Ford/BASF-SE/UM Activities in Support of the Hydrogen Storage Engineering Center of Excellence

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This presentation does not contain any proprietary, confidential, or otherwise restricted information

Overview

Timeline

- Project Start: February 2009
- Project End: June 2015

Budget

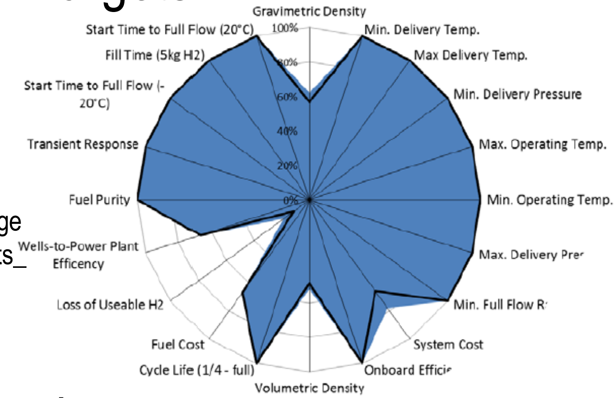
- Total Project Value: $2,783K
  - Cost Share: $643K
  - DOE Share: $2,140K
- DOE Funding Spent*: $2,045K

*as of 3/31/15

Barriers

- All DOE System Targets**
  - Volumetric Density
  - Gravimetric Density
  - System Cost

**http://www1.eere.energy.gov/hydrogenandfuelcells/storage/pdfs/targets_onboard_hydro_storage.pdf



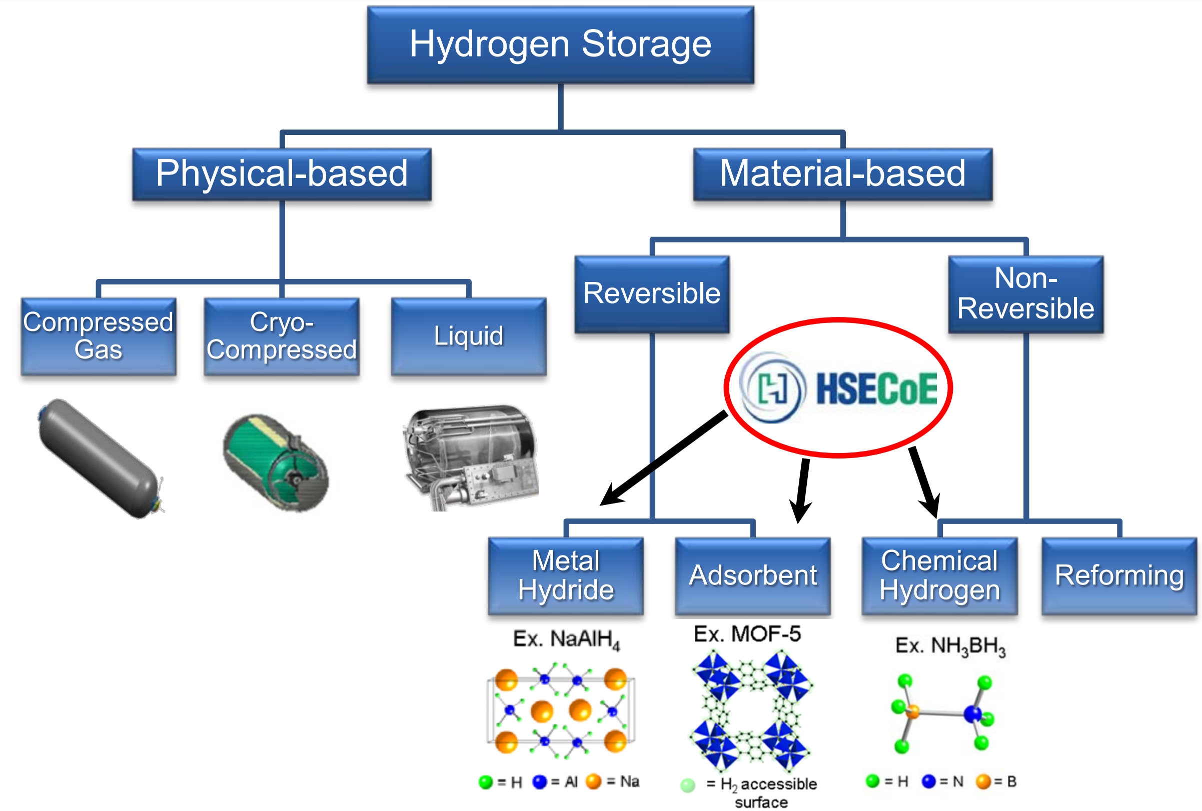
MATI Adsorbent System Example

Partners

- Project Lead: Ford
- Subcontractors: BASF and U. Michigan
- Center Partners:

HSECoE

Relevance: Technical



DOE Target	2020	Ultimate
System Gravimetric Density	5.5% (1.8 kWh/kg)	7.5% (2.5 kWh/kg)
System Volumetric Density	40 g/l (1.3 kWh/l)	70 g/l (2.3 kWh/l)
Storage System Cost	$333/kg ($10/kWh)	$266/kg ($8/kWh)

Material-based hydrogen storage systems have higher potential to meet the DOE targets but have increased complexity over physical-based storage options

Relevance: Technical

Three Technical Tasks Contribute to the Overall HSECoE Mission

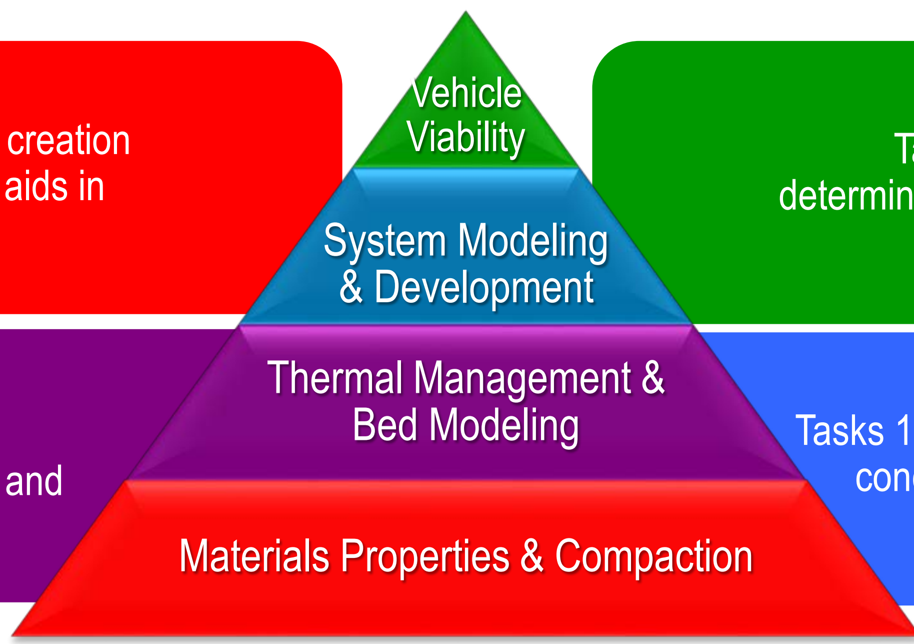
Task 1: Develop dynamic vehicle parameter model that interfaces with diverse storage system concepts (Ford)
Task 2: Development of robust cost projections for storage system concepts (Ford)
Task 3: Devise and develop system-focused strategies for processing and packing framework-based sorbent hydrogen storage media (Ford, BASF, University of Michigan)

Materials Properties

Task 3 data supports the creation of sorbent bed models & aids in tradeoffs analyses

Vehicle Viability

Tasks 1 & 2 models support determination of overall vehicle cost and performance



Bed Modeling

Task 3 data supports the validation of sorbent bed and system models

System Modeling

Tasks 1 & 2 models enable storage concepts to be exercised at the real-world vehicle level

Relevance: Organizational

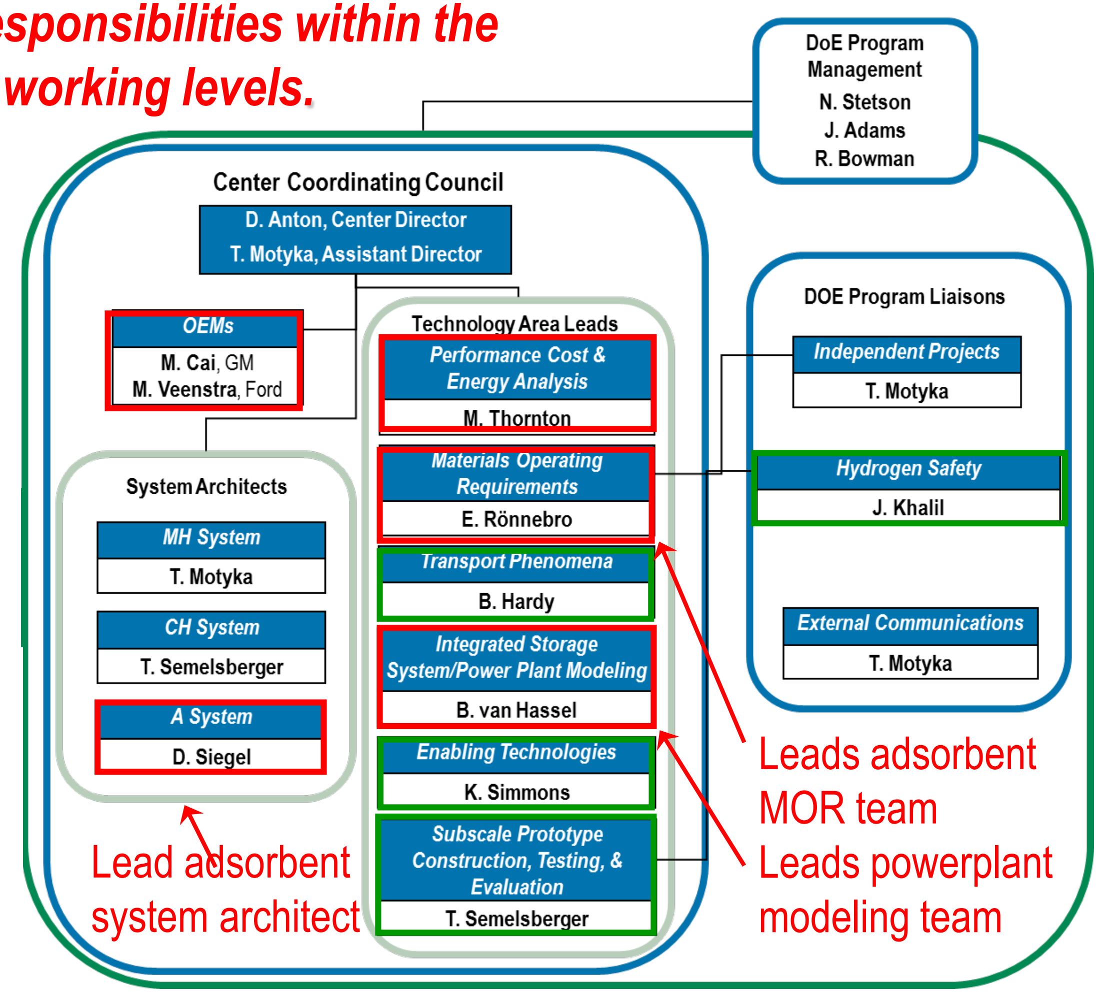
Ford project has many roles and responsibilities within the HSECoE at both the executive and working levels.

OEM Perspective Role (M. Veenstra)

- Provided an automotive perspective
- Acted as liaison to USDRIVE tech teams
- Integrated fuel cell model for HSECoE framework
- Engaged in system trade-offs and cost analysis
- Coordinated FMEA and design verification plan

System Architect Role (D. Siegel)

- Coordinated adsorbent team design activities
- Communicate technical status and gaps
- Developed SMART milestones and GANTT chart
- Organized regular meetings with adsorbent team
- Consolidated progression towards system targets



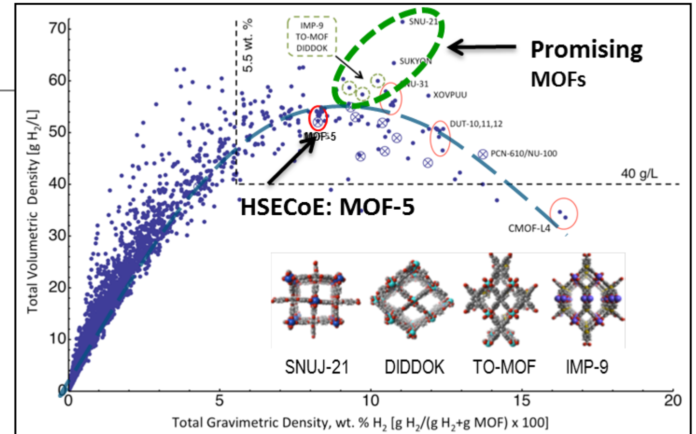
Red = Core contribution areas Green= Ancillary contribution areas of HSECoE

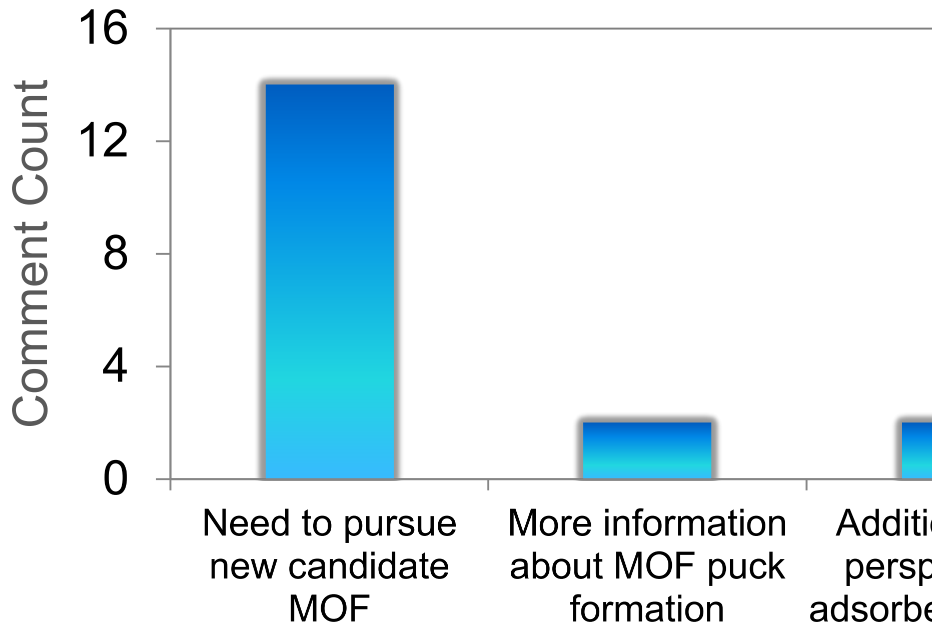
Ford/BASF/UM team provides many important roles in the overall HSECoE

Approach: AMR Comments and Responses

Reviewer Comments from 2014 AMR:

see 2014 AMR





- Need to pursue new candidate MOF → New DOE award (UM) for MOFs
- More information about MOF puck formation → Included later in this presentation
- Additional OEM perspective on adsorbent systems → Summarized on next slide
- Assess problem related to the loss of usable H2 → Out of scope for this project

Many positive comments received with a few general recommendations

OEM perspective on adsorbent systems

System Comparison for Phase 3 (useable 5.6 kg)

Internal HX and Media:	HexCell + powder MOF-5	MATI + 0.32 g/cc MOF-5 pucks	700 bar* Compressed H_2
System Mass (kg)	161	159	128
System Volume (L)	304	263	224
Estimate System Cost at 500K units	$2,720**	$2,897**	$3,134
Gravimetric Capacity ($g\text{-}H_2$/g-system)	3.5 %	3.5%	4.4 %
Volumetric Capacity ($g\text{-}H_2$/L-system)	18.5 g/l	21.3 g/l	25.0 g/l

- Full tank: P = 100 bar, T = 80 K
- Empty tank: P = ~5 bar, T = ~140 K
- Single, Aluminum (6061-T6) Type 1
- LN_2 vessel wall chilling channels

**PNNL PI for cost analysis

*2013 AMR references
ANL Project ID: ST001
SA Project ID: ST100

ADVANTANGES

- ✓ 7x lower pressure
- ✓ Lower tank cost
- ✓ Lower infrastructure capital cost
- ✓ Opportunity for fuel cell thermal sink

CHALLENGES

- ❖ Volumetric capacity
- ❖ Cryogenic certification
- ❖ Insulation robustness
- ❖ Liquid hydrogen pathway penalties

Adsorbent systems offer advantages using low pressure with challenges

Approach: Phase 3 SMART Milestones and Tasks

Component	Partner	Proposed SMART Milestones for Phase 3	Due Date
Adsorbent Media ✓ Complete- see 2014 AMR	Ford/UM/BASF	**Conduct a scale-up of the MOF-5 manufacturing process** to deliver **> 9 kg of material** while maintaining performance, as measured by surface area and particle size, to within **10% of lab-scale procedure**.	12/31/2013
Adsorbent Media	Ford/UM/BASF	**Evaluate MOF-5 degradation** beyond 300 cycles based on maximum allowable **impurity levels as stated in SAE J2719** and report on the ability to **mitigate to less than 10%.**	9/30/2014
Adsorbent Media	Ford/UM/BASF	**Complete the failure mode and effects analysis (FMEA)** associated with real-world operating conditions for a MOF-5-based system, for both HexCell and MATI concepts **based on the Phase 3 test results**. Report on the ability to **reduce the risk priority numbers (RPN)** from the phase 2 peak/mean and identify key failure modes.	6/30/2015
System Modeling	NREL/SRNL/ PNNL/Ford/ UTRC	**Update the cryo-adsorbent system model** with Phase 3 performance data, integrate into the framework; document and **release models to the public.**	6/30/2015
Additional task:		Explore approaches to **maximize the MOF-5 "real-world" material properties**: advance thermal conductivity and compaction effects.	

Project approach based on collaborative HSECoE SMART milestones

Progress: MOF-5 Robustness to H_2 Impurity

Milestone Task: Evaluate MOF-5 degradation beyond 300 cycles based on maximum allowable impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.

Impurity Test Gas

SAE J2719 Constituent	Chemical Formula	Limits	Test Gas Levels	Mixture Combinations
Hydrogen fuel index	H_2	> 99.97%		
Total allowable non-hydrogen, non-helium, non-particulate constituents listed below		100		
Acceptable limit of each individual constituent				
Water	H_20	**5 ppm**	**5 to 10 ppm**	*Test Gas Mixture 1*
Total hydrocarbons	C_1	**2 ppm**	**2 ppm**	*Test Gas Mixture 2*
Oxygen	O_2	**5 ppm**	**5 ppm**	*Test Gas Mixture 2*
Helium	He	**300 ppm**	**500 ppm**	*Test Gas Mixture 2*
Nitrogen, Argon	N_2, Ar	**100 ppm**	**100 ppm**	*Test Gas Mixture 2*
Carbon dioxide	CO_2	**2 ppm**	**5 ppm**	*Test Gas Mixture 2*
Carbon monoxide	CO	**0.2 ppm**	**2 ppm**	*Test Gas Mixture 2*
Total sulfur	S	**0.004 ppm**	**1 ppm**	*Test Gas Mixture 3*
Formaldehyde	HCHO	**0.01 ppm**	**n/a**	*Not in Gas Mixture*
Formic acid	HCOOH	**0.2 ppm**	**n/a**	*Not in Gas Mixture*
Ammonia	NH_3	**0.1 ppm**	**5 to 10 ppm**	*Test Gas Mixture 4*
Total halogenates		**0.05 ppm**	**5 to 10 ppm**	*Test Gas Mixture 5*
Particulate Concentration		1 mg/kg		

See SAE J2719 for original reference

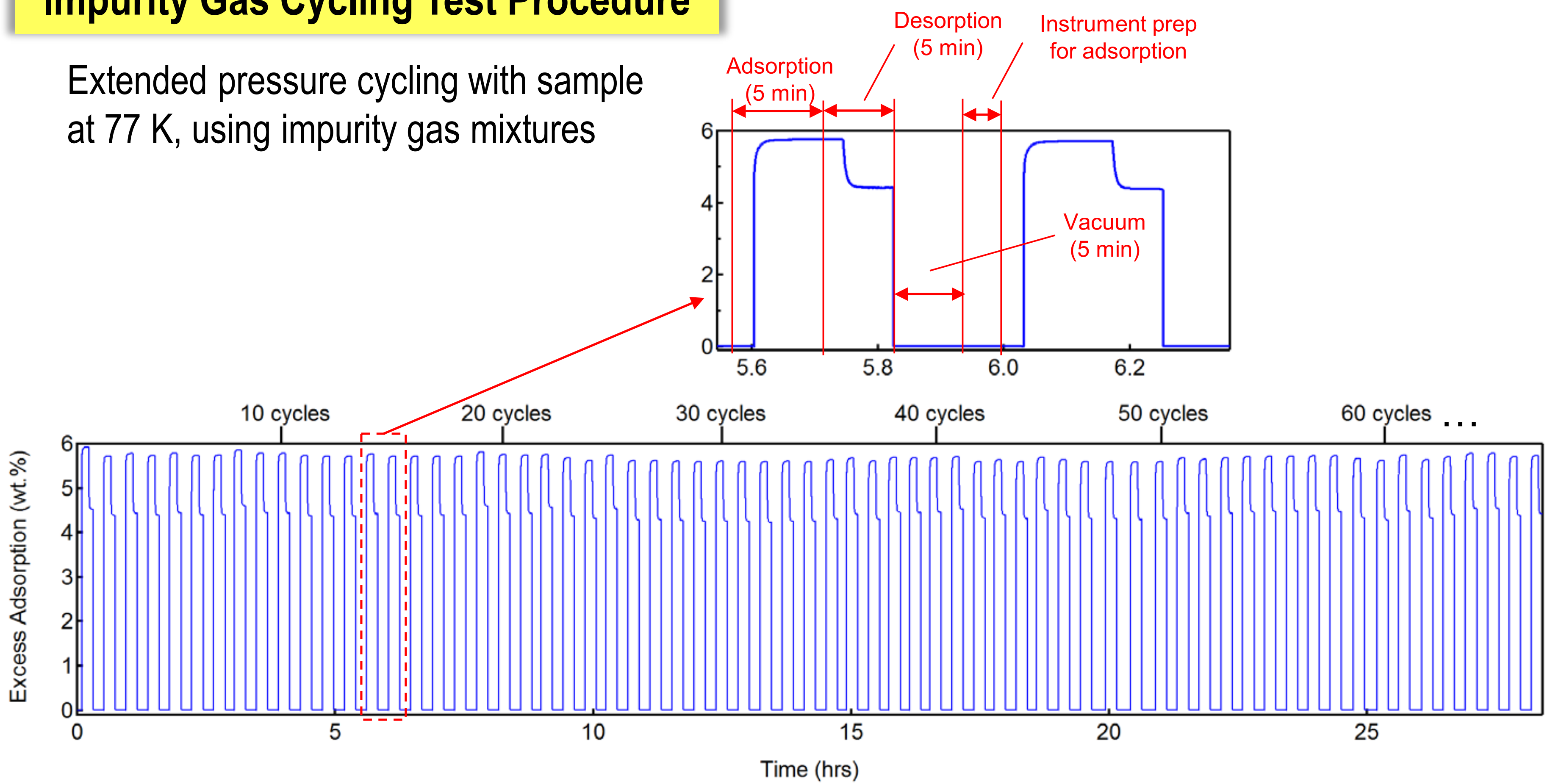
Impurities were tested in separate mixtures to evaluate the impact

Progress: MOF-5 Robustness to H_2 Impurity

Milestone Task: Evaluate MOF-5 degradation beyond 300 cycles based on maximum allowable impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.

Impurity Gas Cycling Test Procedure

Extended pressure cycling with sample at 77 K, using impurity gas mixtures

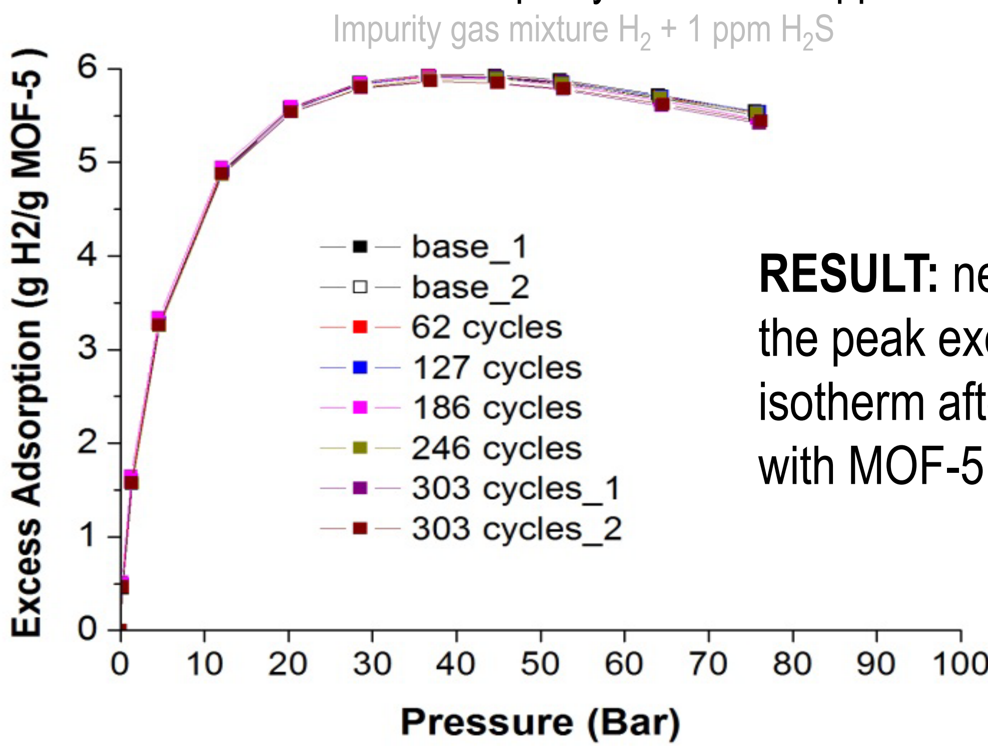


Progress: MOF-5 Robustness to H_2 Impurity

Milestone Task: Evaluate MOF-5 degradation beyond 300 cycles based on maximum allowable impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.

Impurity Test Gas Mixture: 1 ppm Sulfur

NOTE: J2719 impurity level: 0.004 ppm



RESULT: negligible change in the peak excess adsorption isotherm after 303 cycles (77 K) with MOF-5 powder

Extreme sulfur impurity cycling only resulted in negligible change

Progress: MOF-5 Robustness to H_2 Impurity

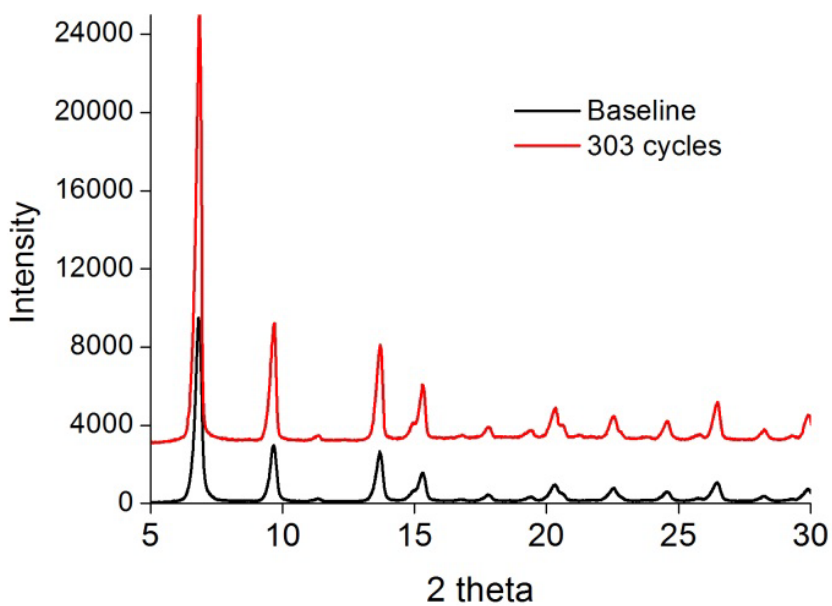
Milestone Task: Evaluate MOF-5 degradation beyond 300 cycles based on maximum allowable impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.

Impurity Test Gas Mixture: 1 ppm Sulfur

NOTE: J2719 impurity level: 0.004 ppm

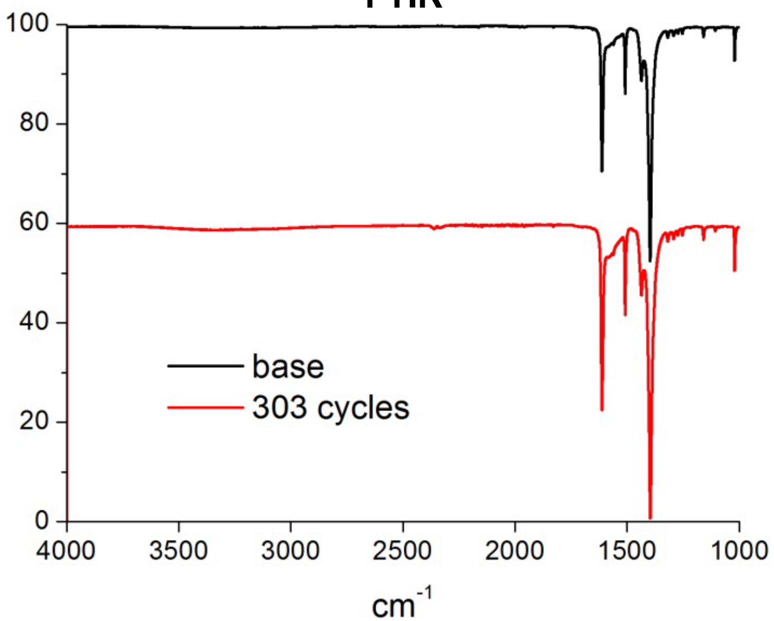
Impurity gas mixture H_2 + 1 ppm H_2S

XRD



RESULT: No damage to the crystal structure

FTIR

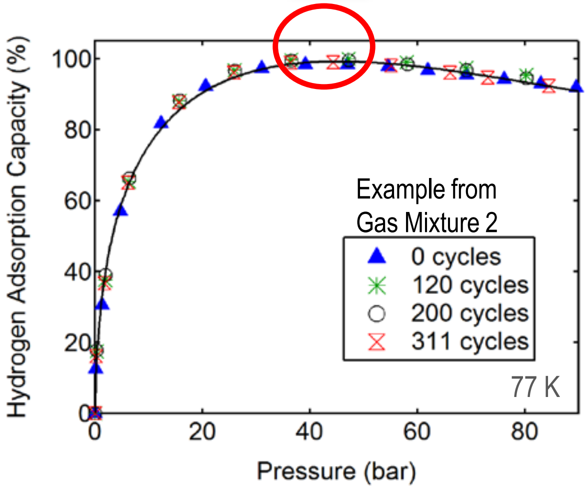


RESULT: No change from the sulfur exposure

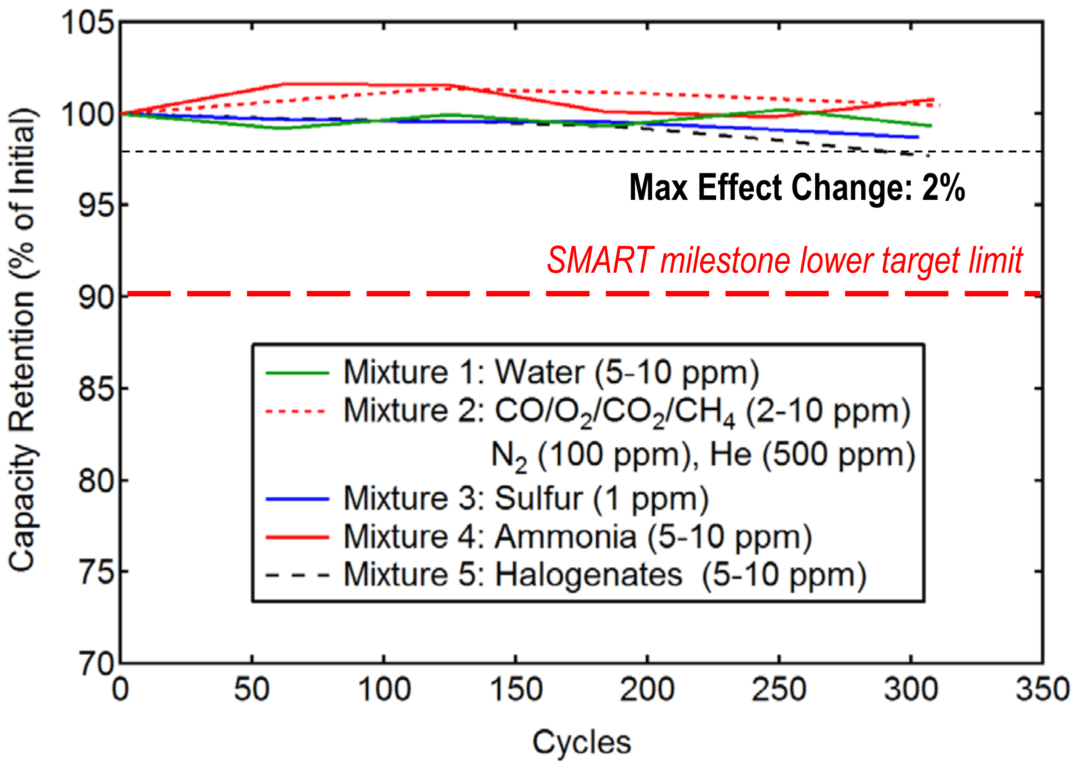
Impurity cycle testing results were confirmed with XRD and FTIR

Progress: MOF-5 Robustness to H_2 Impurity

Milestone Task: Evaluate MOF-5 degradation beyond 300 cycles based on maximum allowable impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.



Peak Adsorption Retention Versus Cycles



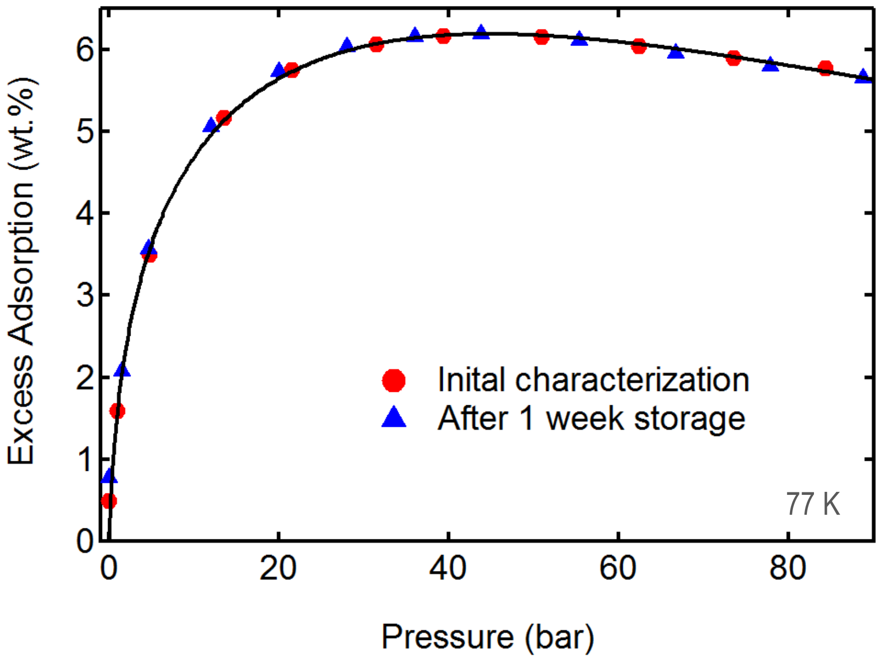
MOF-5 robust to J2719 impurity levels cycled at low temperature (77 K)

Progress: MOF-5 Robustness to H_2 Impurity

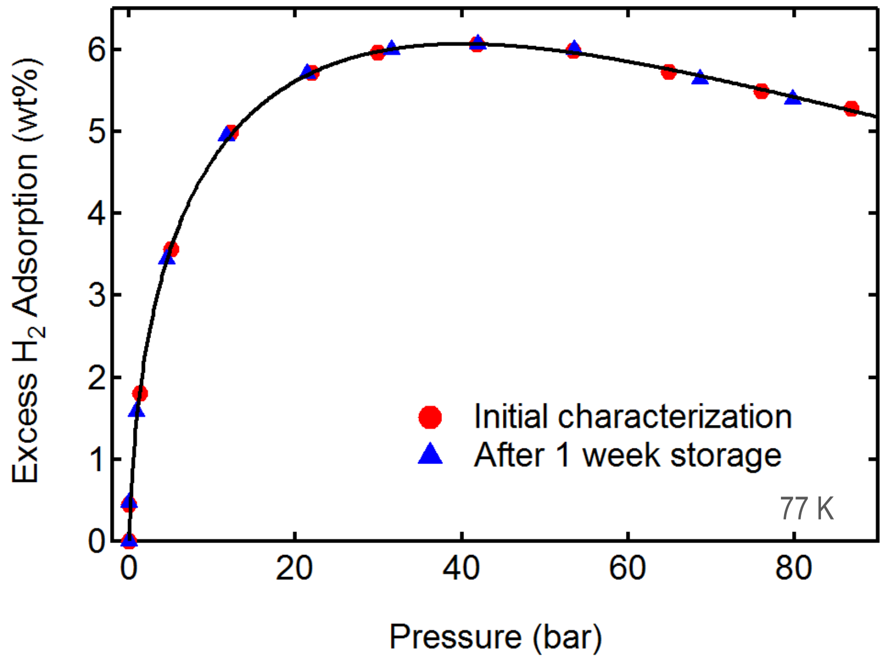
Additional evaluation of potential MOF-5 degradation based on long term static exposure to impurity levels as stated in SAE J2719.

Long Term Impurity Gas Exposure at 25°C

Storage : H_2 + 1 ppm H_2S



Storage: H_2 + 9 ppm H_2O (at 82 bar)

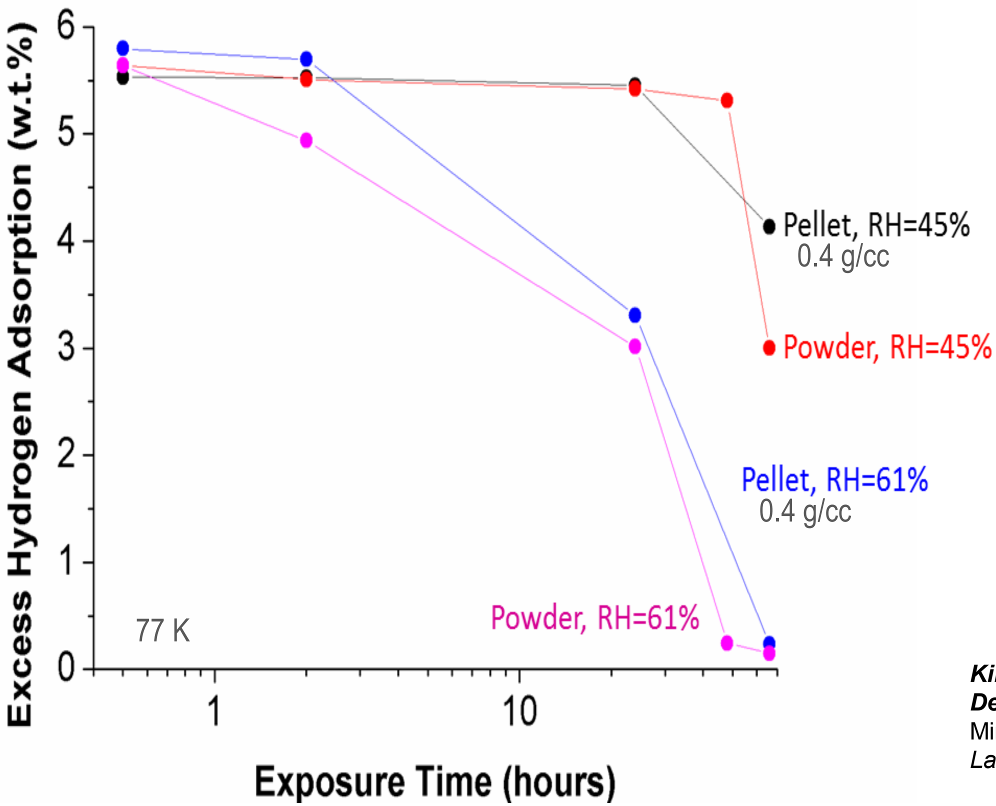


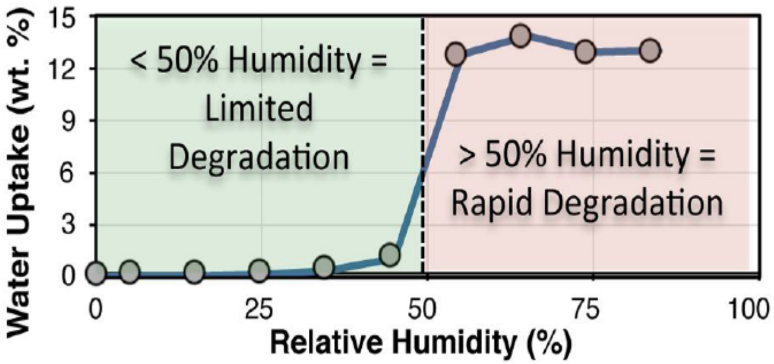
MOF-5 exposed to impurity gas mixtures for 1 week had no degradation

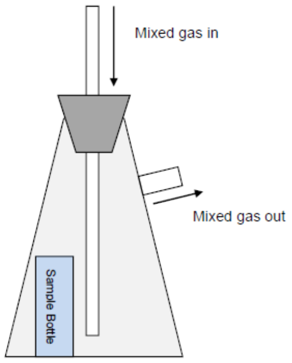
Progress: MOF-5 Robustness to Humidity

Additional evaluation of MOF-5 based on static exposure to humidity levels and time.

Peak Adsorption versus Humidity Exposure







Kinetic Stability of MOF-5 in Humid Environments: Impact of Powder Densification, Humidity Level, and Exposure Time
Ming, Purewal, Yang, Xu, Soltis, Warner, Veenstra, Gaab, Müller, and Siegel
Langmuir, **2015**, *31* (17), pp 4988–4995, **DOI:** 10.1021/acs.langmuir.5b00833

MOF-5 is stable under moderate humid conditions and exposure time

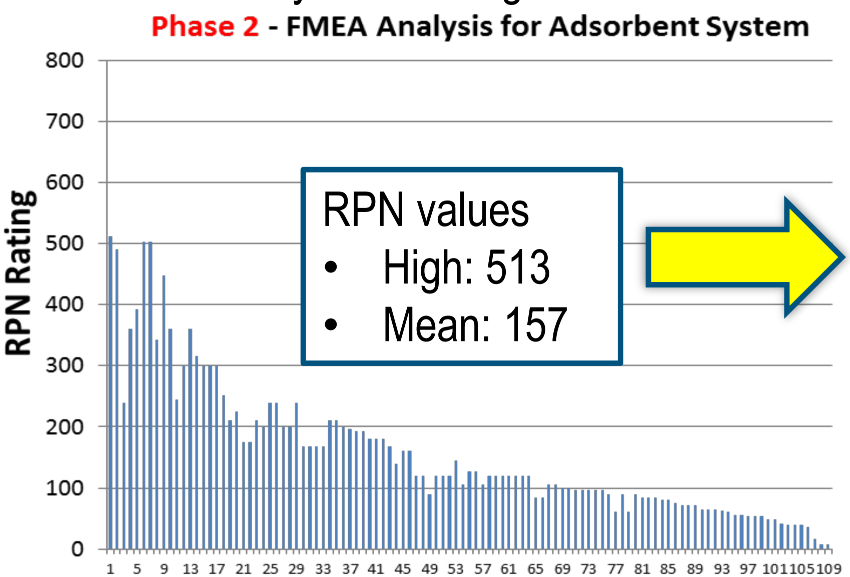
Progress: FMEA - Failure Mode Reduction

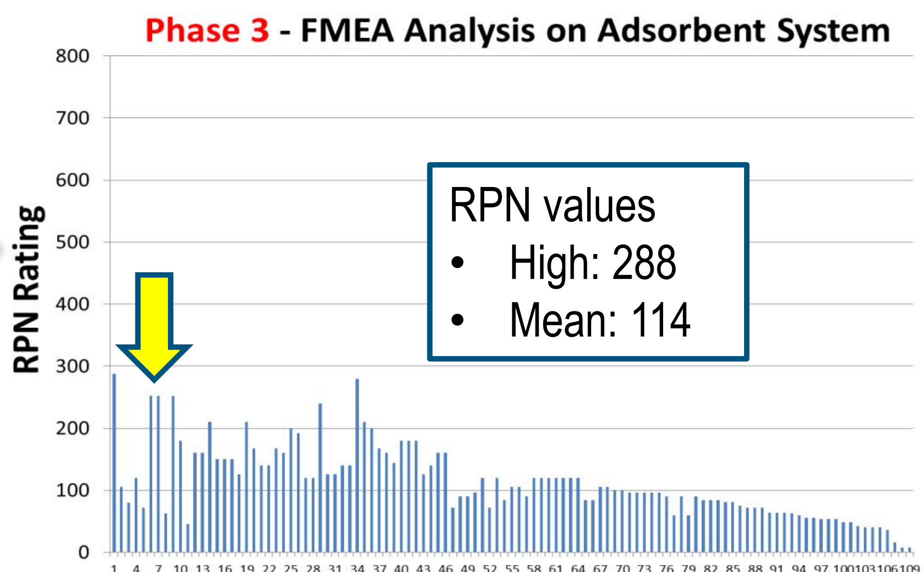
FMEA = Failure Mode and Effects Analysis (industry tool per SAE J1739)

- Identifies and evaluates the potential failure of a product and its effects
- Documents the risk and helps prioritize the key actions to reduce failures

Example actions during phase 3 for reducing the Risk Priority Number (RPN)

1. Completed testing to reduce occurrence ratings associated with hydrogen impurity concerns
2. Assessed tank robustness with adsorbent material and cryogenic operating conditions
3. Conducted thermal management evaluation testing to assess performance in adsorbent bed
4. Performed system testing to assess material variability and effects of non-homogenous bed



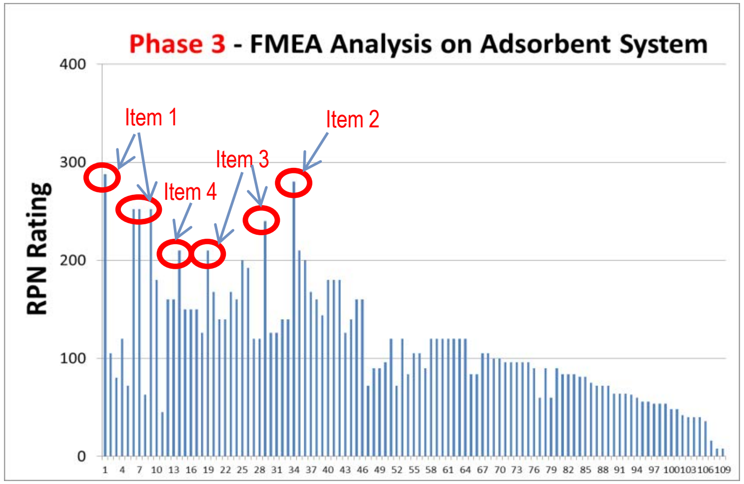


Phase 3 FMEA updates identified reductions in potential failure modes

Progress: FMEA - Failure Mode Reduction

Top RPN Items based on the Phase 3 FMEA assessment (200):

Potential Cause	Failure Mode (RPN)	Comments
(1) Material release rate insufficient **due to non-homogenous materials** at end of life	Hydrogen supply unable to achieve full flow rate (288) Storage system only accepts partial fill (252) Storage system on-board efficiency < 90% (252) System only supplies partial capacity (252)	Confirmed performance was stable after material cycle and Phase 3 system testing. Further key life tests should be considered.
(2) Component interfaces and connections between BOP parts **leak at cold temperatures**	System exceeds allowable external leak rate limit. (280)	Further development of cryogenic sealing solutions should be considered.
(3) Material uptake insufficient rate due to **performance of thermal isolation system** such as vacuum stability	Loss of useable hydrogen is greater than .05 [g/hr]/kg H2 (240) System only supplies partial capacity (assumes complete fill) - from initial use through lifetime (1,500 cycles) (210)	Further reliability assessment of vacuum system integrity over the lifetime should be considered.
(4) Material release rate insufficient due **degradation in heat transfer in bed** and to the thermal management system at end of life	System only supplies partial capacity (assumes complete fill) - from initial use through lifetime (1,500 cycles) (210)	Confirmed performance of thermal management during Phase 3 system testing. Further key life tests should be considered.

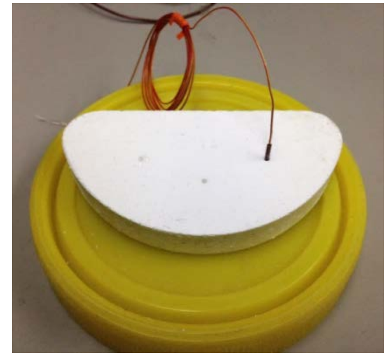


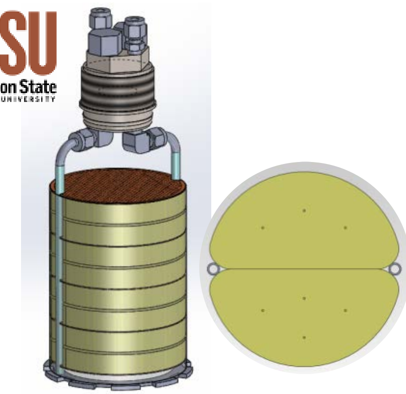
FMEA process supports the key outcome for the HSECoE:

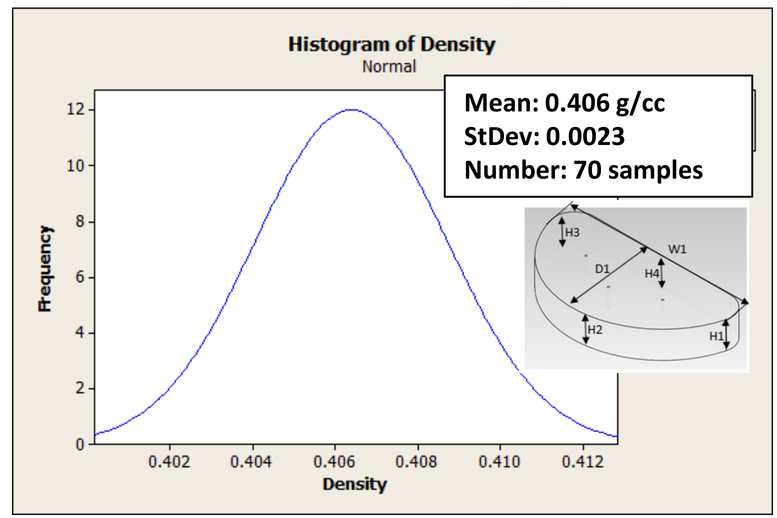
Develop and provide material-based system designs and gap analysis for consideration in further research efforts.

Progress: Maximize MOF-5 Material Properties

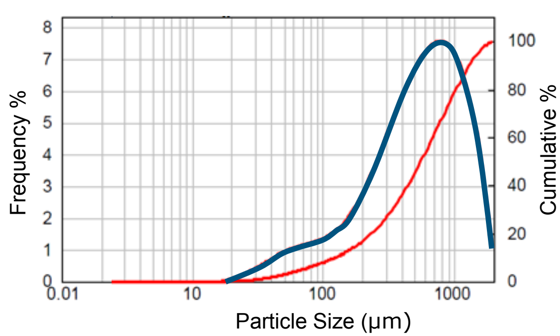
Explore approaches to maximize material properties by advancement in compaction.







MOF-5 particle distribution facilitates positive compaction

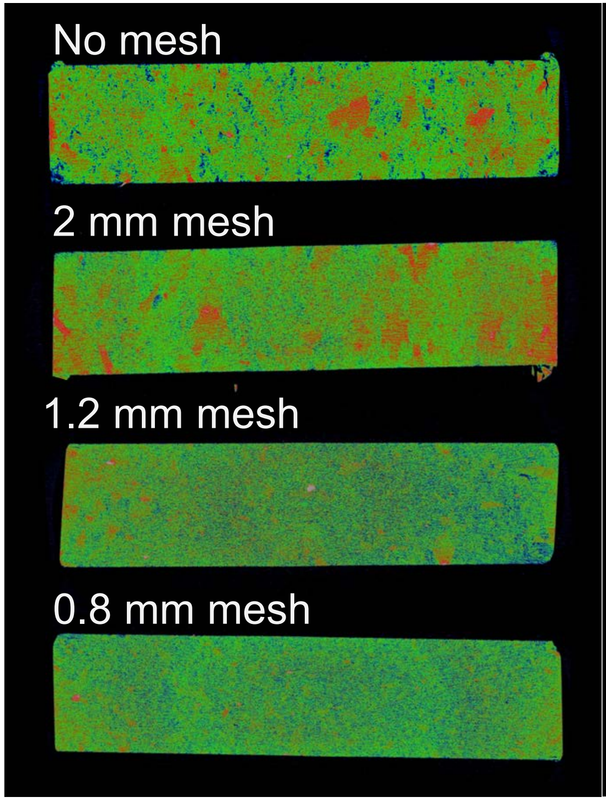


MOF-5 compaction was highly repeatability and formable without binders

Progress: Maximize MOF-5 Material Properties

Explore approaches to maximize material properties by advancement in compaction.

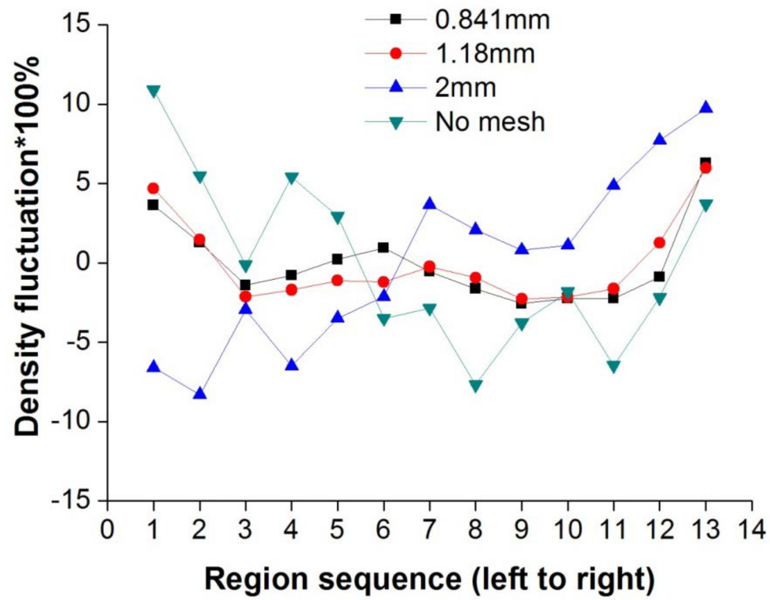
average density of 0.41g/cc
(density: red > green > blue)



Sample	StDev (g/cc)
No mesh	0.0271
2 mm mesh	0.0246
1.2 mm mesh	0.0114
0.8 mm mesh	0.0107

Divided the pellet cross section into 13 small regions in CT scan to calculate the average density in the region

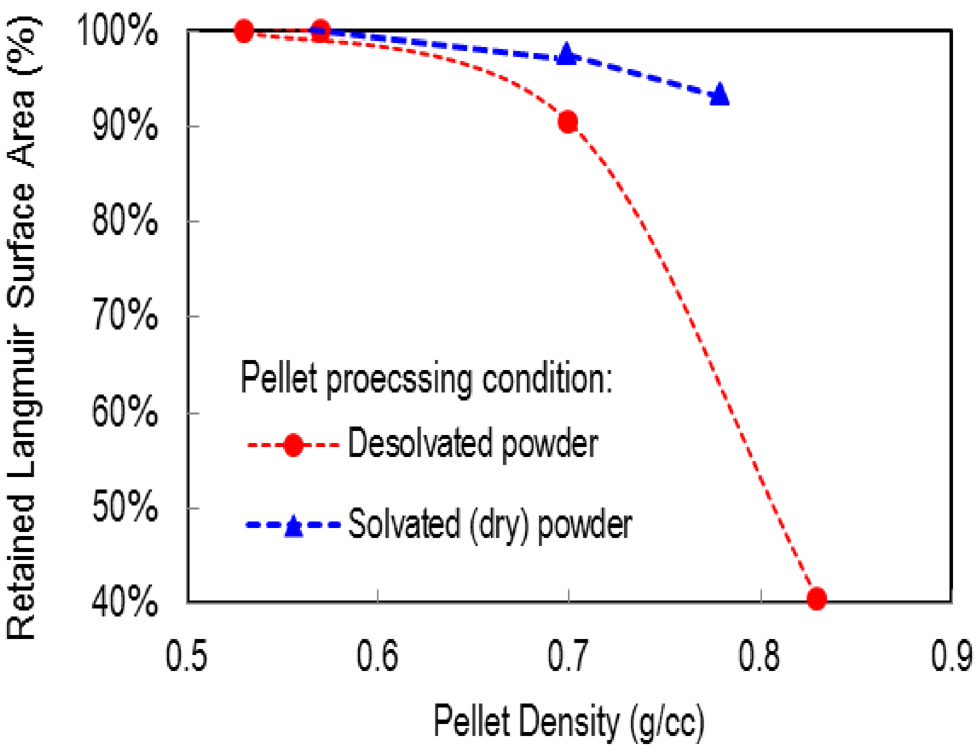
Density fluctuation:
[ρ(region)-ρ(pellet_avg)]/ρ(pellet_avg)x100%



Compaction density variation improves significantly with powder sieve

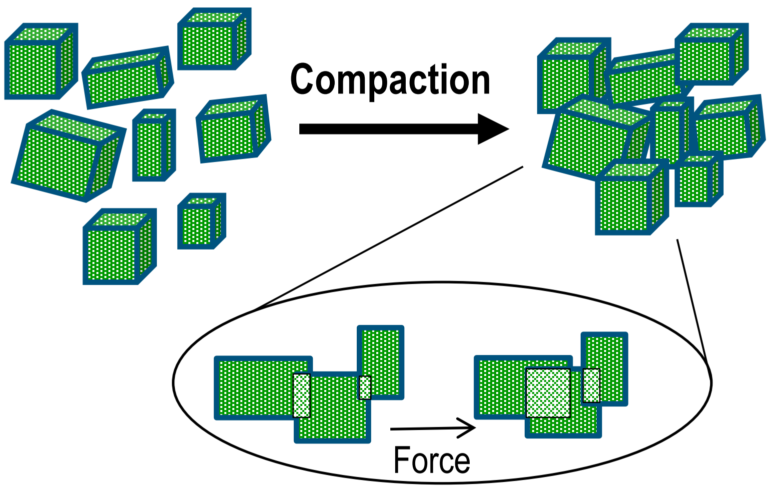
Progress: Maximize MOF-5 Material Properties

Explore approaches to maximize material properties by advancement in compaction.



Evaluated with Ni-MOF-74
results with MOF-5 on-going

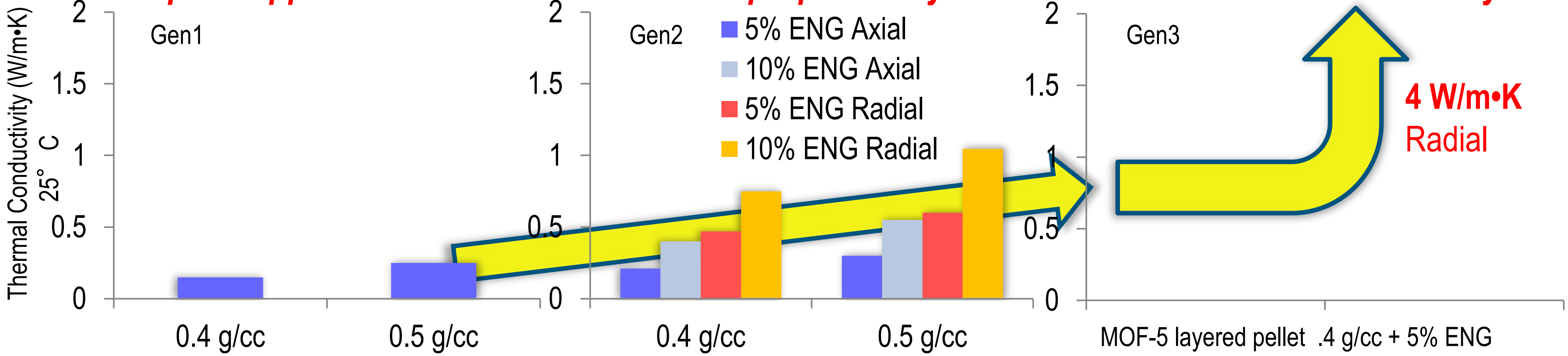
Compaction prior to solvate removal and activation provides supports to crystal structure for potential improvement in surface area retention.

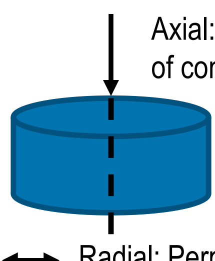


Additional compaction techniques can offer further advantages

Progress: Maximize MOF-5 Material Properties

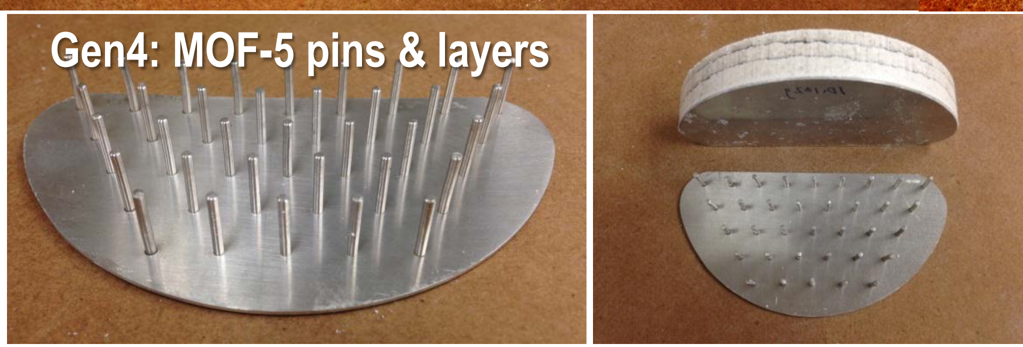
Explore approaches to maximize material properties by advancement in thermal conductivity.





Axial: Direction of compression

Radial: Perpendicular to compression

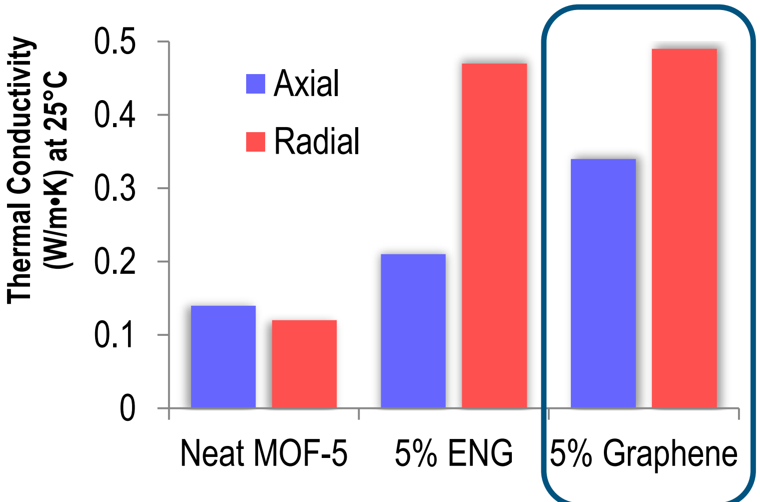


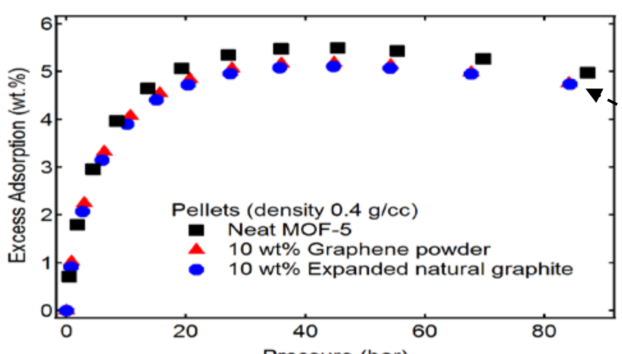
Gen4 concept has been formed for testing which combines aluminum pins and layer ENG to advance thermal conductivity while reducing the anisotropic variation

Progress: Maximize MOF-5 Material Properties

Explore approaches to maximize material properties by advancement in thermal conductivity.

Improved thermal conductivity



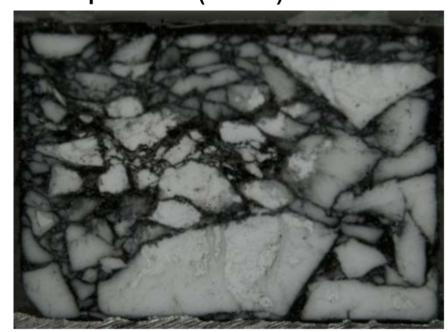


Reduced anisotropy

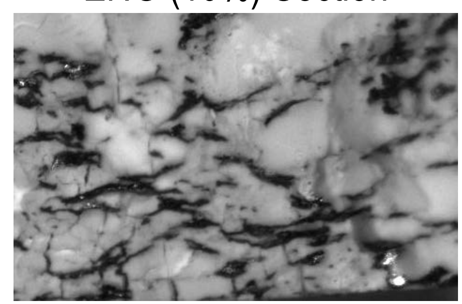
with equivalent hydrogen adsorption performance

Graphene powder completely coats all MOF5 particles (light), creating a continuous carbon matrix (dark) in both radial and axial directions (reduced anisotropy).

Graphene (10%) Section



ENG (10%) Section



Additional thermal conduction additives can offer further advantages

Summary: Phase 3 SMART Milestones and Tasks

SMART Milestone Tasks	Status
Conduct a scale-up of the MOF-5 manufacturing process > 9 kg	✓ Delivered 9.3 kg of MOF-5 for Phase 3 to HSECoE partners within 10% of lab-scale synthesis material
Evaluate MOF-5 degradation cycles using impurity levels as stated in SAE J2719	✓ Completed impurity gas cycle testing that indicate low or no degradation from the exposure.
Complete the failure mode and effects analysis (FMEA) based on the Phase 3	✓ FMEA action items have been updated and reduced the risk priority numbers (RPN) from phase 2 values
Support system model release and validation with Phase 3 performance results	✓ Supported the Simulink® framework release and model testing through development in the modeling group

Additional Tasks	Status
Consider additional potential improvement for MOF-5 and/or other adsorbents	✓ Provided a further OEM outlook for the possibility of an on-board adsorbent system
Improved densification	✓ Demonstrated improvement in puck density variation using filtering techniques and performance.
Enhance thermal conductivity	✓ Developed alternative materials and approaches to increase thermal conductivity beyond ENG mixtures

Future Work: Successful Completion

- ❑ Ensure a successful completion to the HSECoE project including finalizing material research, system cost studies, modeling validation, and FMEA.
- ❑ Expected documentation to publish prior to completion:
  - ➢ MOF-5 robustness J2719 impurity cycle testing techniques and results
  - ➢ Molecular scale water insertion mechanisms in MOF-5
  - ➢ Neutron and X-ray imaging studies of MOF-5 kinetics and tomography
  - ➢ HSECoE adsorbent system final report and material targets

Progress: Significant Project Publications

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Collaborations: HSECoE Partners

- SRNL (federal lab collaborator): team lead for sorbent (bed) transport phenomena, adsorbent system modeling, and center management
- Universite du Quebec a Trois-Rivieres (university collaborator): adsorption system test bench and MOF-5 isotherm validation
- GM (industrial collaborator): sorbent materials operating parameters, sorbent system modeling, and helical coil heat exchanger development
- Oregon State University (university collaborator): development of micro-channel internal bed heat exchanger and combustors
- Hexagon Lincoln (industrial collaborator): pressure vessel development for hydrogen storage system concepts
- PNNL (federal lab collaborator): team lead for cost modeling, bill of materials, and materials operating requirements
- UTRC (industrial collaborator): material particulate testing, MOF-5 thermal conductivity measurements, and on-board system modeling
- NREL (federal lab collaborator): vehicle level modeling, wells-to-wheels analysis, MOF-5 isotherm validation, and low temperature isotherms
- JPL (federal lab collaborator): insulation development and cryogenic parameter evaluation

Interactions include monthly team meetings (adsorbent system, coordinating counsel, and system modeling), regular data and information exchanges, and eleven HSECoE face-to-face meetings

Technical Back-up Slides

General FMEA Overview and Approach

The FMEA is based on the required system functions from the technical targets.

Technical System Targets: Onboard Hydrogen Storage for Light-Duty Fuel Cell Vehicles [a] (updated January 2015)			
Storage Parameter	**Units**	**2020**	**Ultimate**
System Gravimetric Capacity: Usable, specific-energy from H_2 (net useful energy/max system mass) [b]	kWh/kg (kg H_2/kg system)	1.8 (0.055)	2.5 (0.075)
System Volumetric Capacity: Usable energy density from H_2 (net useful energy/max system volume) [b]	kWh/L (kg H_2/L system)	1.3 (0.040)	2.3 (0.070)
Storage System Cost : • Fuel cost [c]	\$/kWh net (\$/kg H_2) \$/gge at pump	10 333 2-4	8 266 2-4
Durability/Operability: • Operating ambient temperature [d] • Min/max delivery temperature • Operational cycle life (1/4 tank to full) • Min delivery pressure from storage system • Max delivery pressure from storage system • Onboard Efficiency [e] • "Well" to Powerplant Efficiency [e]	 ºC ºC Cycles bar (abs) bar (abs) % %	 -40/60 (sun) -40/85 1500 5 12 90 60	 -40/60 (sun) -40/85 1500 5 12 90 60
Charging / Discharging Rates: • System fill time (5 kg) • Minimum full flow rate • Start time to full flow (20°C) • Start time to full flow (-20°C) • Transient response at operating temperature 10%–90% and 90%–0%	 min (kg H_2/min) (g/s)/kW s s s	 3.3 (1.5) 0.02 5 15 0.75	 2.5 (2.0) 0.02 5 15 0.75
Fuel Quality (H_2 from storage) [f]:	% H_2	SAE J2719 and ISO/PDTS 14687-2 (99.97% dry basis)	
Environmental Health & Safety: • Permeation & leakage [g] • Toxicity • Safety	 - - -	Meets or exceeds applicable standards	
Loss of useable H_2 [h]	(g/h)/kg H_2 stored	0.05	0.05

Cost of Ownership
(Provide a competitive system)

Accept Fuel
(Fill storage system)

Deliver Fuel
(Supply H_2 from storage system)

Store Fuel
(Manage H_2 in the system)

General FMEA Overview and Approach

Severity x Occurrence x Detection = RPN

Effect	Ranking
Hazardous without warning	10
Hazardous with warning	9
Very High	8
High	7
Moderate	6
Low	5
Very Low	4
Minor	3
Very Minor	2
None	1

Probability of Failure	Ranking
Very High: Persistent Failures	10
	9
High: Frequent Failures	8
	7
Moderate: Occasional Failures	6
	5
	4
Low: Relatively Few Failures	3
	2
Remote: Failure is Unlikely	1

Likelihood of Detection	Ranking
Absolute Uncertainty	10
Very Remote	9
Remote	8
Very Low	7
Low	6
Moderate	5
Moderately High	4
High	3
Very High	2
Almost Certain	1

Risk

Priority

Number