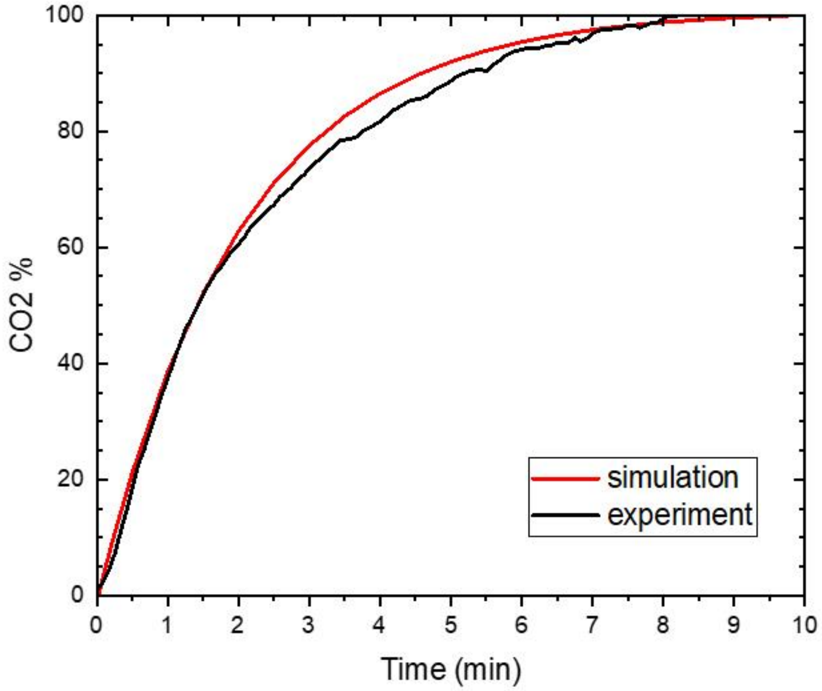

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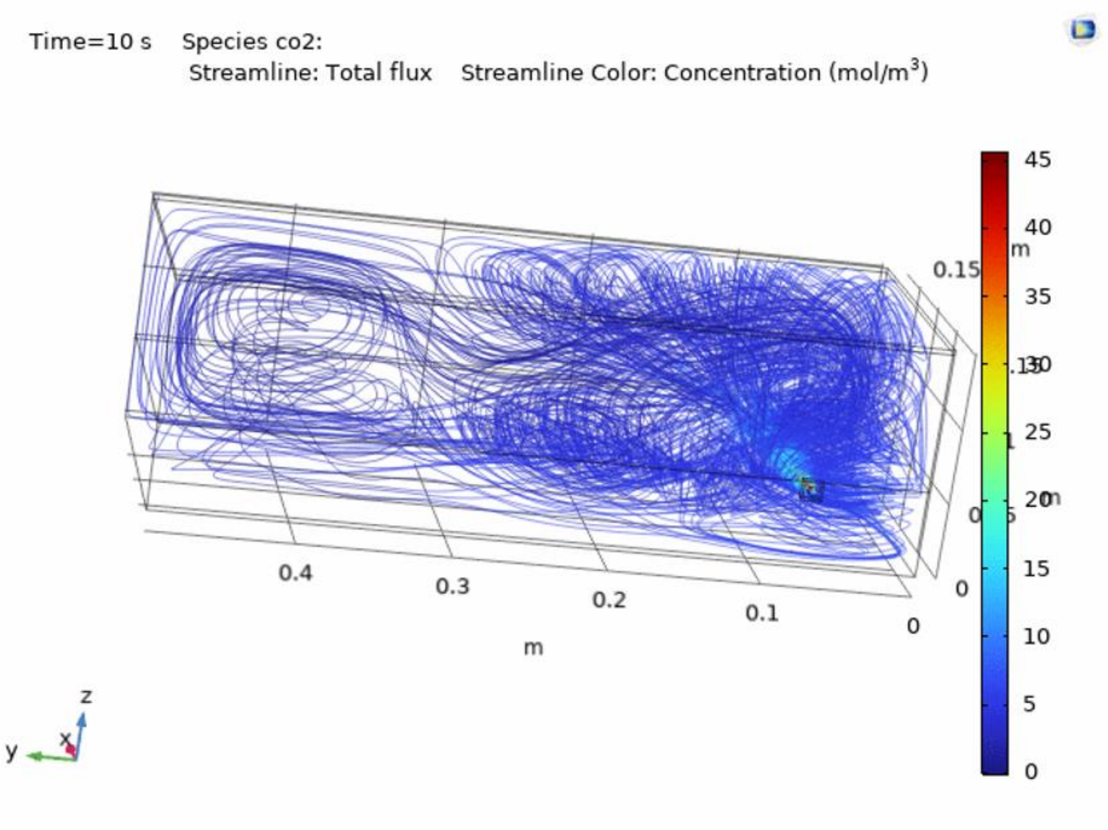

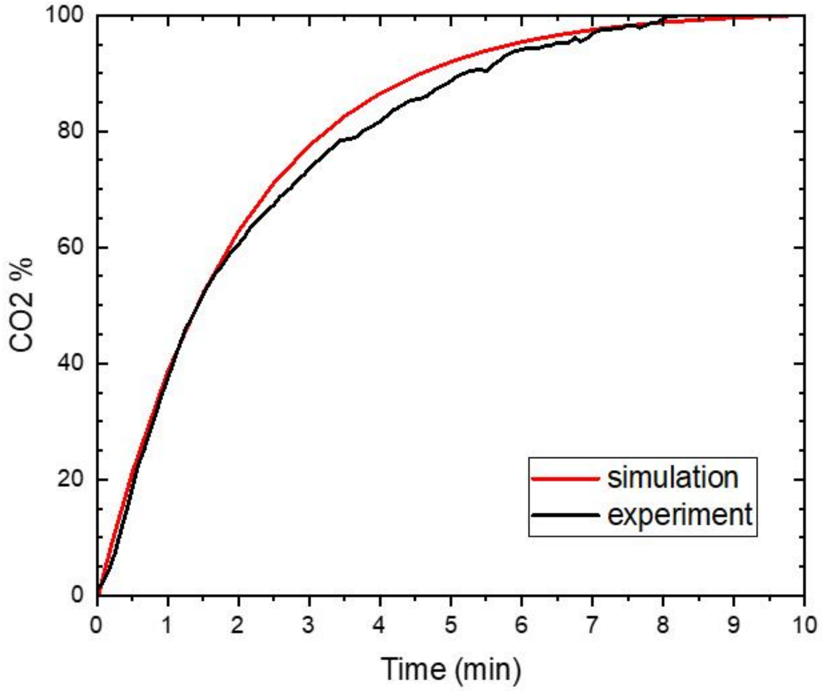

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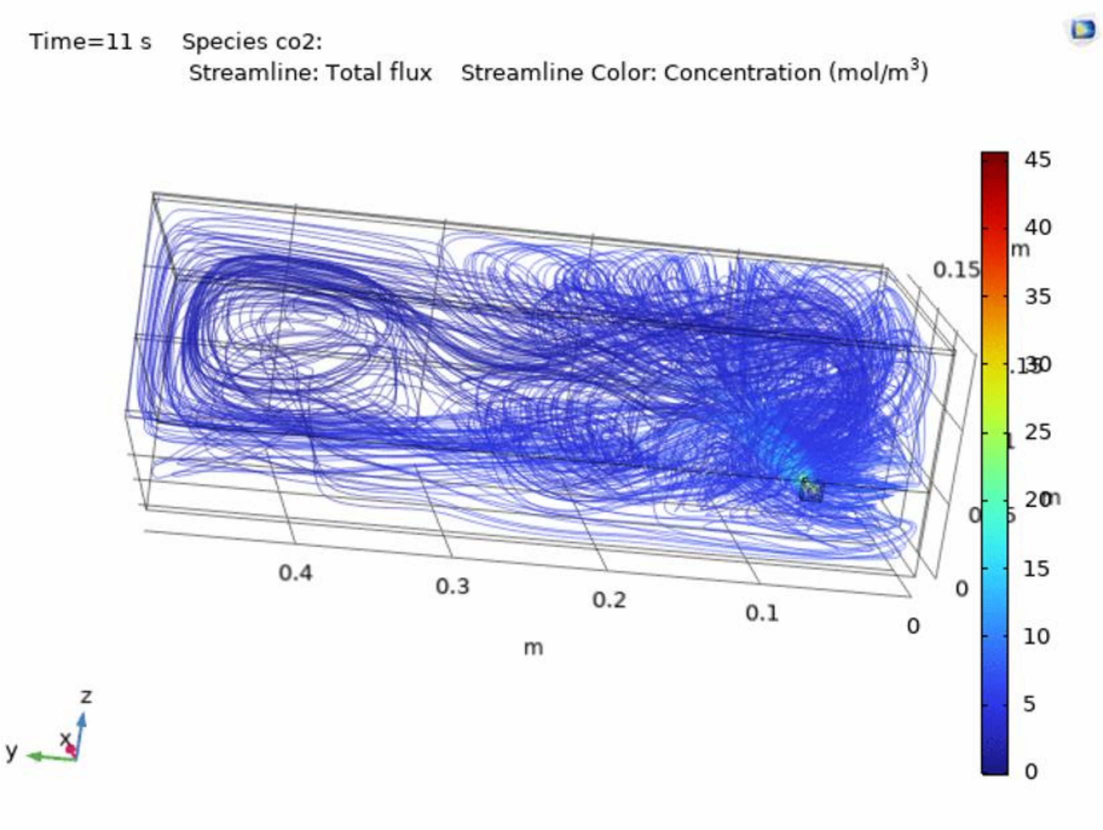

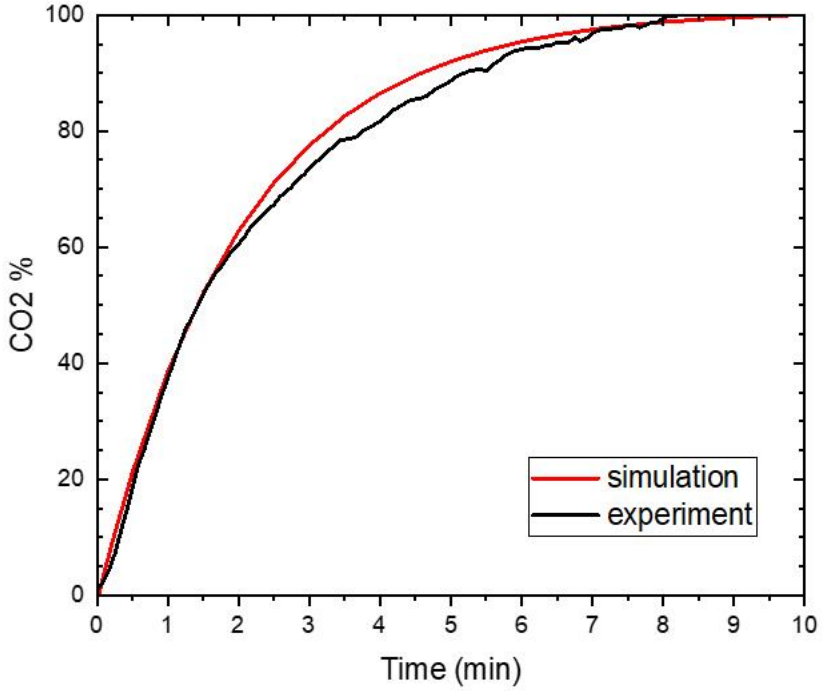

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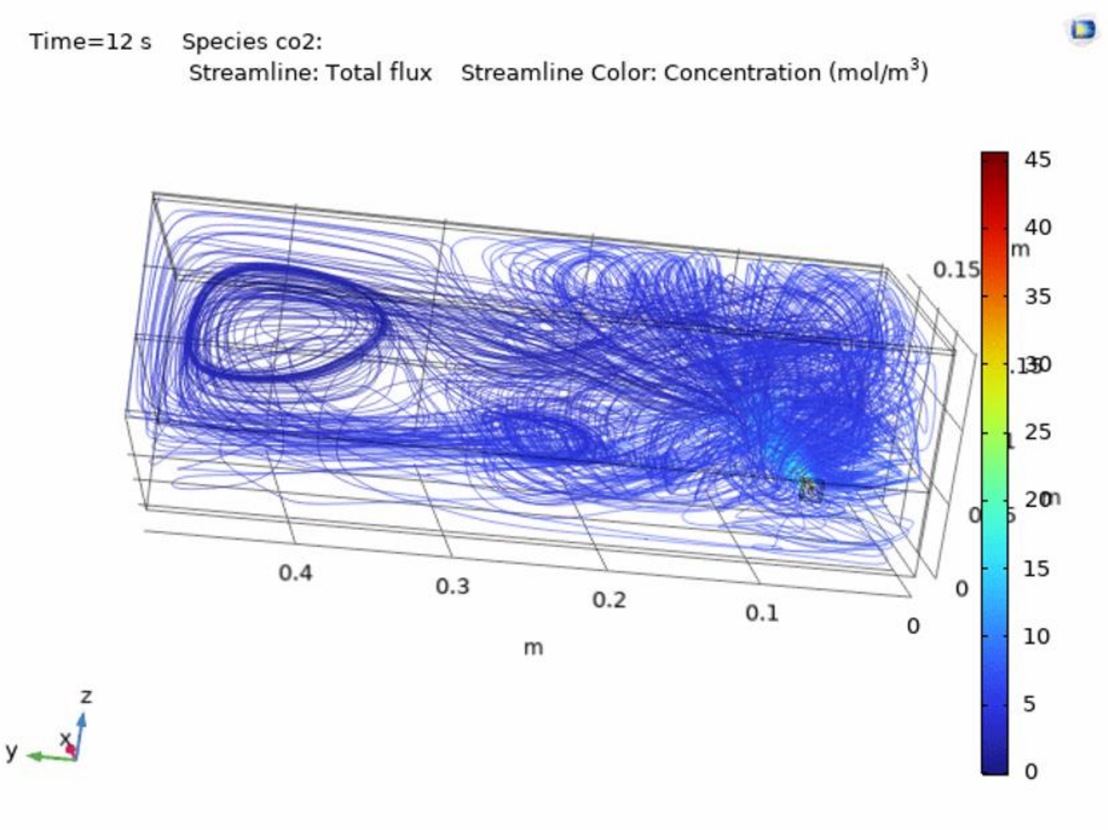

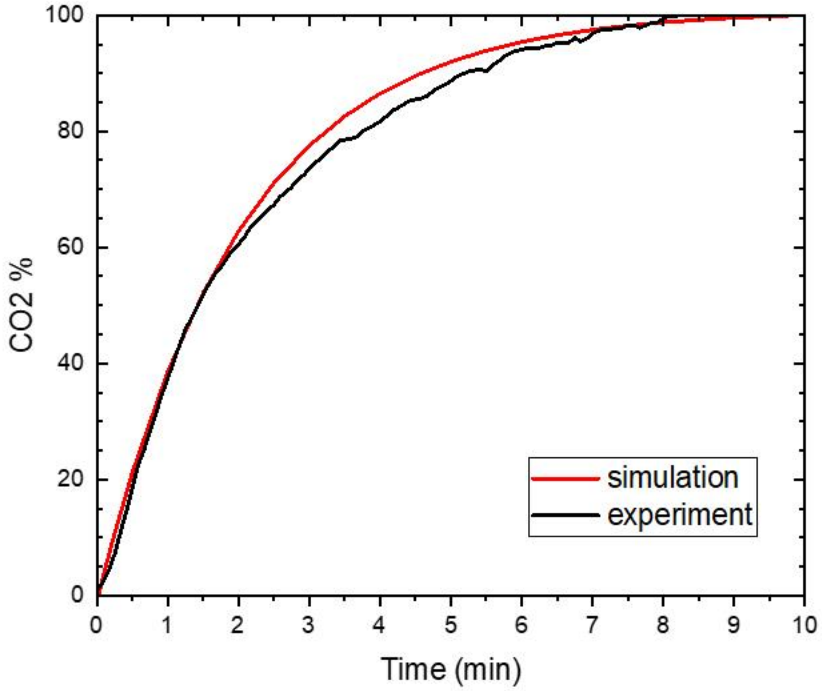

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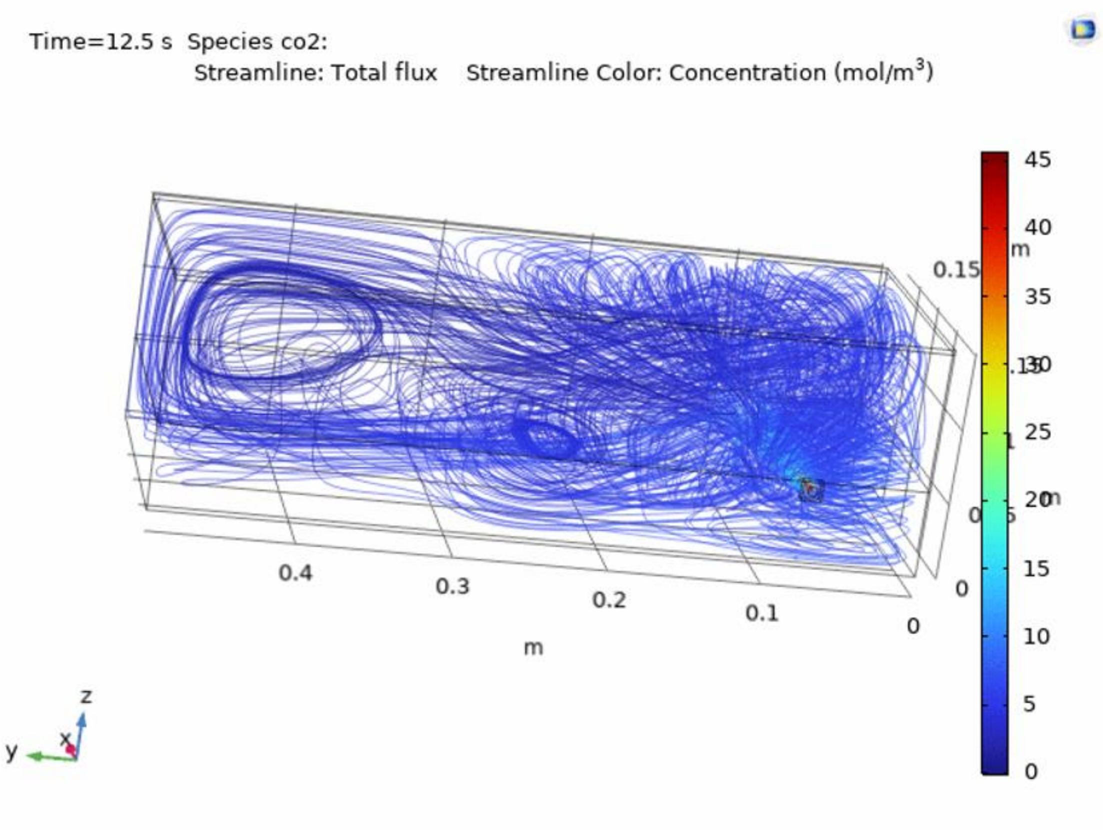

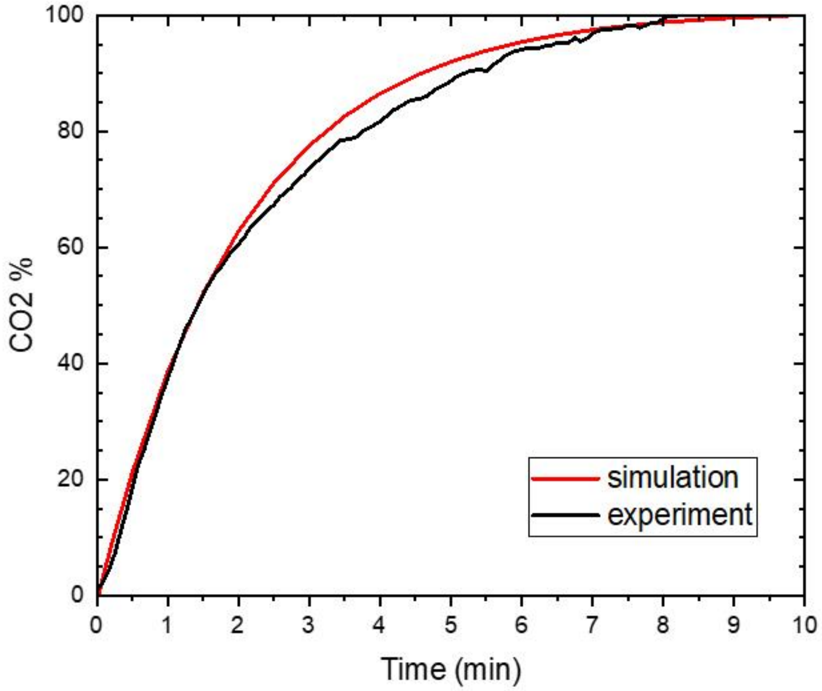

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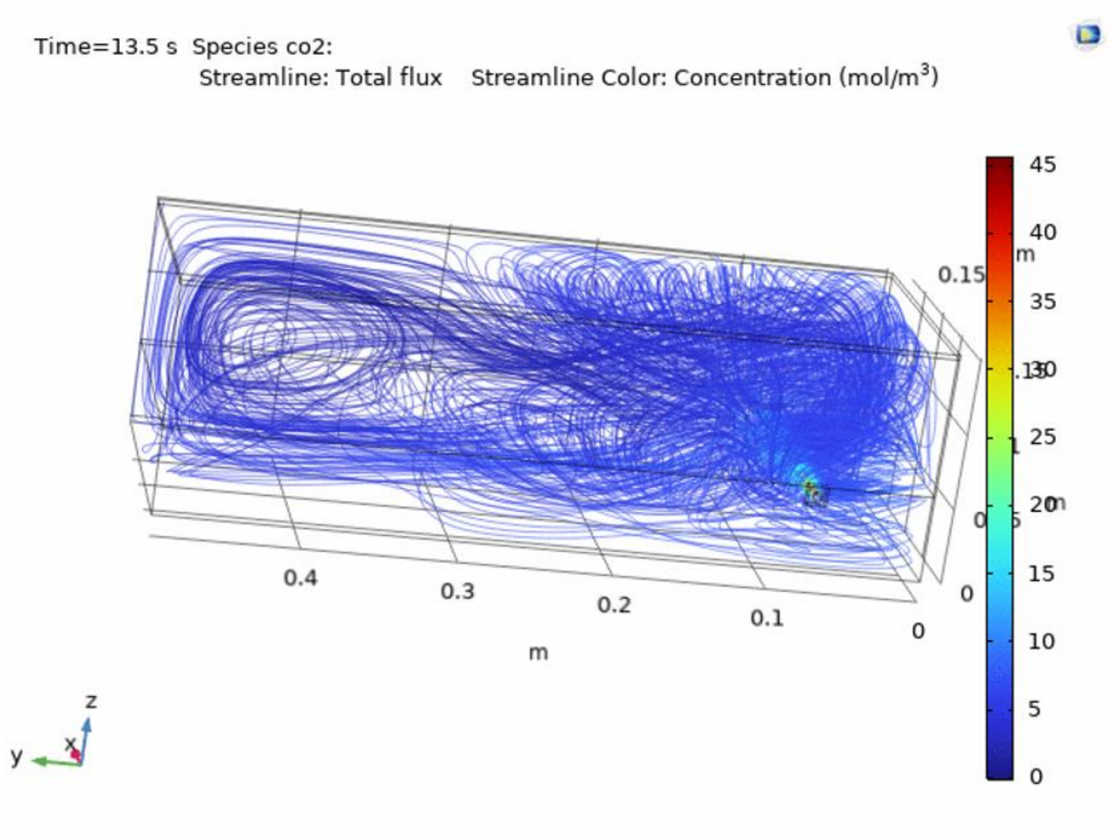

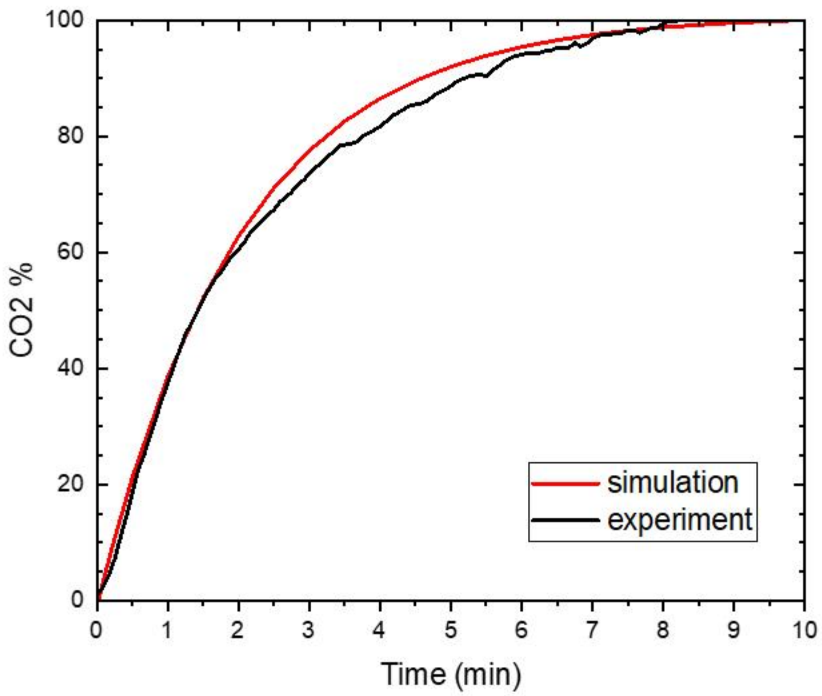

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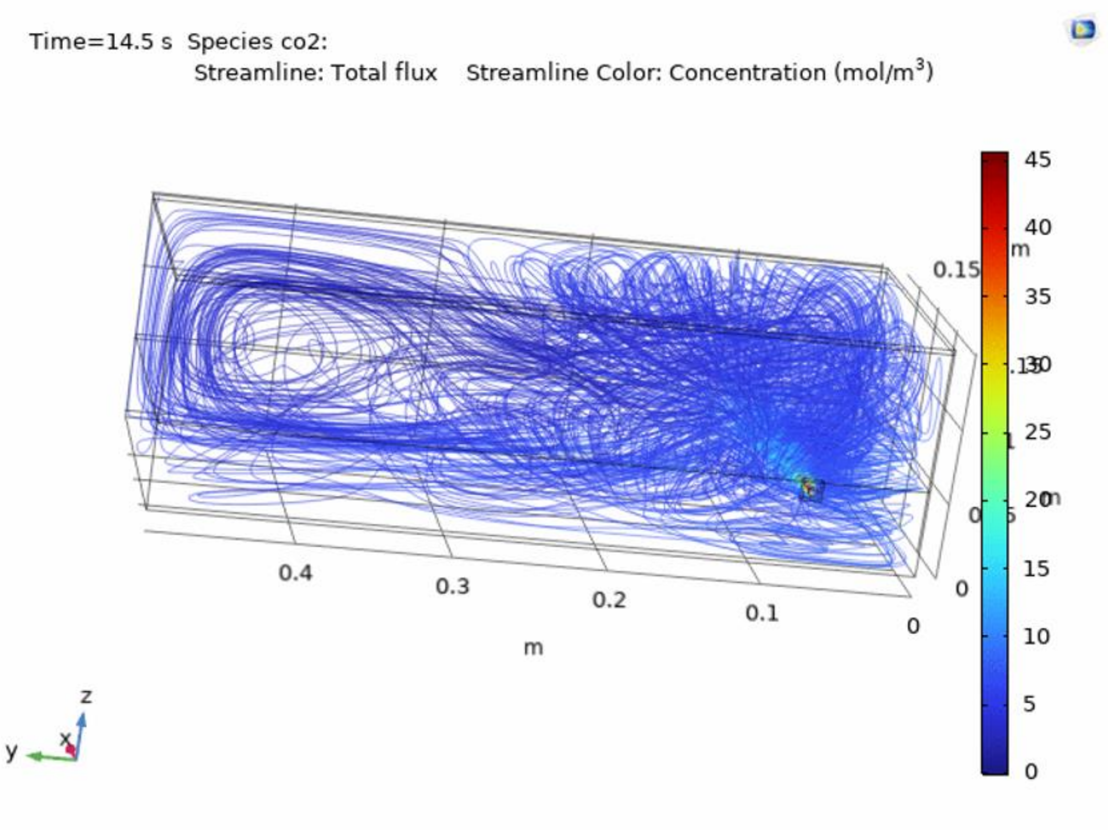

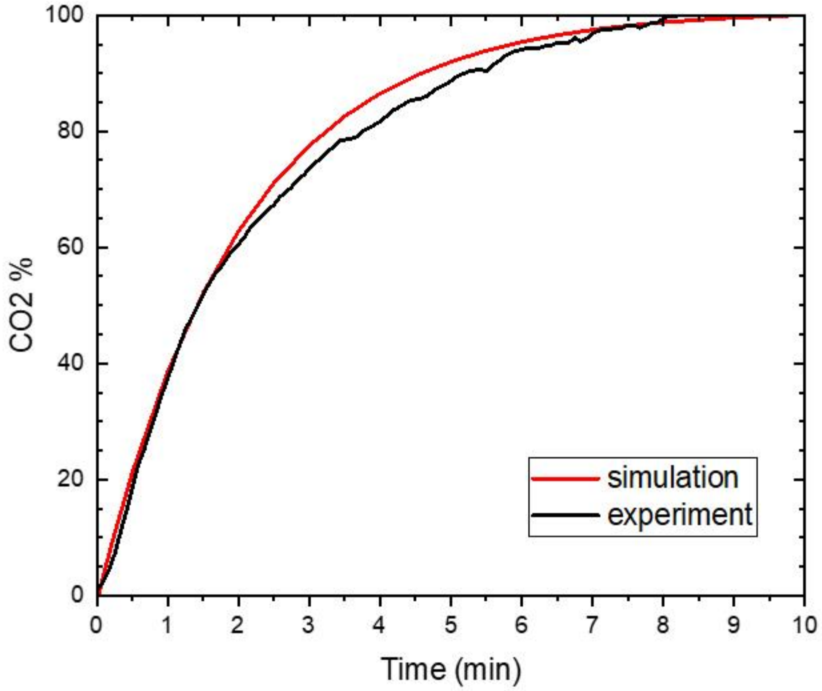

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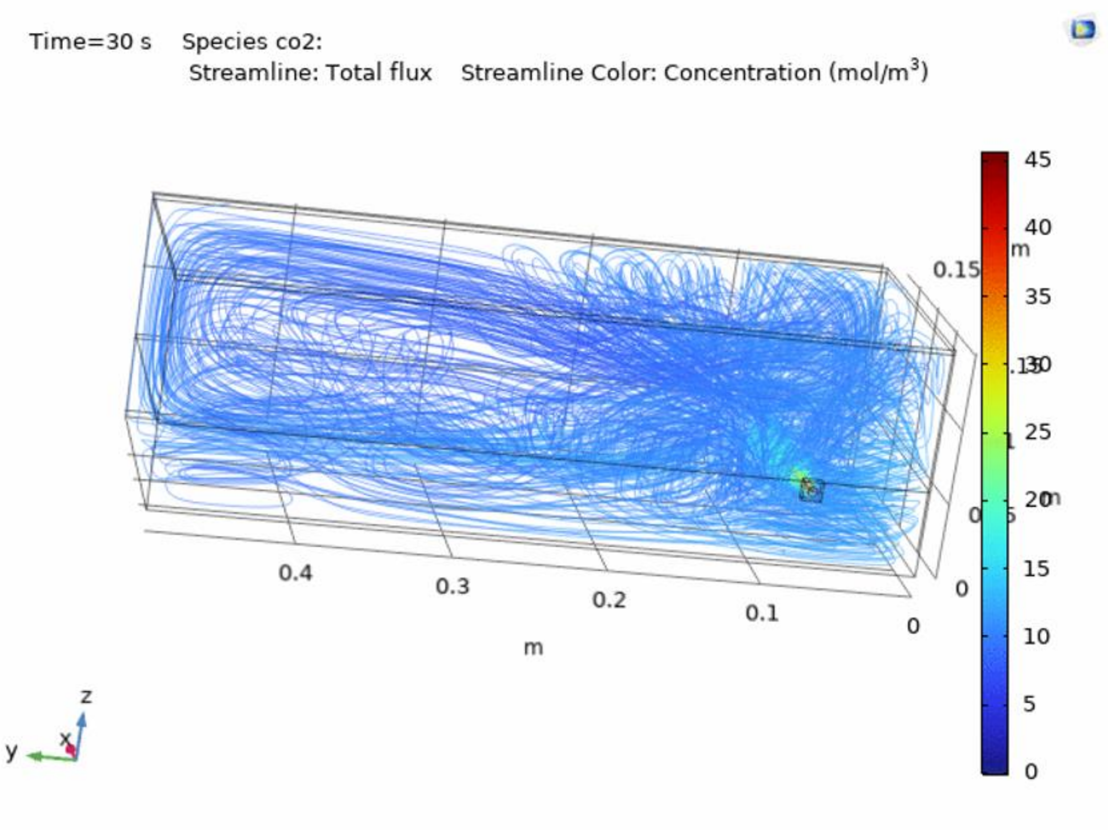

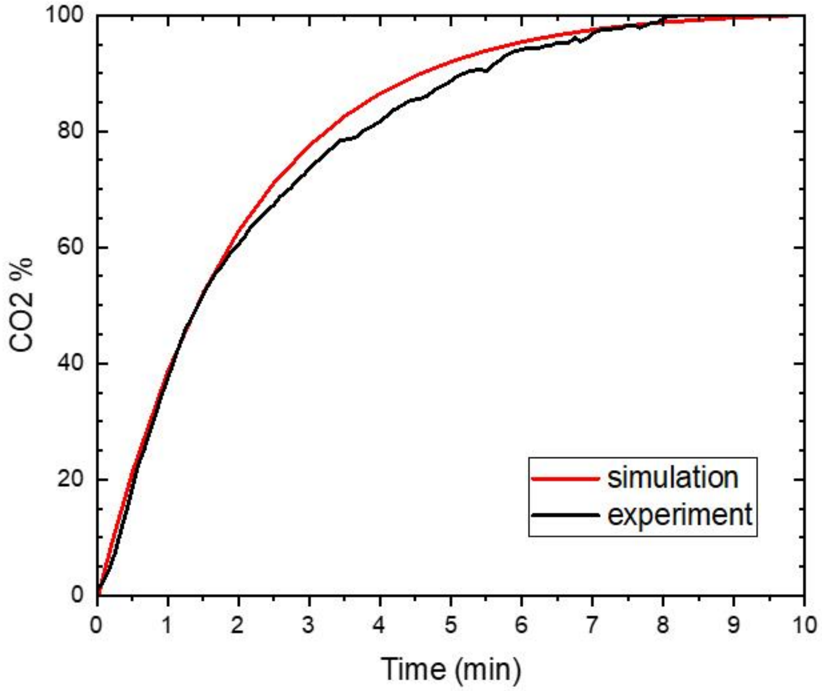

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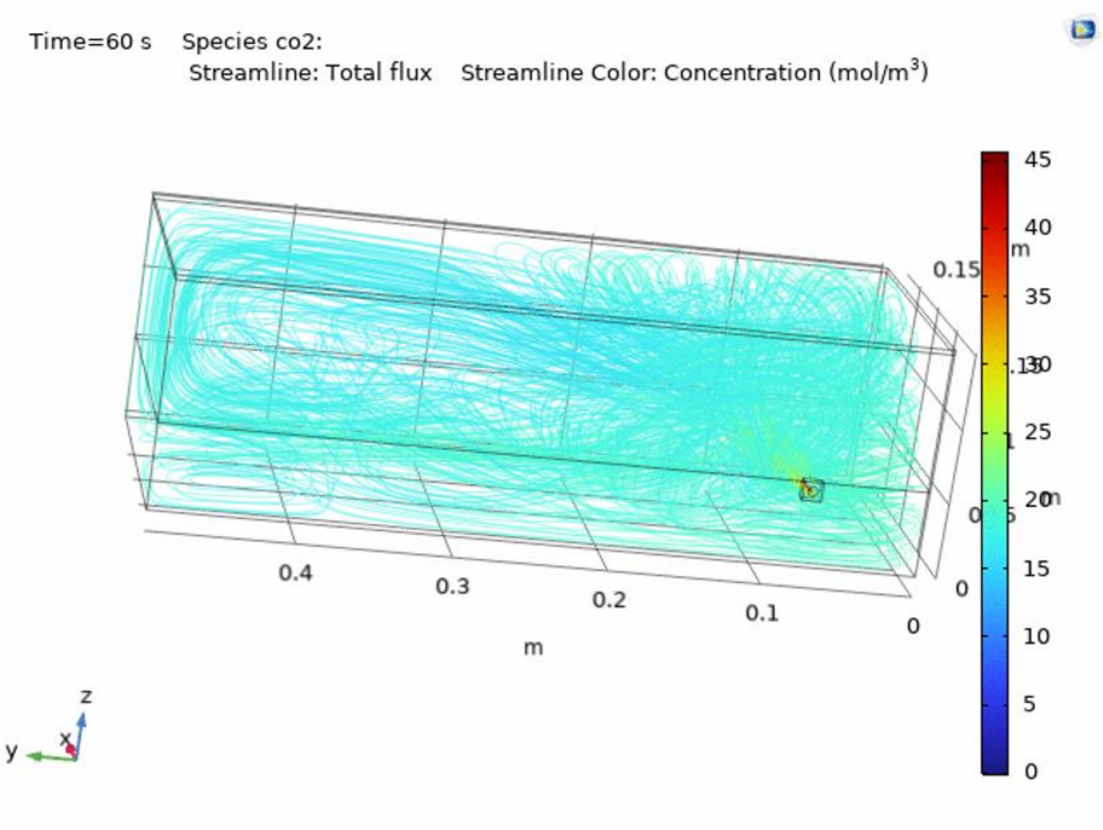

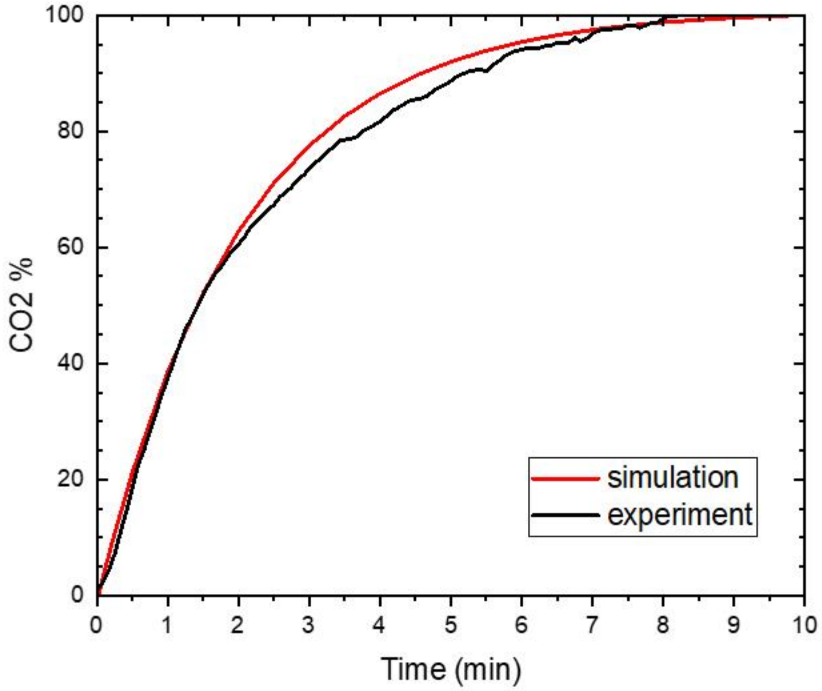

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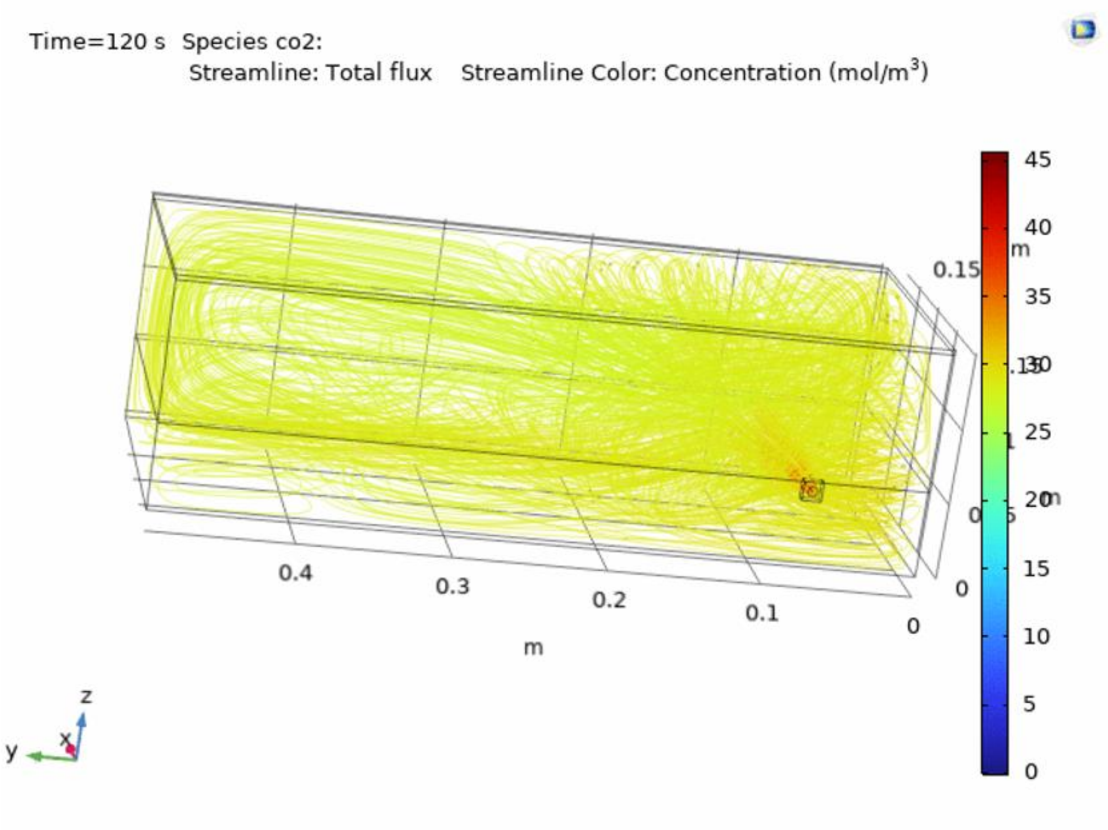

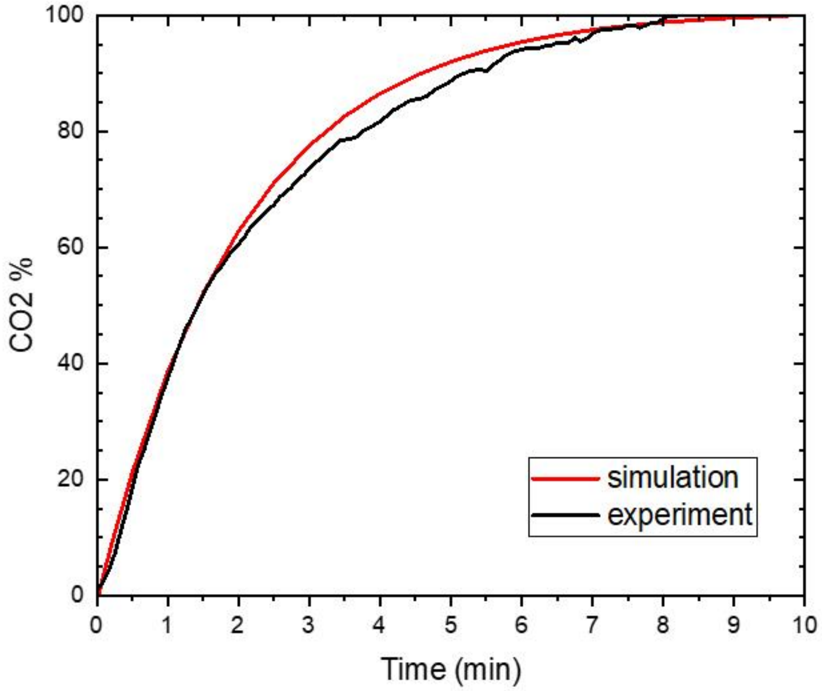

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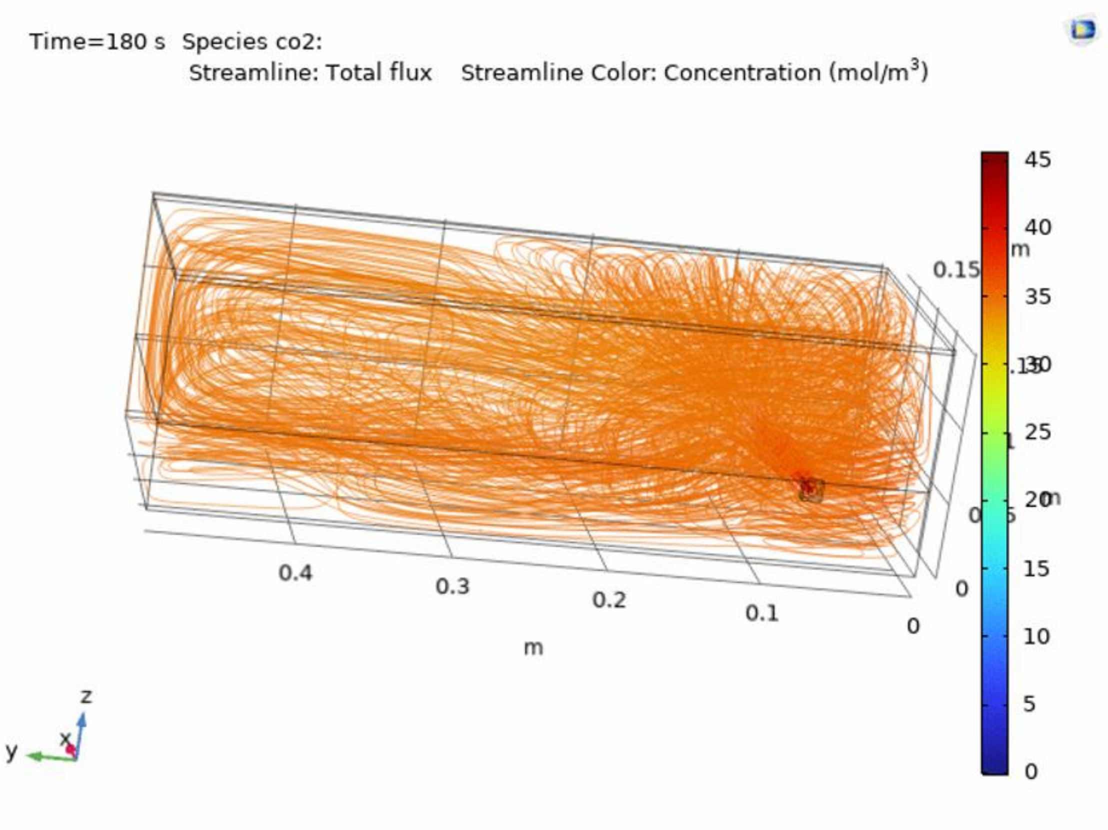

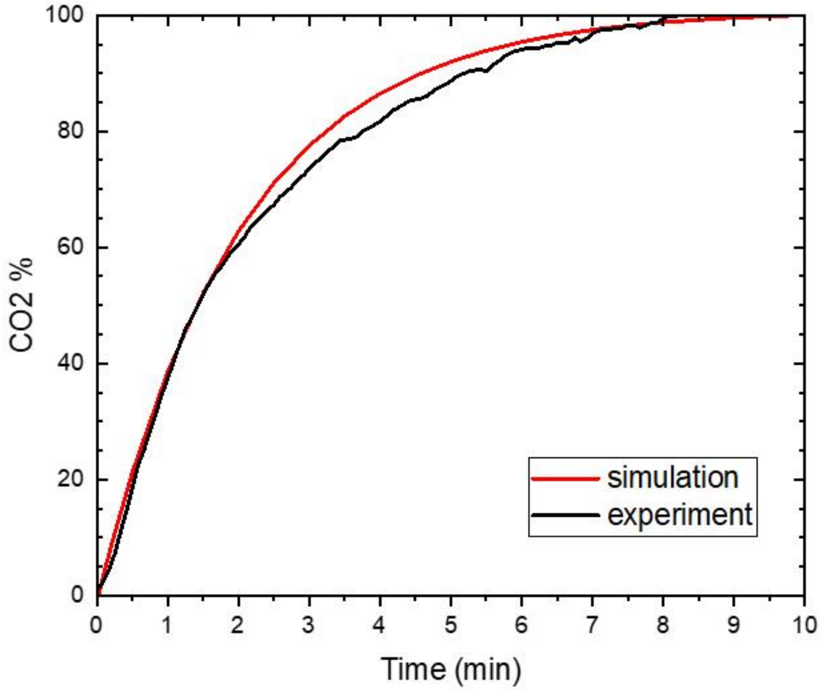

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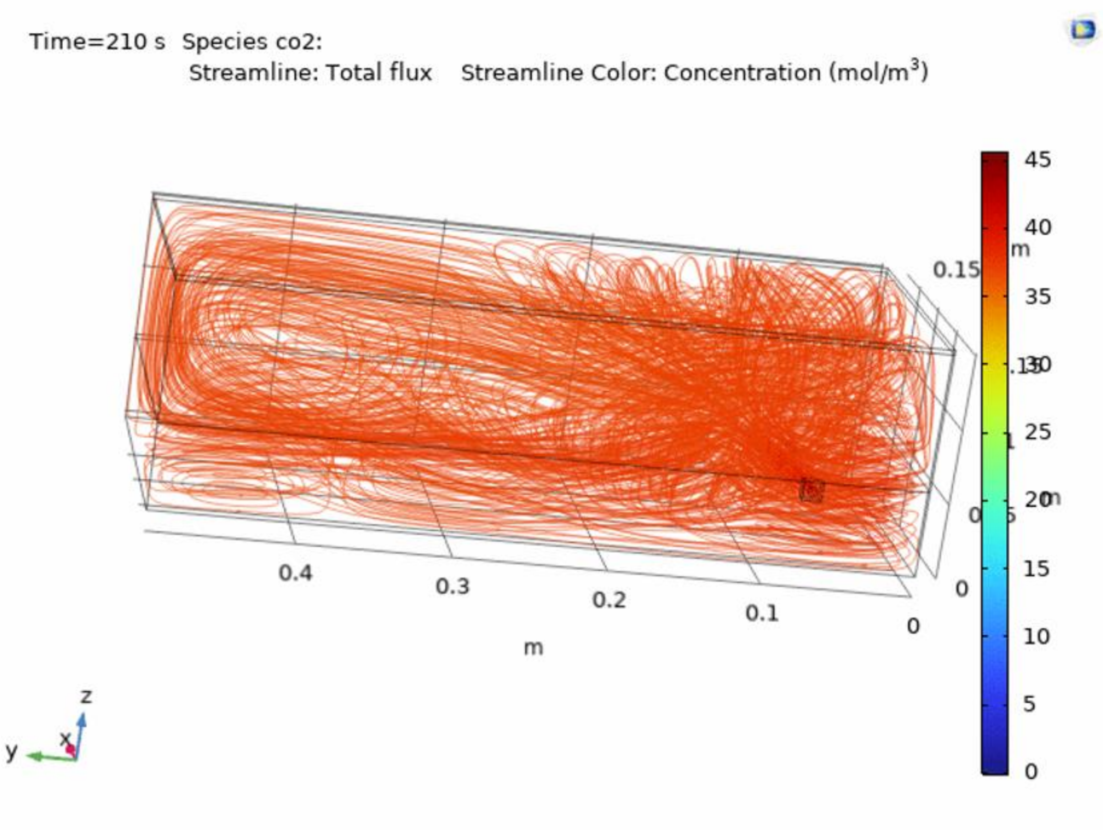

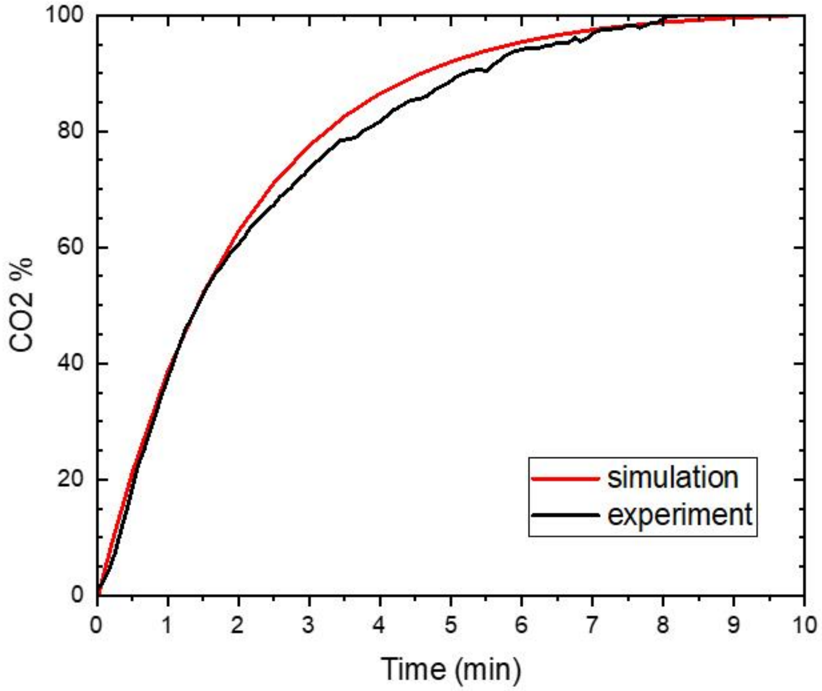

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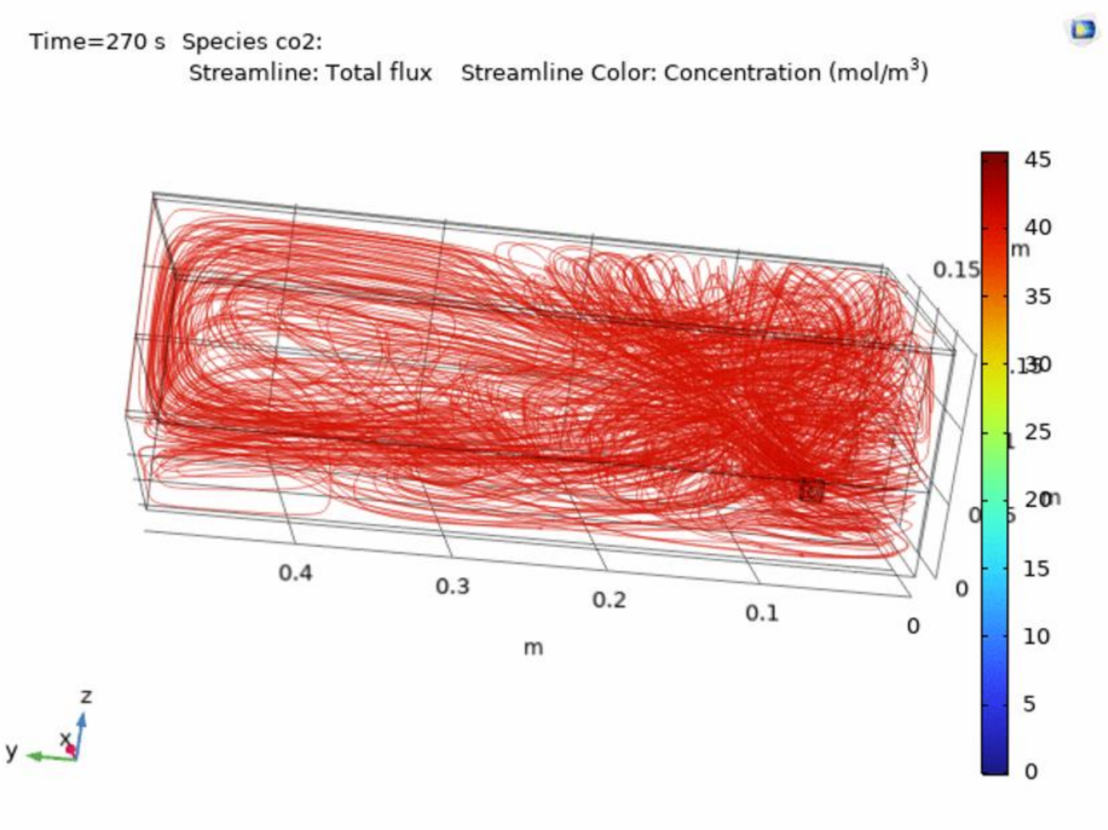

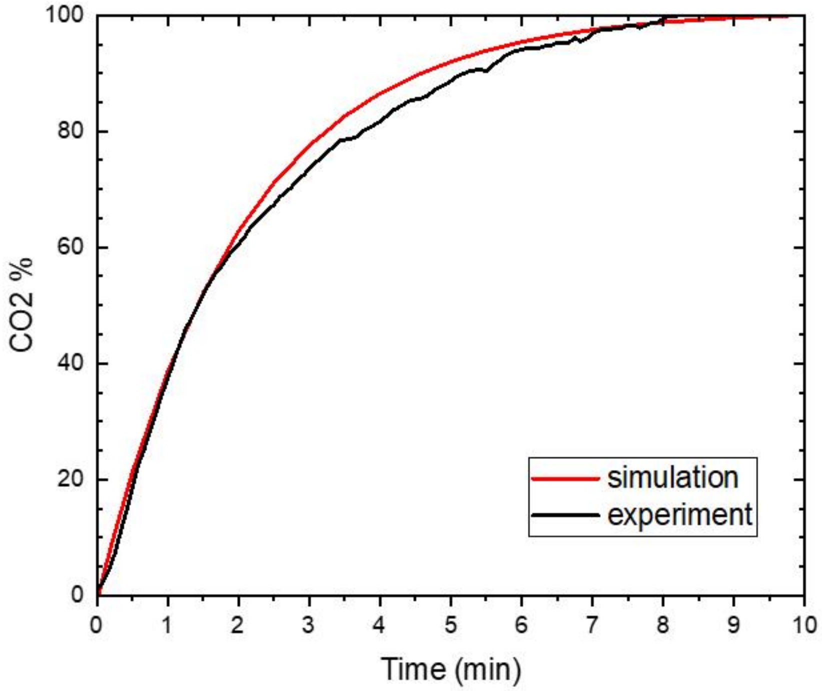

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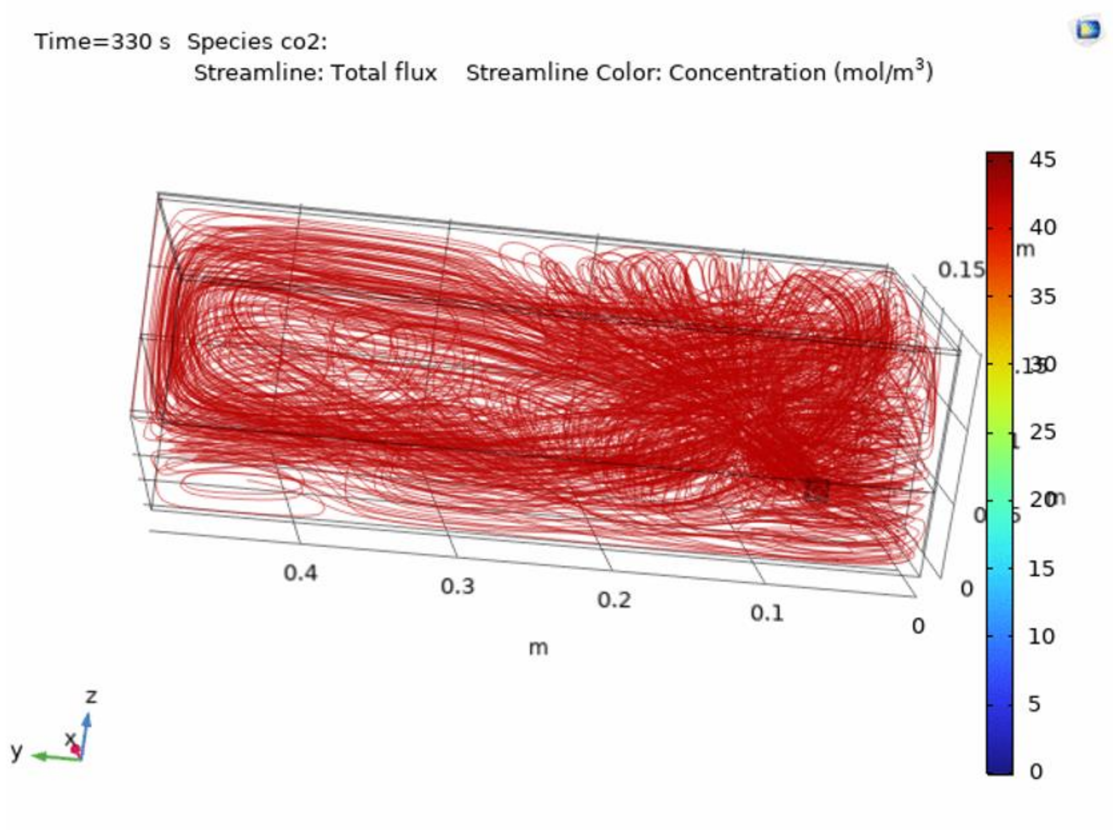

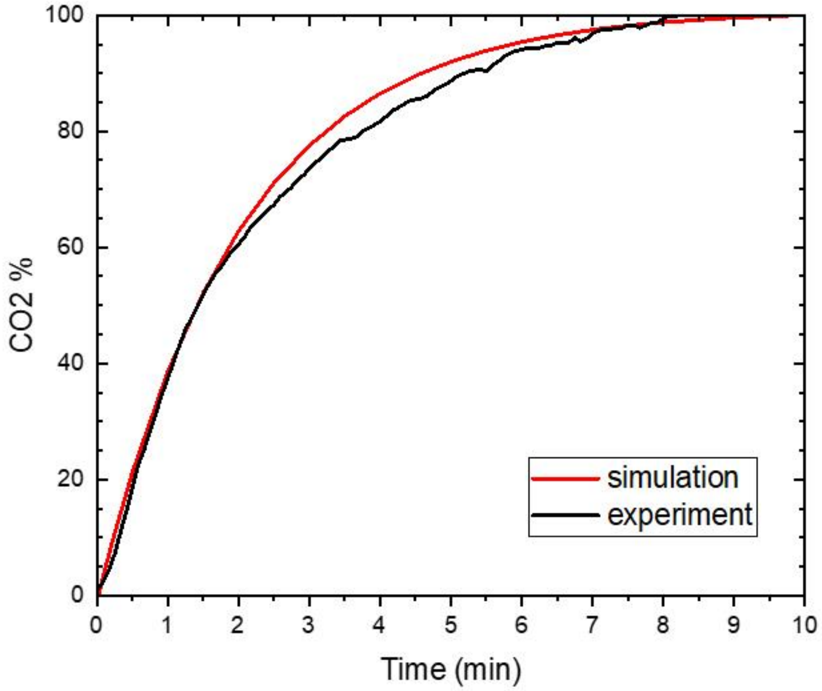

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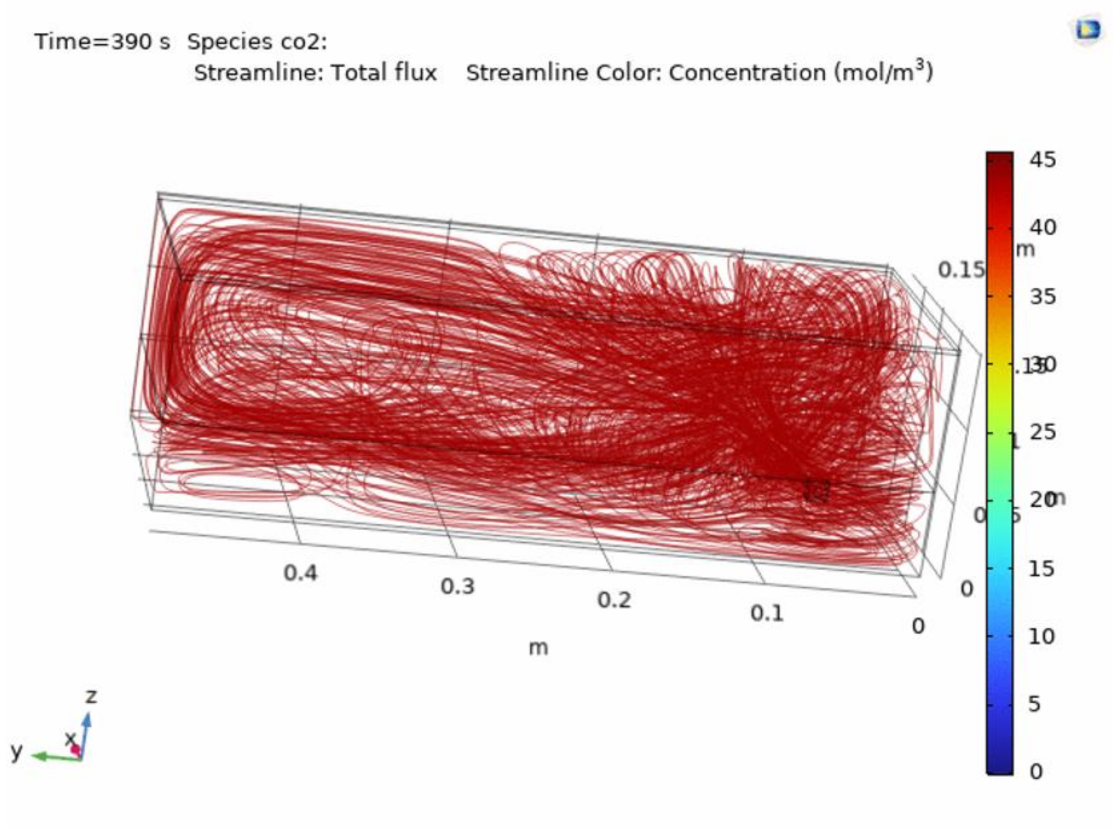

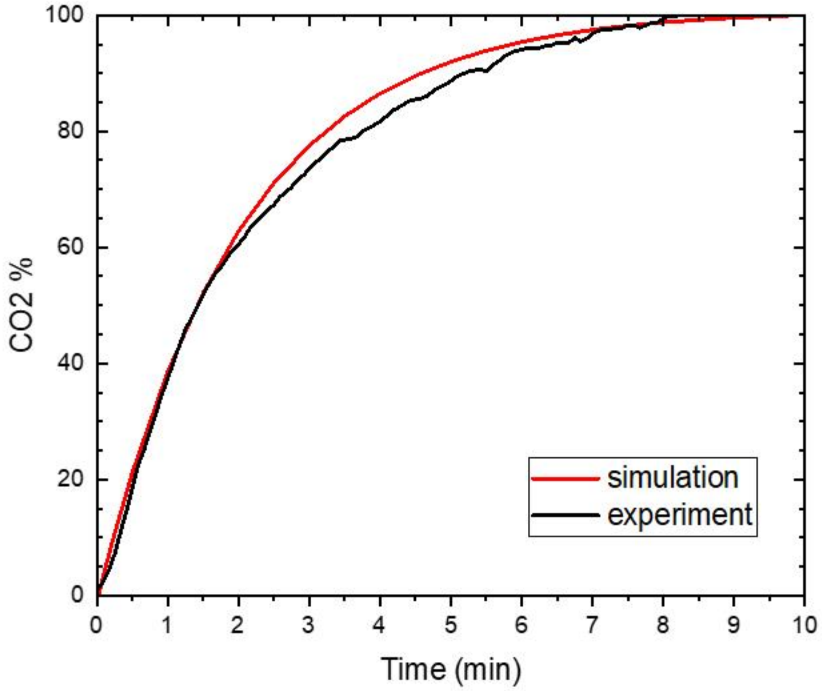

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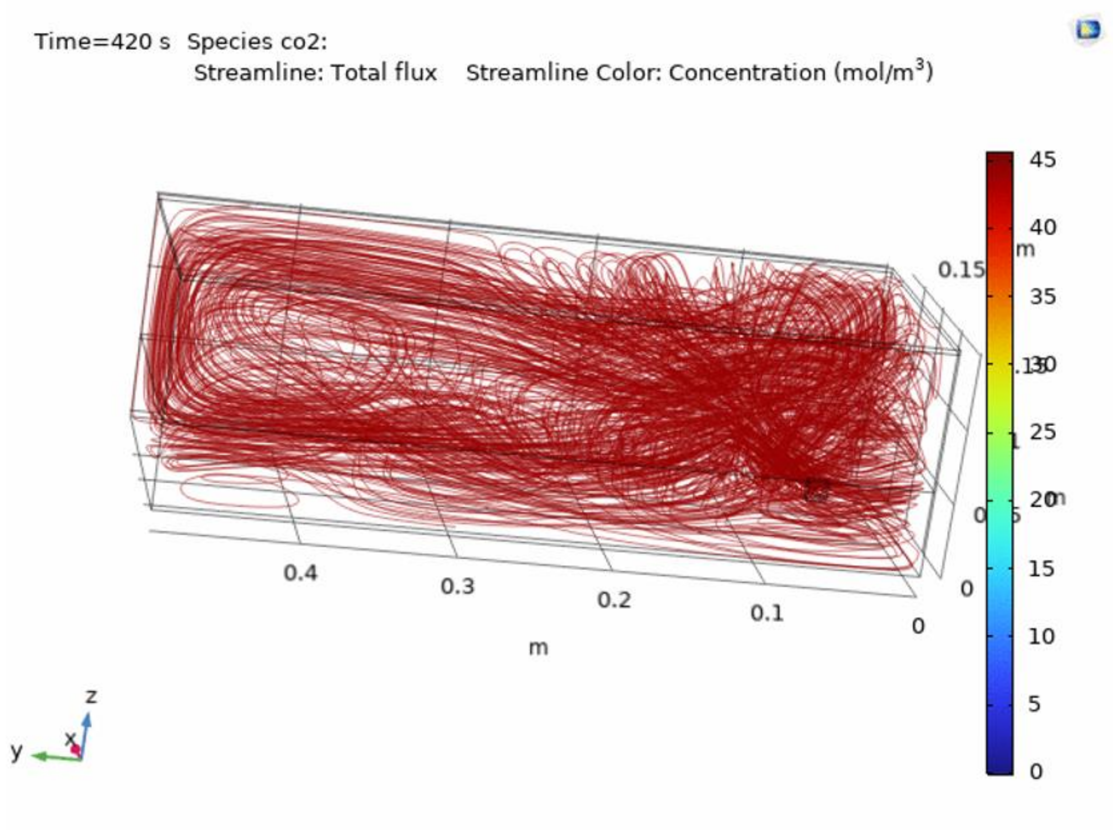

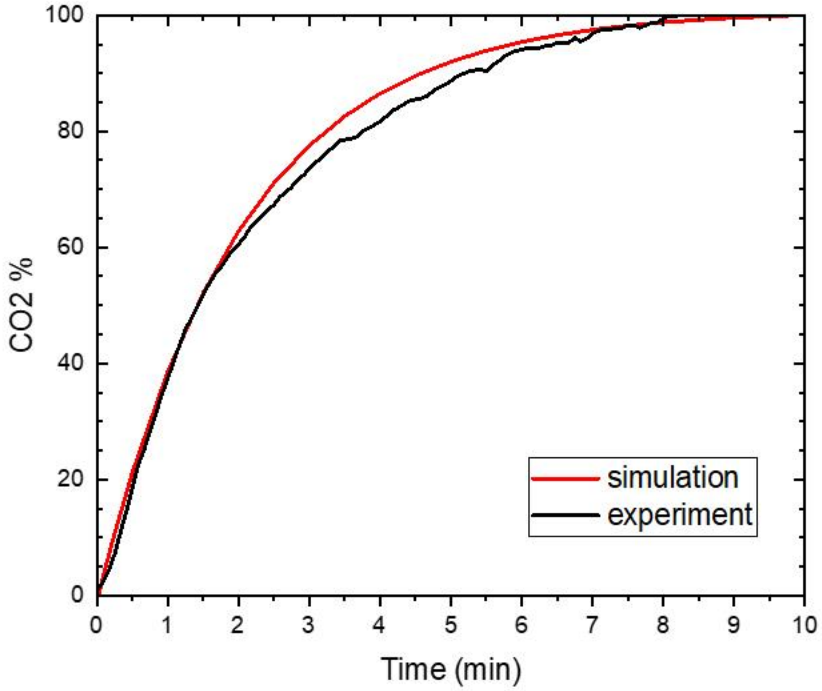

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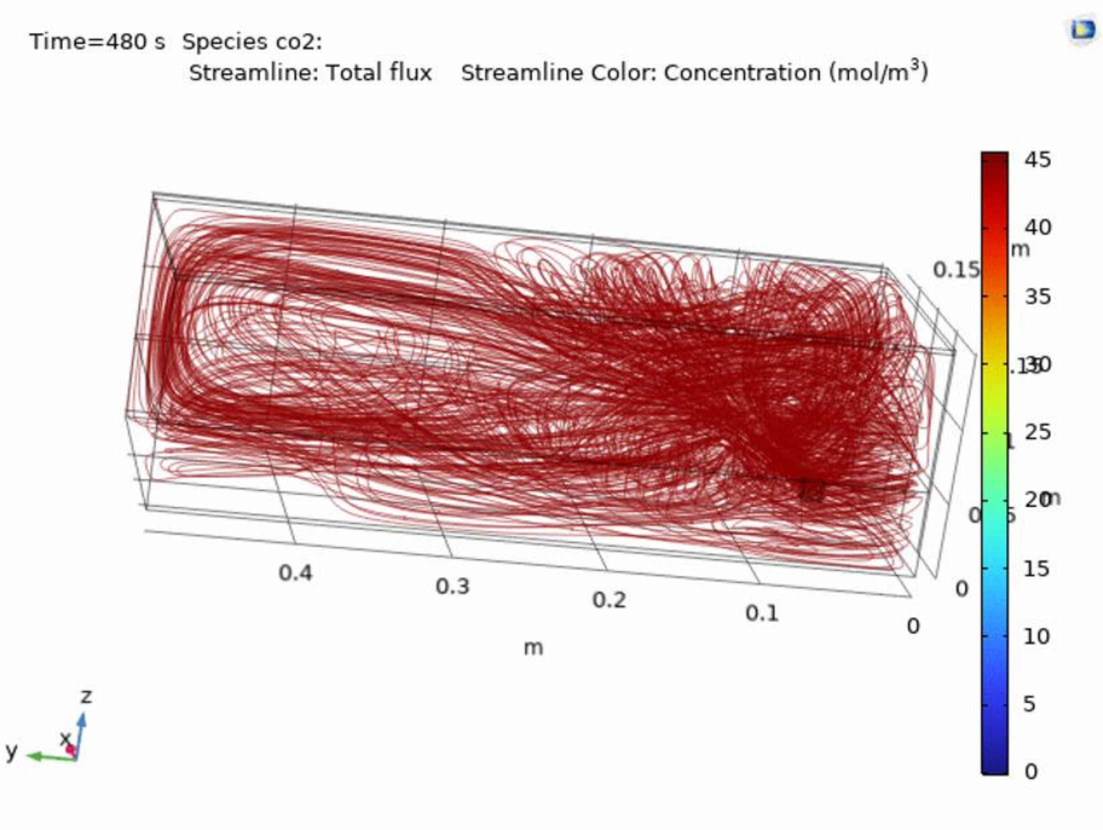

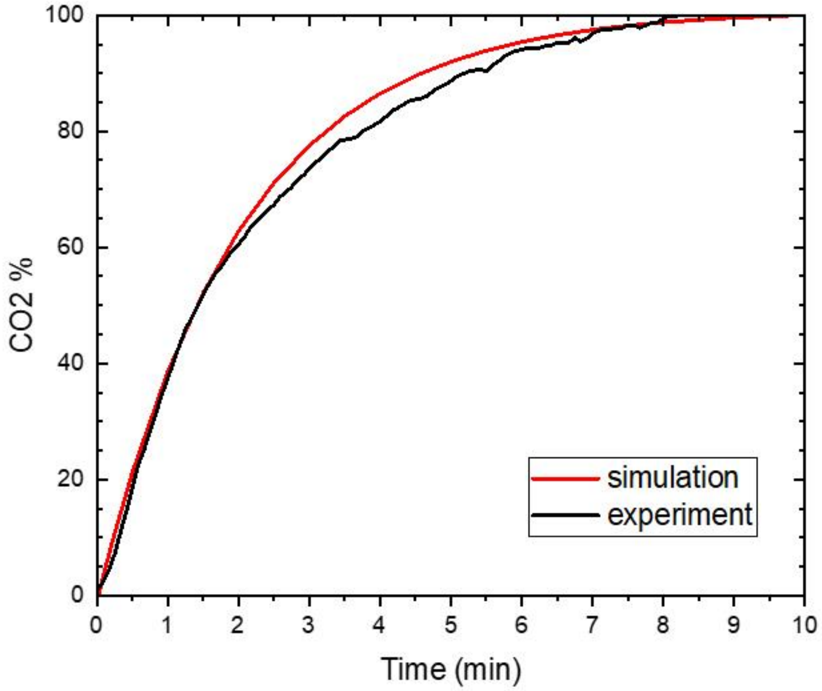

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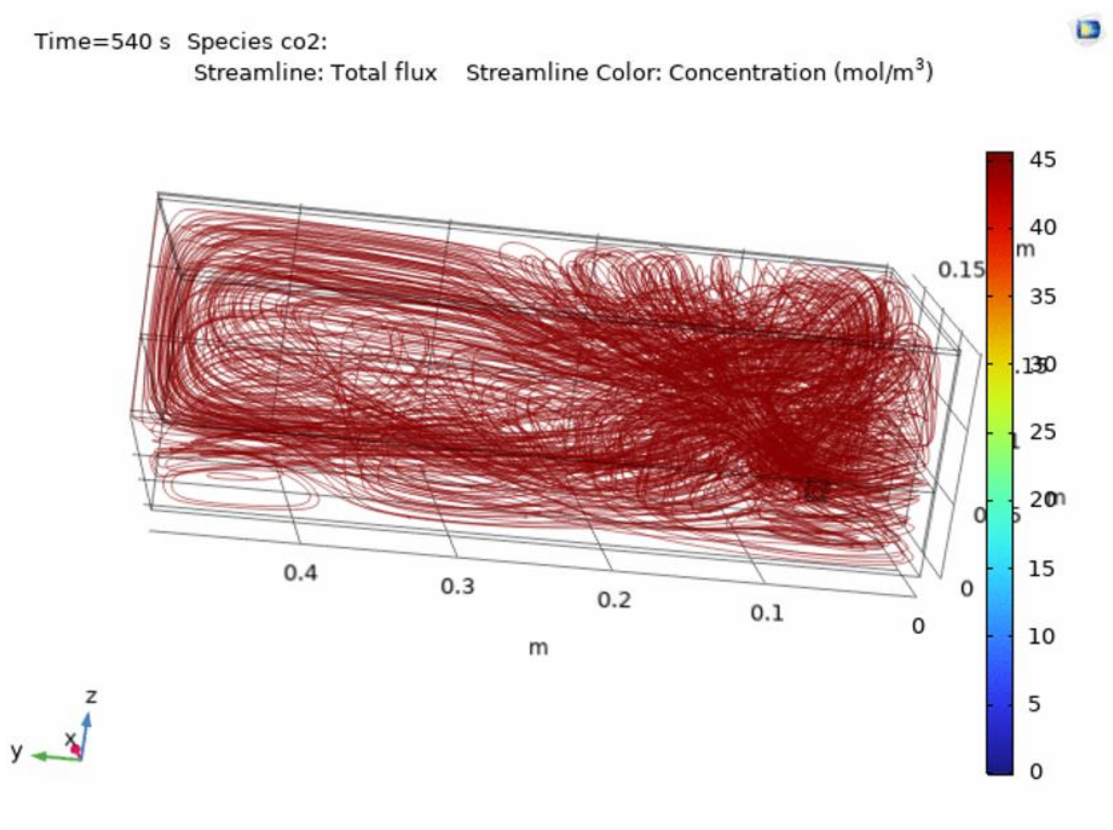

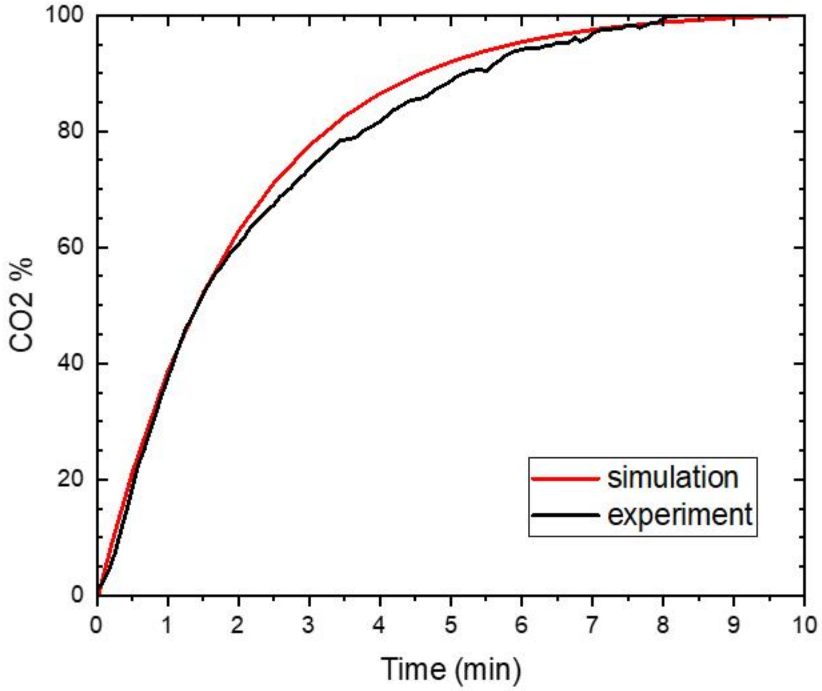

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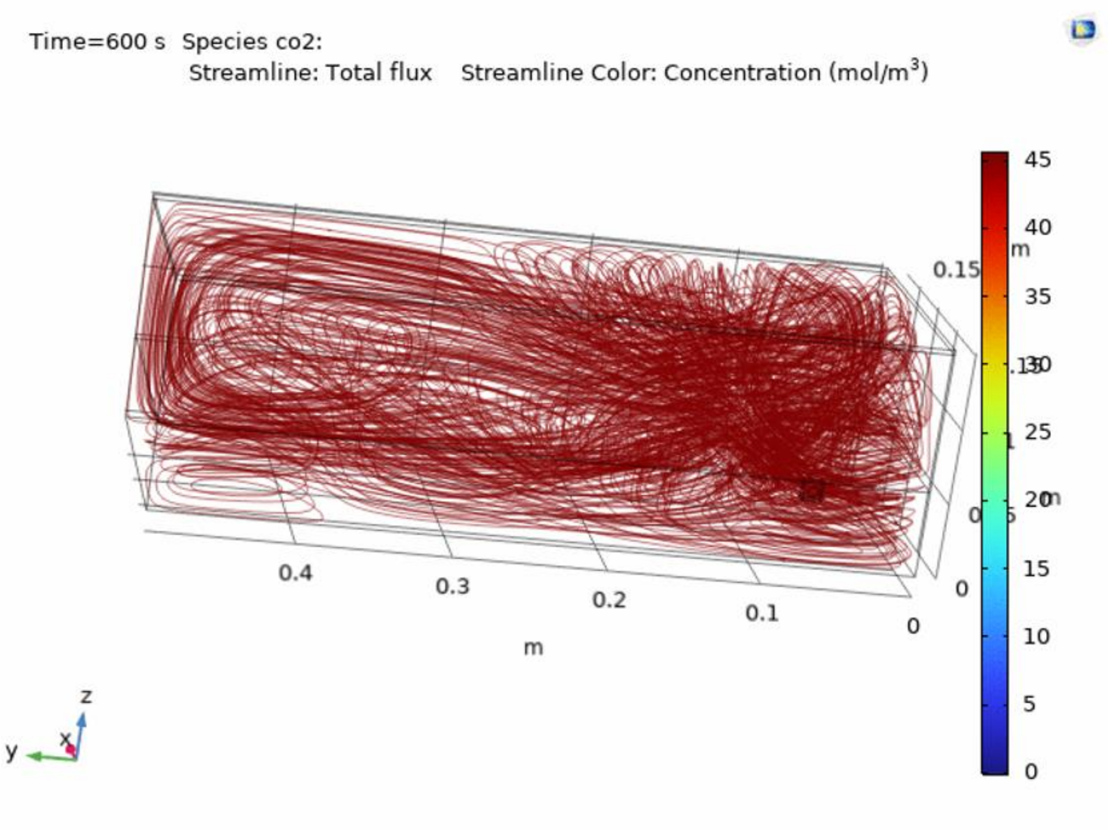

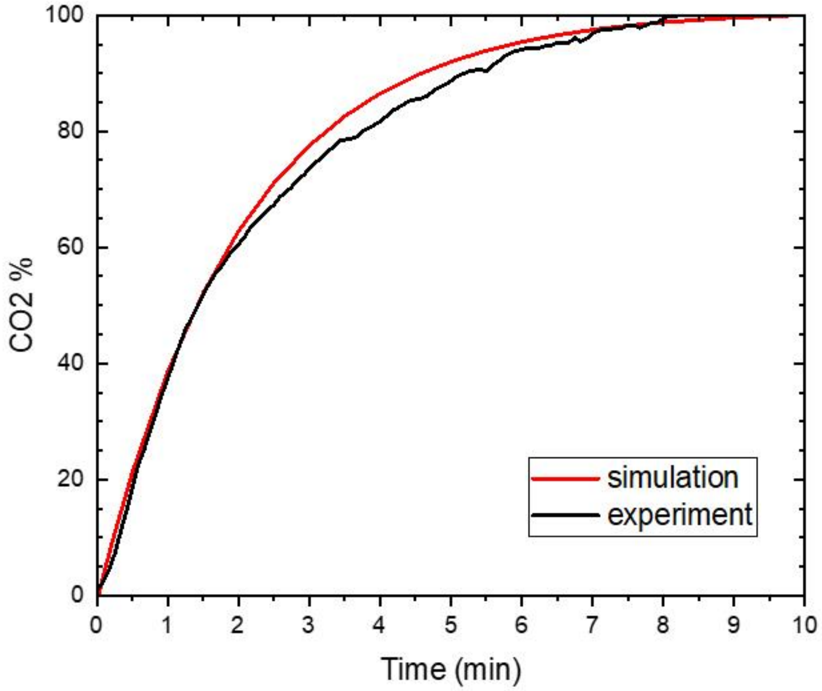

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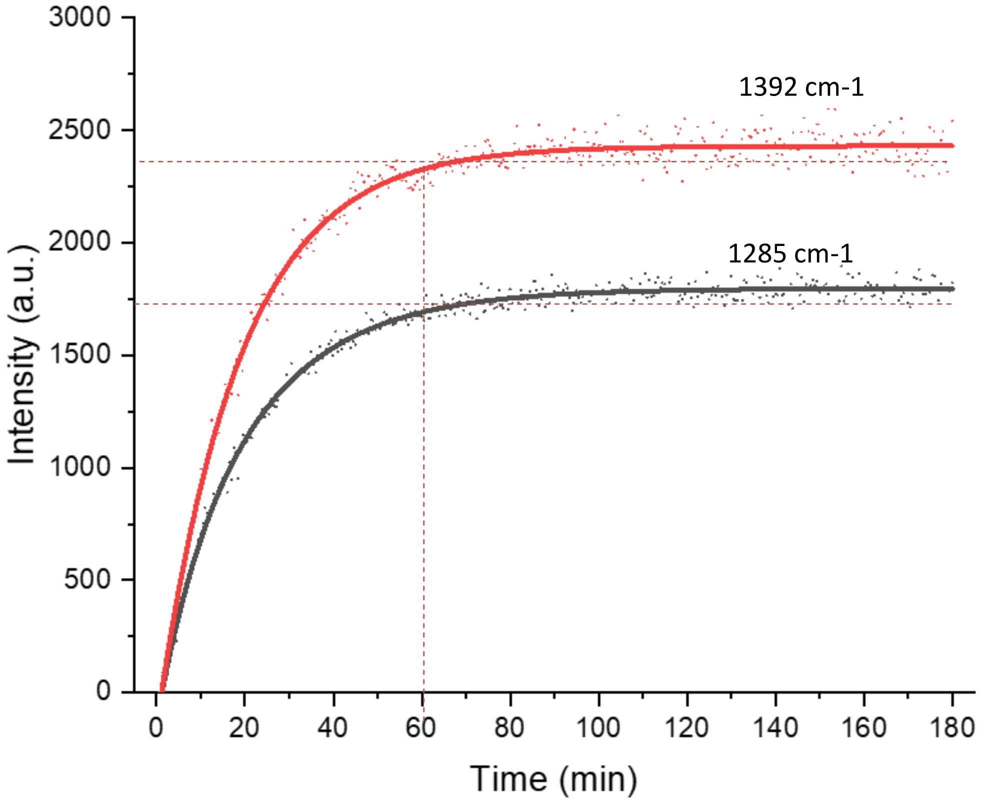
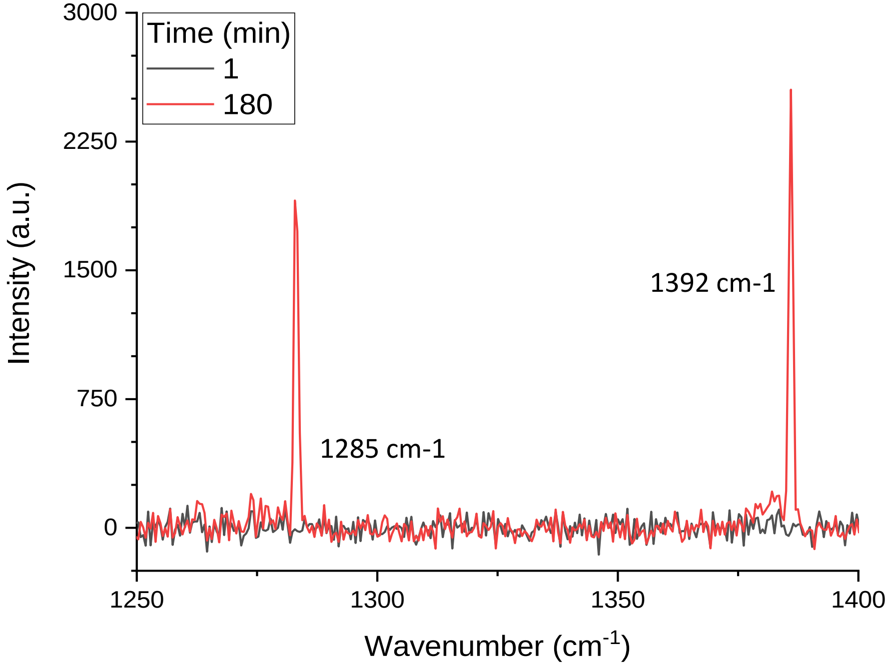
1.1a CO_2 concentration and uptake kinetics studies: from 0 to 20% CO_2 over 40 cm long HoF

Kinetics of CO_2 /N_2 mix in HoF

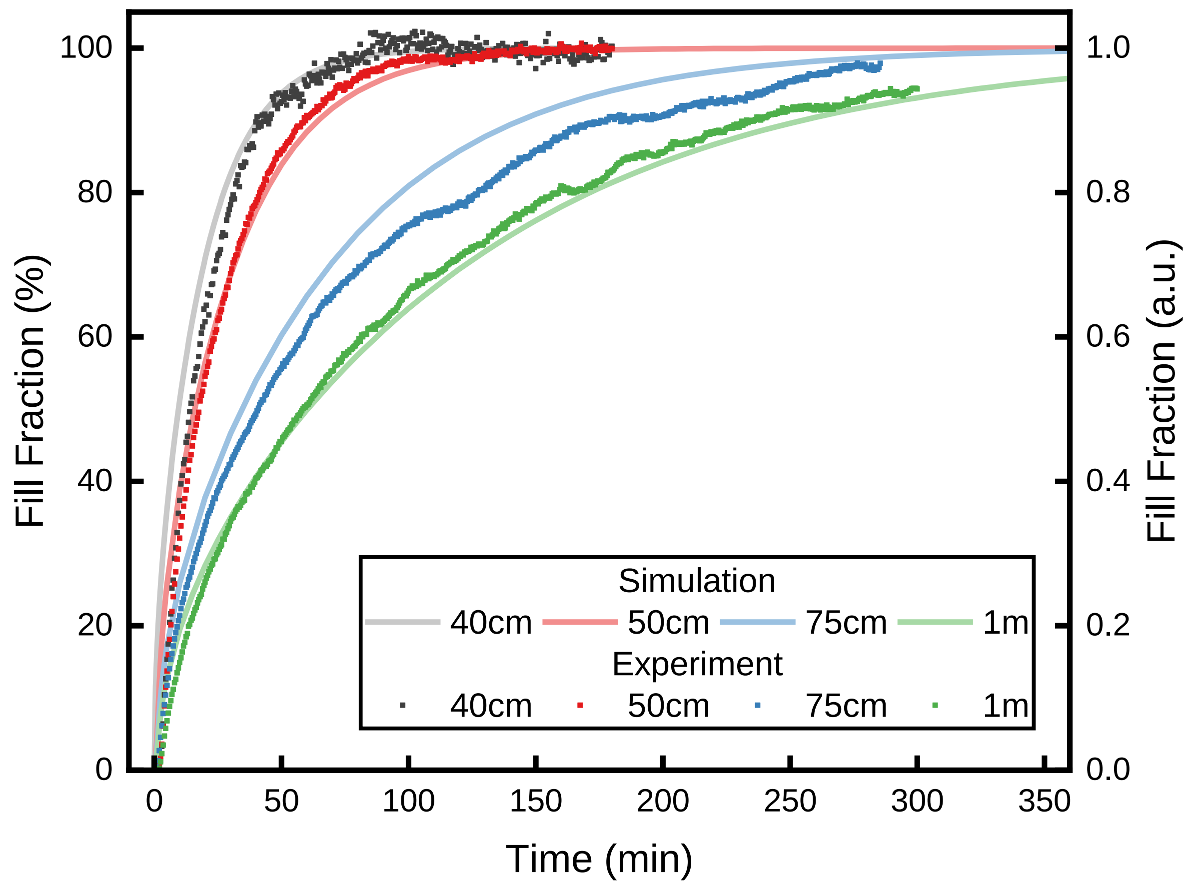


CO_2 Raman Spectrum in HoF ; λ_{ex}= 785 nm



We are able to fit the uptake curves with a exponential – the uptake time to reach 95% filling ~ 60 minutes .

1.1a COMSOL model of gas diffusion into hollow core fibers validate and provide guidance on expected filling times for various fiber length



Gas diffuses into fiber core through open facets at both fiber ends

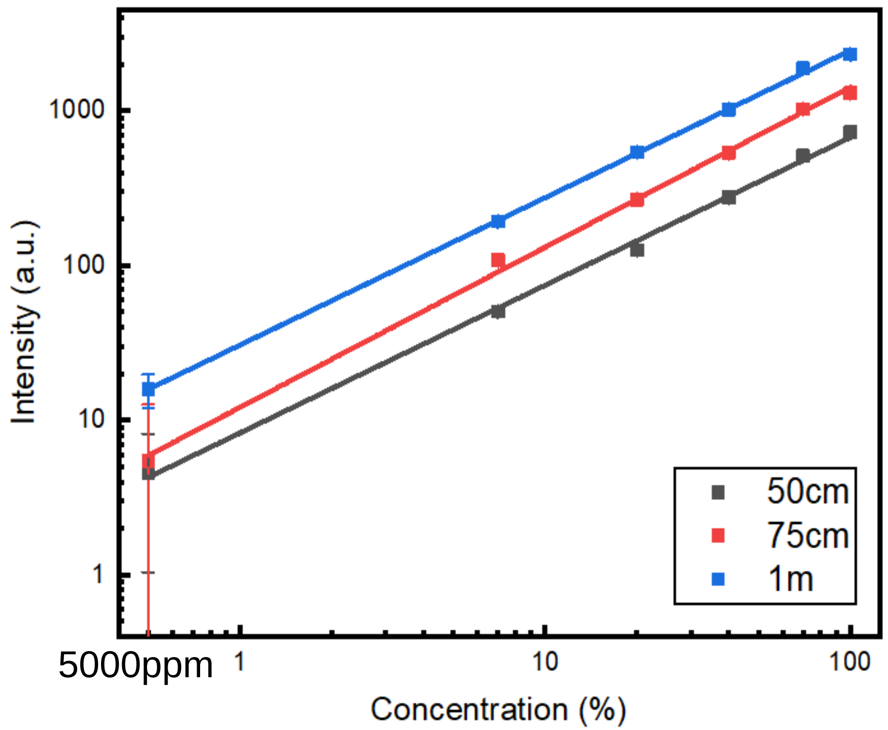
Convection-diffusion model coupled with Navier-Stokes in slow channel approximation (height of channel smaller than length)

Time to reach 95% CO2 (min)		
Fiber	**Simulation**	**Experiment**
40cm	54.5	59
50cm	90	77.5
75cm	200	248
1m	340	300 (94%)

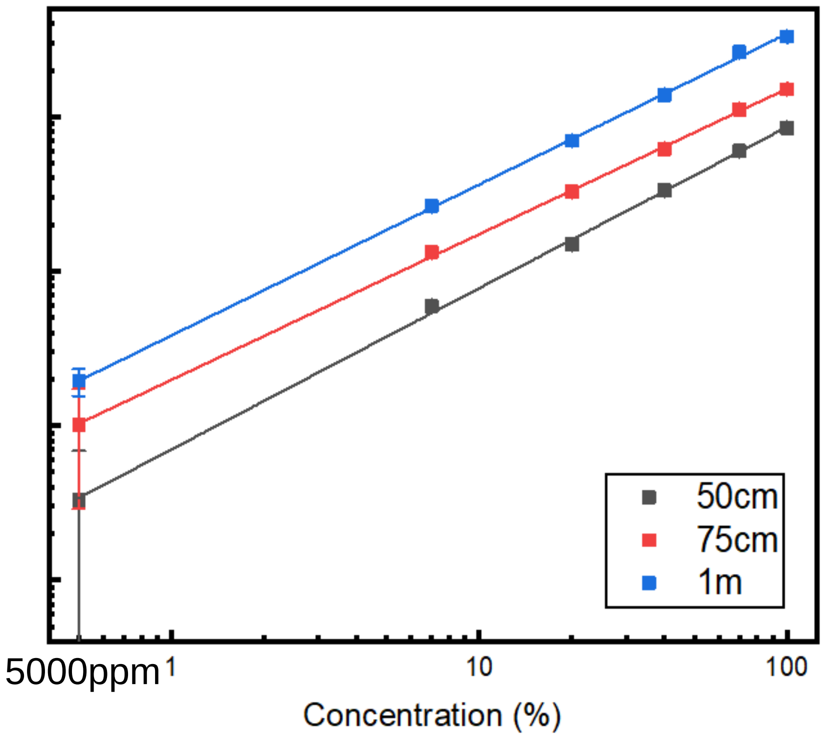
Δ~15%

1.1a Raman intensity vs. CO_2 concentration for different HoF lengths in 785nm setup shows expected behavior

1285 peak

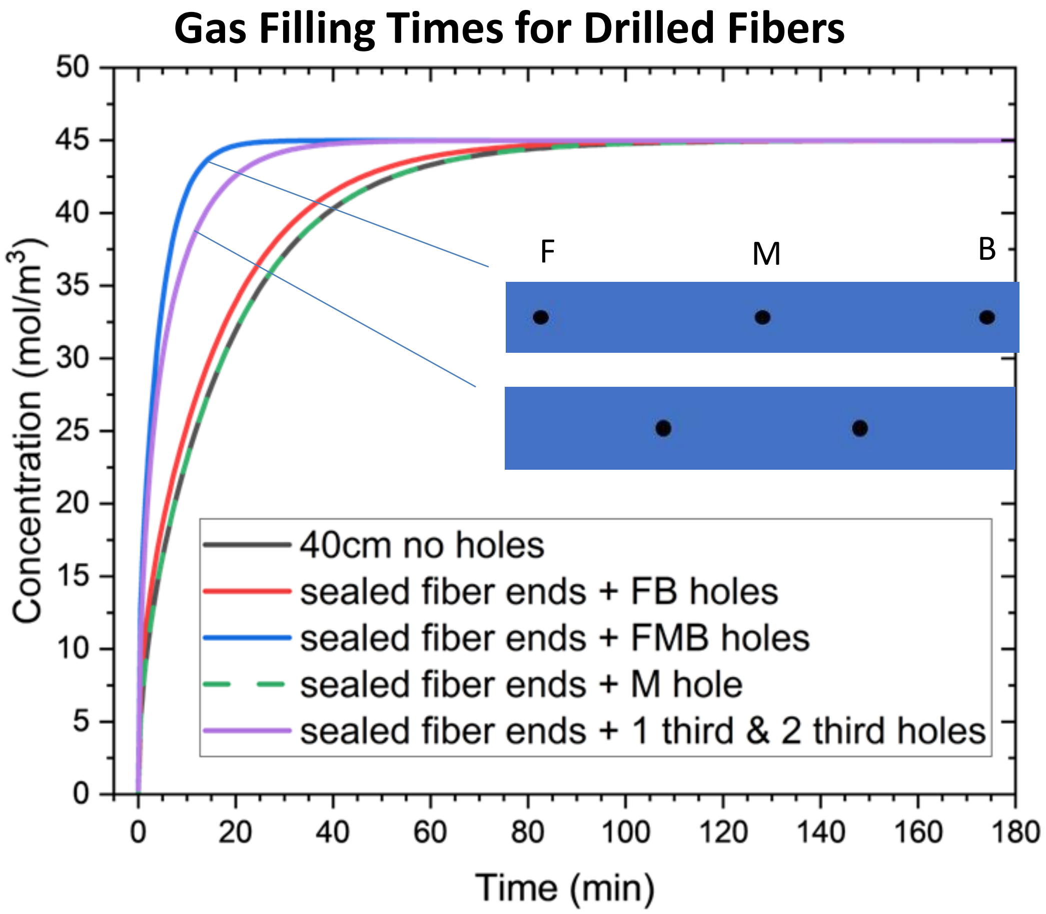


1392 peak



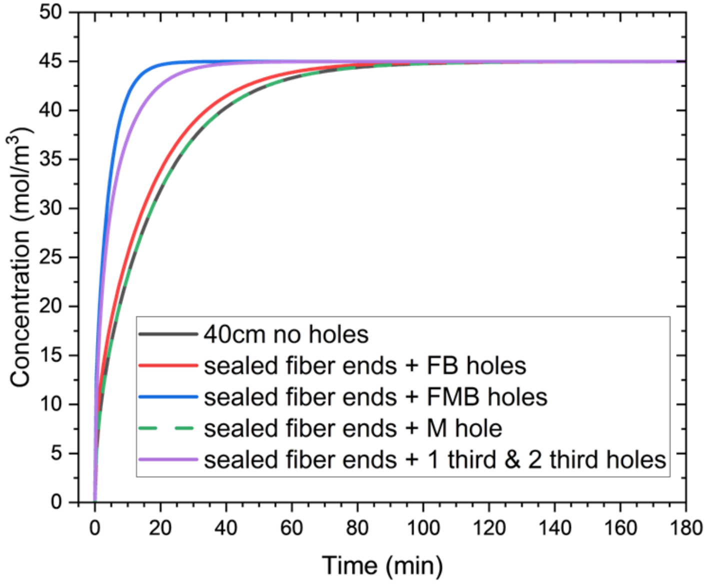
We have demonstrated <5000 ppm CO2 detection, now working on getting down to 100 ppm with higher power laser.

1.1a Gas infusion holes along HoF to speed up gas intake can be strategically positioned to optimize gas uptake with minimal number

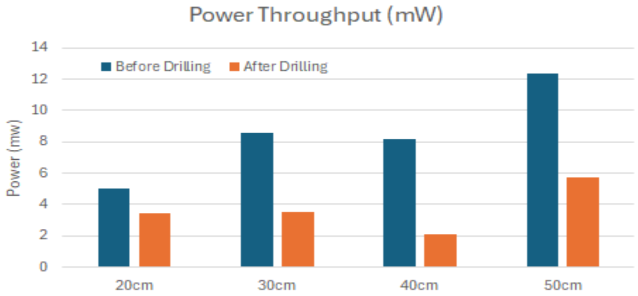


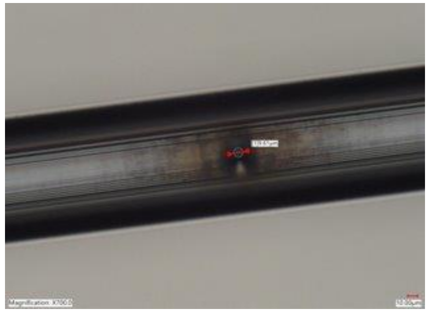
By optimizing infusion hole placement configuration along fiber, gas filling time can be reduced by an order of magnitude (e.g. from > 1 hr to mins in a 40cm fiber segment).

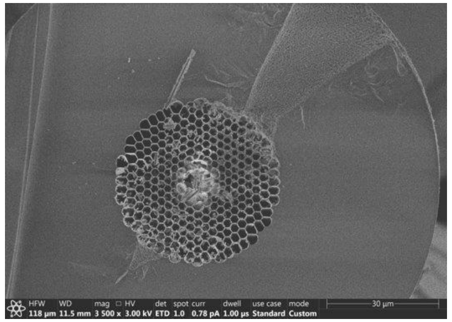
1.2a Laser drilling of the designed fibers was successfully performed by Precision MicroFab



Fiber	Before Drilling (mW)		After Drilling (mW)		Power Throughput (%)		
	Input Power	Through Power	Input Power	Through Power	Before Drilling	After Drilling	Throughput Ratio
20 cm	30.72	5.05	27.83	3.42	16.44	12.27	0.75
30 cm	30.72	8.59	27.83	3.5	27.95	12.56	0.45
40 cm	30.72	8.18	27.83	2.1	26.62	7.54	0.28
50 cm	30.72	12.34	27.83	5.73	40.15	20.57	0.51

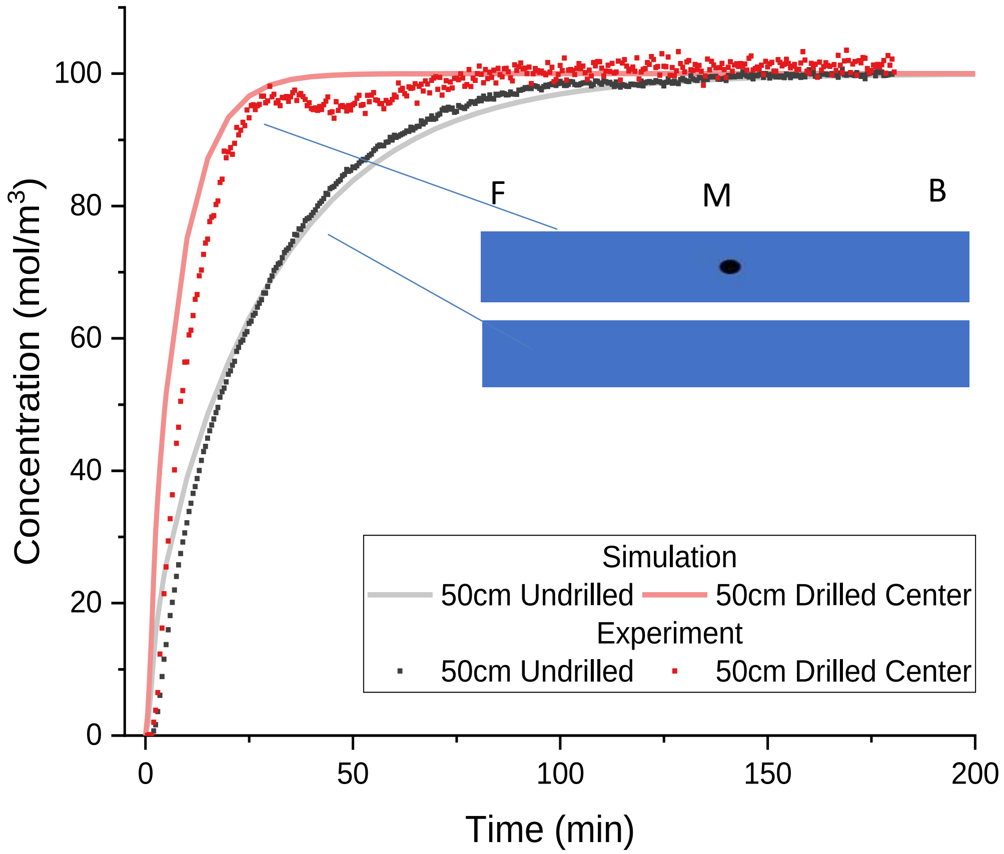






Optical power throughput measurements of show that throughput after fiber drilling can be as much as 77% of pre-drilled.

1.2a Drilled fibers: initial gas uptake kinetics data as designed and encouraging



Time to reach 95% CO_2 (min)		
Fiber	**Simulation**	**Experiment**
50cm	80	77
50cm drilled	25	31

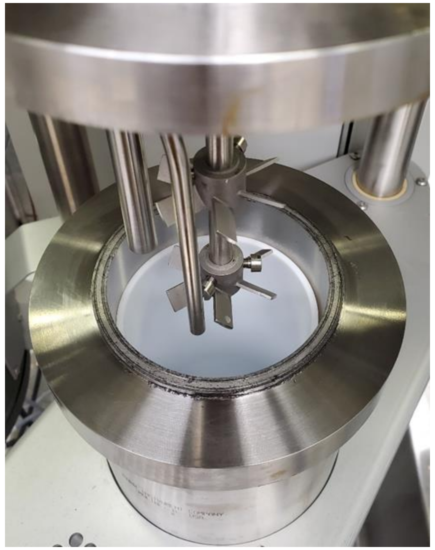
2.5x reduction in response time demonstrated

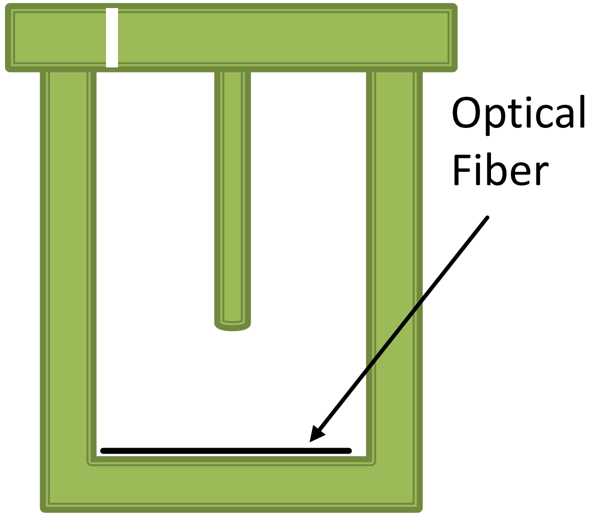
Drilled fibers were successfully tested to demonstrate a faster uptake as expected from simulation. We are currently performing measurements of latest batch of drilled configurations over 50cm and 1m HOFs.

1.2b Environmental Testing: first check survivability then optical behavior

Pressure (PSI)	Temperature	Environment
100	80°C	Dry Nitrogen
250	80°C	Dry Nitrogen
500	80°C	Dry Nitrogen
250	80°C	CO_2 pressurized 0.1 M NaCl
500	80°C	CO_2 pressurized 0.1 M NaCl

- After placing fiber in reactor, pressure was increased to desired PSI.
- Temperature was then increased to 80°C.
- All fibers were tested for 2.5 hours.

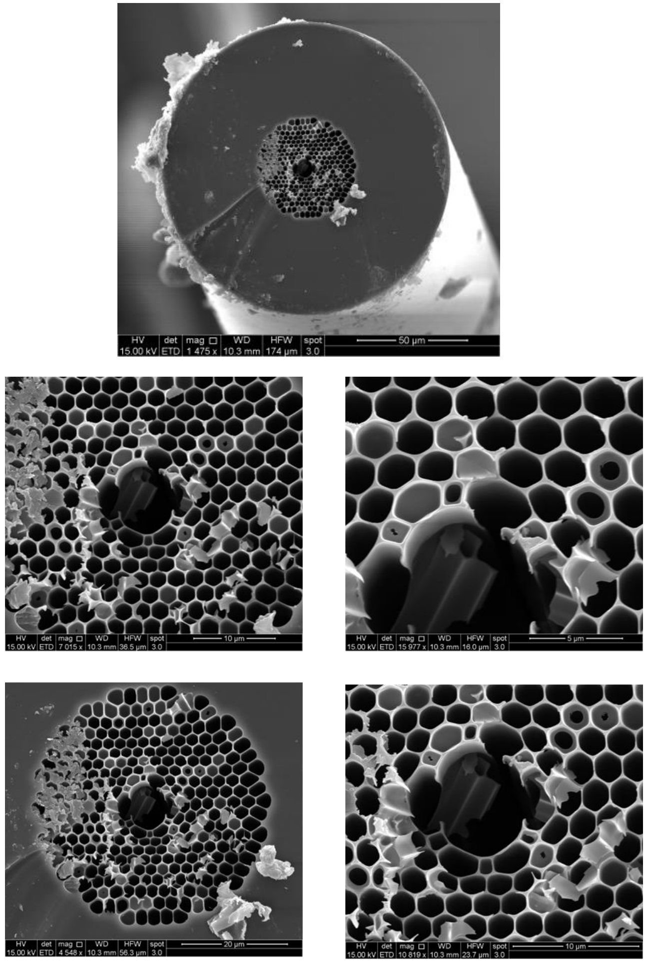




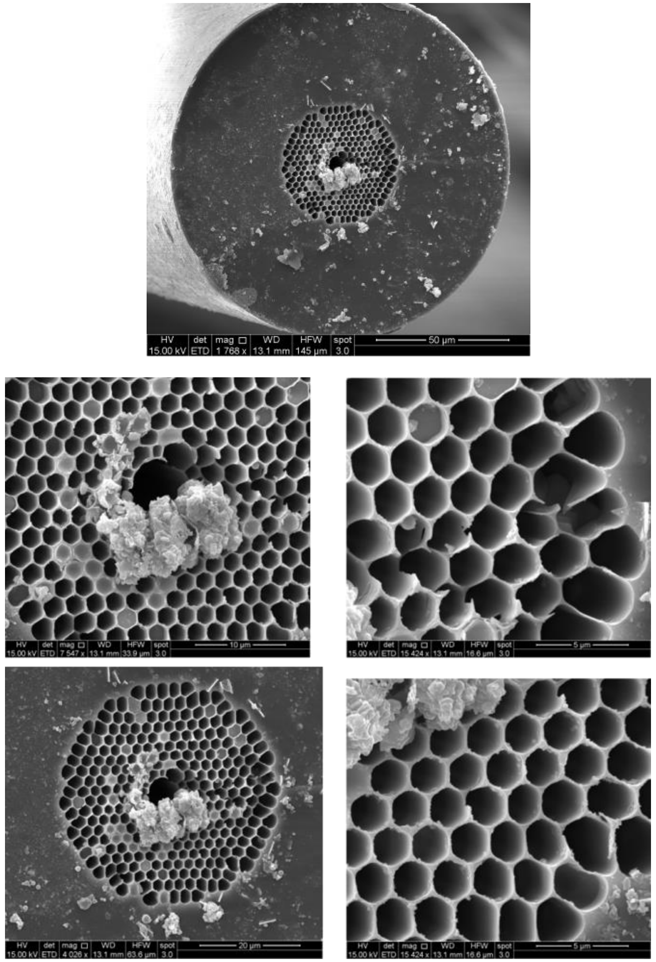
The mechanical testing on fiber resilience in harsh conditions was performed at NETL.

1.2b Harsh conditions tests results

500 psi N_2 Dry



500 psi CO_2 0.1M NaCl



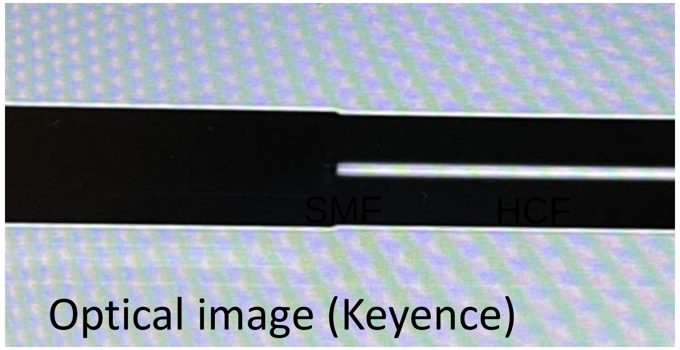
Hollow core fibers seem to withstand the harsh conditions in dry and wet conditions. We are working with NETL to measure the optical throughput once spliced to solid core fibers.

Subtask 2 – Integration of HoFs with Solid Core Fibers and FBGs

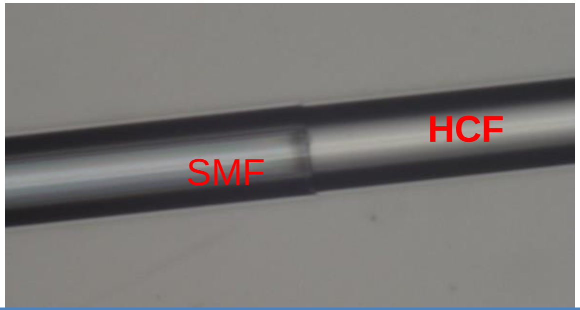
2.1 Splicing hollow core fiber to solid core fibers will be important for deployment

- Developed multi-step splicing recipe
- Successfully spliced photonics crystal hollow-core fibers (HC-800-02) and Thorlabs 780HP solid-core fiber using an Ericsson FSU 975 fusion splicer.

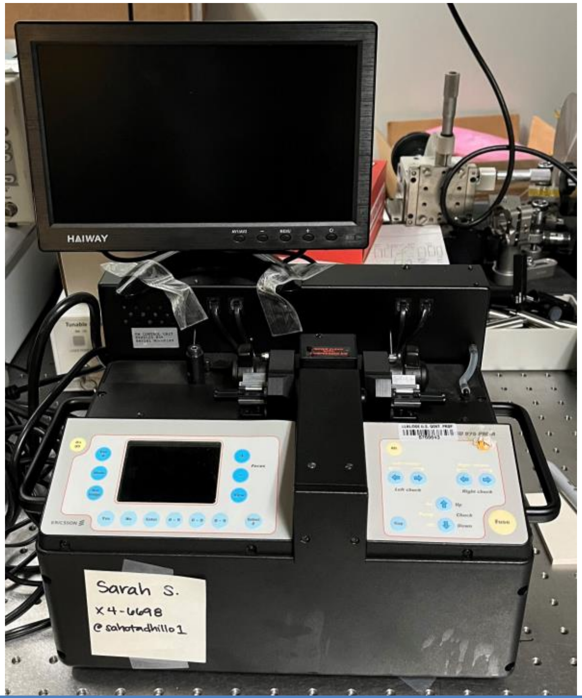
In-situ camera



Optical image (Keyence)



Splicer

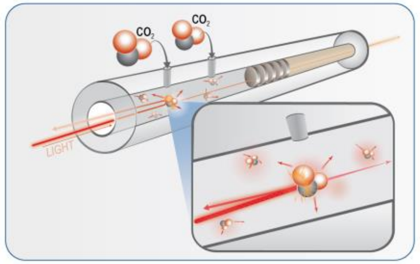


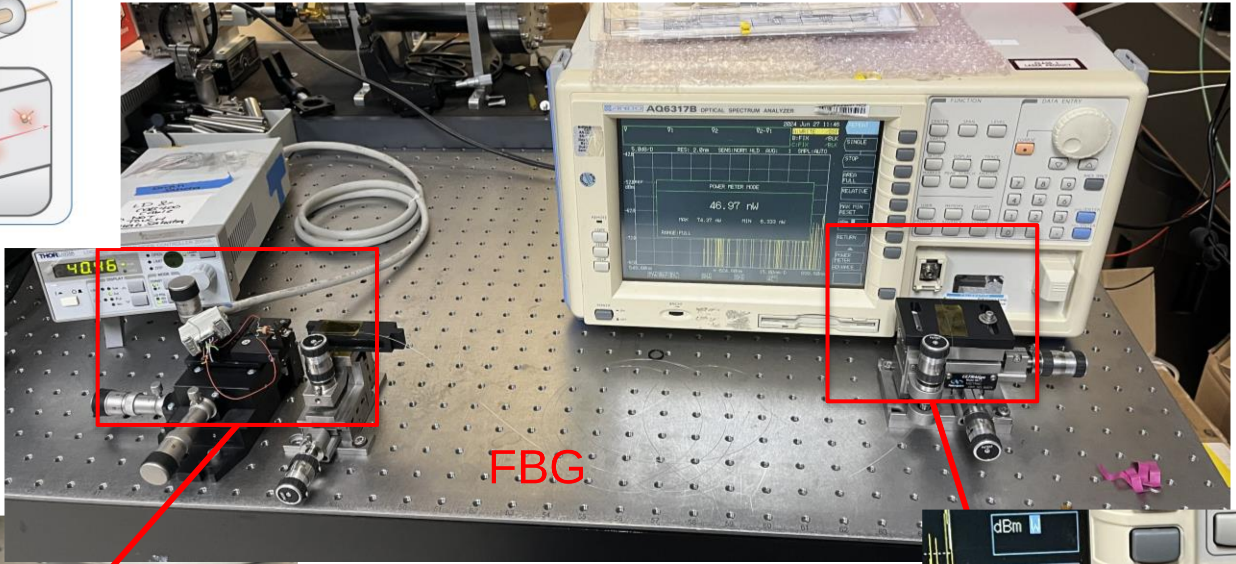
Splice Parameters		
Fiber Type	HC-1550-02	HC19-1550-01
Prefuse time	0.2 s	0.2 s
Prefuse current	10 mA	10 mA
Gap	10 µm	10 µm
Overlap	10 µm (9 µm)	15 µm
Fusion time 1	0.2 s	0.2 s
Fusion current 1	10 mA (9 mA)	10 mA
Fusion time 2	12 s (8 s)	12 s
Fusion current 2	7 mA (6.9 mA)	7 mA
Fusion time 3	3 s	3 s
Fusion current 3	6.5 mA (6.3 mA)	6.5 mA
Offset	260	260

The splicing process was repeated multiple times successfully and we are currently characterizing the optical throughput and deriving the statistical performance .

2.2 Fiber Bragg Gratings (FBGs) were designed to reflect back the CO_2 Raman Signal

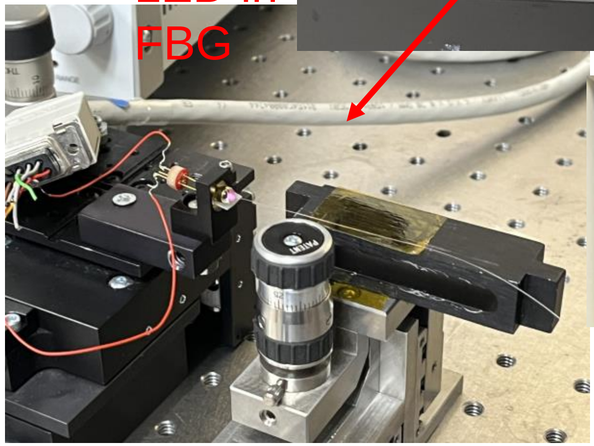
- FBG was fabricated by O/E Land Inc.

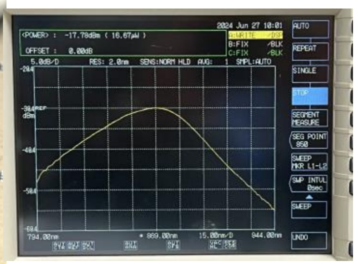




FBG

LED in FBG





FBG into OSA

Once we fully characterize the spectrum the FBG will be spliced to hollow core fiber to measure enhanced Raman.

Concluding remarks

We have demonstrated the fundamental capability of Hollow Core Fibers (HoFs) for CO_2 detection and laser drilling of gas infusion holes along fiber length

- We have been able to successfully demonstrate the uptake of CO_2 at various fiber lengths at various diffusion conditions, showing uptakes of ~1hr for HoFs, and ¼ reduction with S-HoFs. We are completing assessment of limit of detection and uptake time studies of HOF and S-HoFs in FY24.
- We have developed the capability of splicing of HOFs to solid core fibers for deployment ease and planning to implement it with FBG to provide back reflections to further enhance the Raman signals this year.

The proposed technology aim to detect, locate, and quantify migration of CO_2 and formation fluids within and above the storage complex main seal through surface deployment and/or within monitoring well, therefore:

- We have verified the survivability of the fibers in harsh environment and working on the optical characterization in FY25
- We are working with a couple of companies/organizations (Makel Engineering , PERTT/LSU, NETL) to identify test location and help with armoring designs (FY25)

3rd year: field demo of spectroscopic detection of CO_2 over 1Km stretch with HoFs coupled with solid core fibers and FBGs

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