Oxygen Binding Materials and Highly Efficient Modular System for Oxygen Production
DE-FE-0027995

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Project Summary

Objective: Develop a process for efficient O_2 separation from air using adsorbents or membranes with oxygen carriers to produce high purity oxygen

- Process can be scaled up in modular form or in large-scale industrial processes
- Process should be competitive with current state-of-the-art processes
- Sorbent bed-factor of less than 600 lb-sorbent/TPD O_2 (tons/day O_2)
- An O_2/N_2 separation factor greater than 20 for membranes

Specific Challenges:

- Develop reversible oxygen binding solid materials with high O_2 capacity and selectivity
- Translate the reversible oxygen adsorption property of cobalt complexes to solid adsorbent or membrane form
- Optimize the chemical kinetics of oxygen adsorption on the sorbent
- Optimize the coordination environment of cobalt on the surface of the sorbent
- Determine oxygen separation process performances
- Consider use of the solid reversible oxygen binding materials on the surface of a membrane for oxygen separation at ambient temperatures

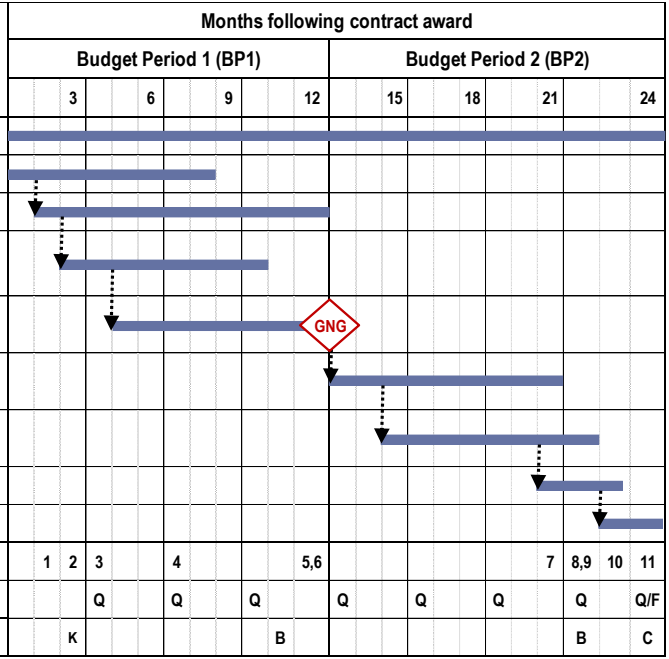
***Timeframe*:** BP1:10/1/16 to 09/30/17, BP2:10/1/17 to 09/30/18

Oxygen Separation Processes

- Vacuum-swing adsorption process can be used with the O_2 sorbents developed as indicated by the adsorption isotherm
- Membrane separation process is a partial pressure driven process and requires high feed-side pressure and low permeate-side pressure. In this case, a vacuum pressure can be used for the permeate side

Project Timeline

Task	Task title	Start date	End date
1.0	Project Management and Planning	10/01/16	09/30/18
2.0	Synthesis of O_2 Binding Materials	10/01/16	05/31/17
3.0	Characterization of O_2 Binding Materials	11/01/16	09/30/17
4.0	Formation of O_2 Binding Materials into Adsorbents and Membranes	12/01/16	07/31/17
5.0	Adsorbents and Membranes in Air Separation Processes	02/01/17	09/30/17
6.0	Optimization of O_2 Binding Materials for Adsorbent and Membrane Formation	10/01/17	06/30/18
7.0	Investigation of Material Performance in Air Separation Processes	12/01/17	07/31/18
8.0	Design of a 5-TPD Modular Air Separation Unit	06/01/18	08/31/18
9.0	Preliminary Techno-economic Analysis	08/01/18	09/30/18
Milestone Log		*(As noted)*	*(As noted)*
Reporting / Deliverables		*(See footnote.)*	*(See footnote.)*
Project Meeting		*(See footnote.)*	*(See footnote.)*



Q = Quarterly report due one month after quarter's end; F = Final report due three months after project end.

K = Project kick-off meeting; B = Project briefing (annual); C = Project closeout meeting

GNG = BP1 Go/No-Go Decision Point: (i) O_2-binding solid adsorbents with O_2 capacity equal to or greater than 0.5 wt% and/or (ii) a membrane O_2/N_2 selectivity equal to or greater than 20.

BP1 Milestones

BP	Task No.	Milestone Description	Planned Completion	Actual Completion	Verification Method
1	1	Updated Project Management Plan (PMP)	11/30/16	11/02/16	PMP file
1	1	Kickoff Meeting	12/31/16	12/02/16	Presentation file
1	2	Oxygen binding materials developed to initiate material characterization and O_2 separation process testing	01/31/17	1/31/17	Quarterly Report
1	3	O_2 binding material characterization completed	09/30/17	9/30/17	Quarterly Report/Final Report
1	2	Optimal O_2 binding down selected for adsorbent and membrane formation.	04/30/17	05/30/17	Quarterly Report
1	5	Achieve ≥0.5 wt% O_2 capacity for adsorbents and O_2/N_2 selectivity>20 for membrane	9/30/17	9/30/17	Quarterly Report

Our Innovation

- Materials that selectively adsorb/bind oxygen
- Vacuum-pressure-swing adsorption (VPSA) process can be used with the O_2 sorbents developed as indicated by the adsorption isotherm
- Higher purity oxygen product stream
- Higher oxygen recovery rate
- No need to treat the larger stream of nitrogen
- Possible to produce pure N_2 at the same time

Comparison of State-of-Art (SOTA) Oxygen Separation Technologies

Technology	Status	Production (TPD)	Purity (vol%)	By-product Capability	Start-Up Time
Cryogenic	Mature	>1,000	99+	Excellent	Hours
PSA	Mature	<150	95	Poor	Minutes
Membrane	Semi-mature	<20	~40	Poor	Minutes
ITM	Developing	Unknown	99+	Poor	Hours
RTI Technology	Developing	<50	95+	Excellent	Minutes

BSF as a Function of Oxygen Selective Sorbent O_2 Capacity

Sorbent Working Capacity (wt%)	O_2 (TPD)	BSF (lb sorbent/TPD O_2)
0.5	3.97	556
1	7.95	278
2	15.8	139
3	23.8	92.8
4	31.7	69.6
1.3*	2.59	850

*SOTA N_2 selectivity sorbent

- BSF: Bed Size Factor for PSA based system

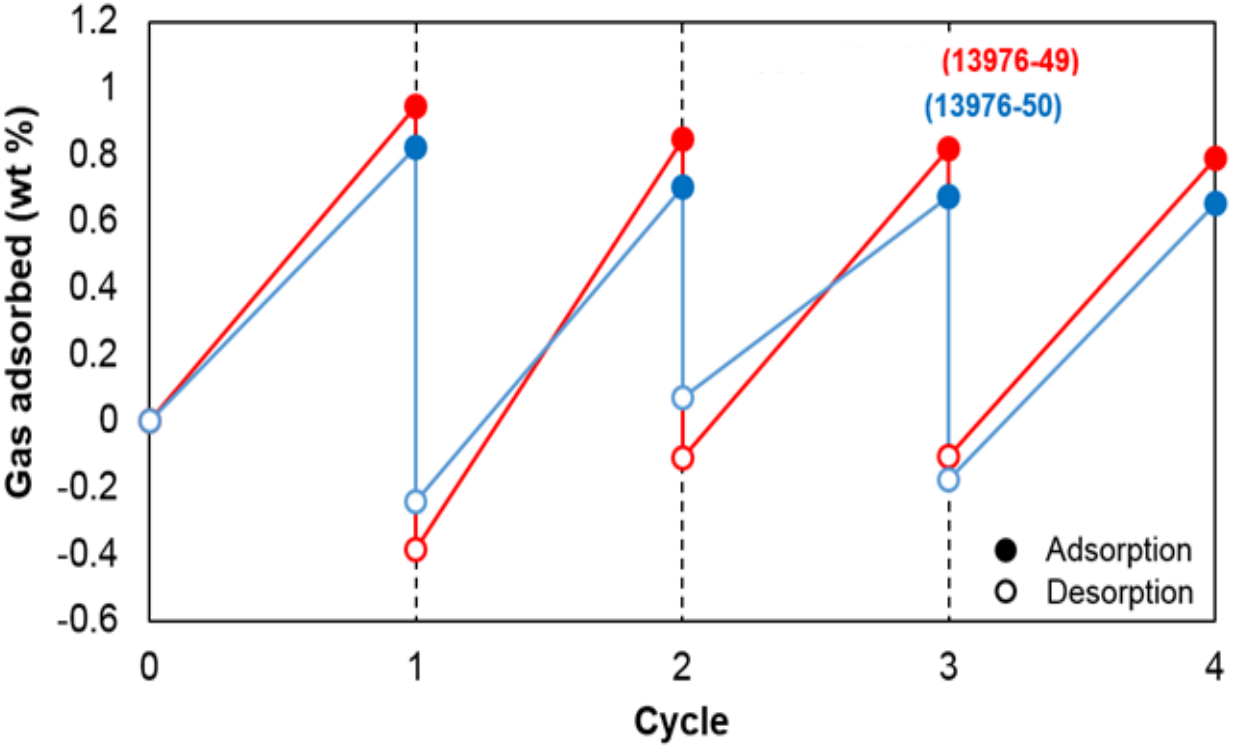
Success Criteria

Decision Point	Date	Success Criteria
GO/NO-GO decision points	9/31/2017	1) Selected O_2 binding materials made into adsorbents and membranes 2) Adsorbents with reversible O_2 capacity > 0.5wt% or 3) Membrane with O_2/N_2 selectivity > 20.
Completion of the project	9/30/2018	1) Techno-economic analysis delivered to DOE; and 2) Final report shows ≥95% O_2 purity and modular O_2 production system design

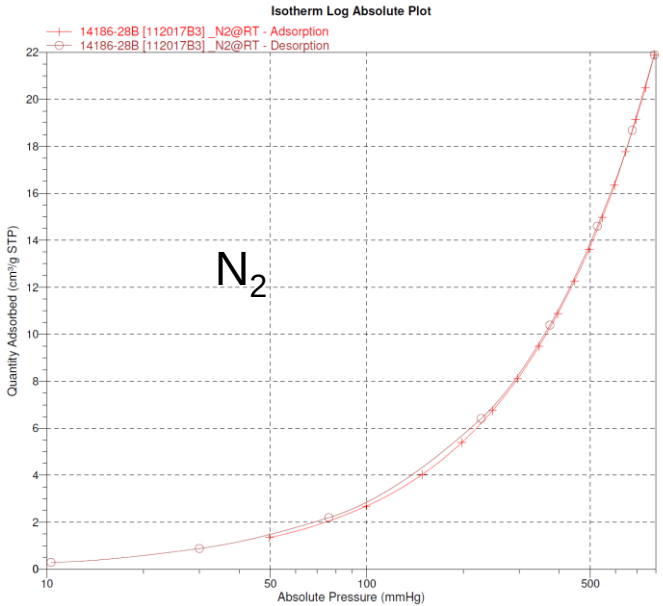
Characterization Adsorbent Materials

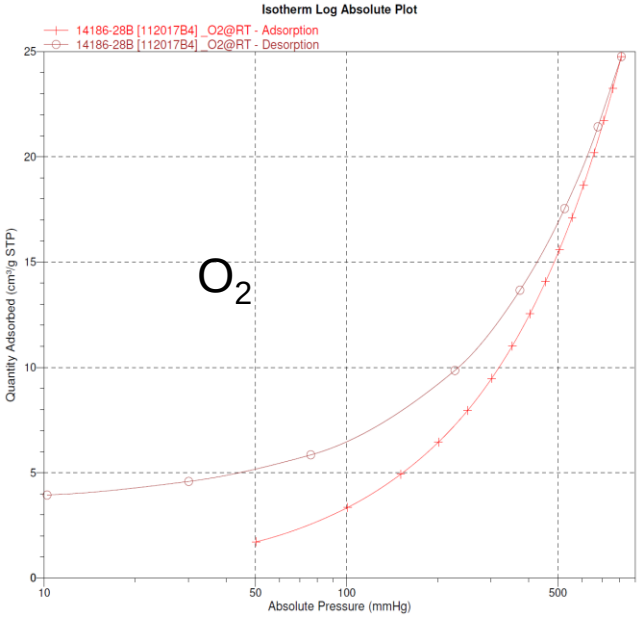
Sample #	Name	Sorbent synthesis	BET surface area (m^2/g)	Elemental content (%)			O_2 ads. (wt %)
				C (wt %)	N (wt %)	C/N	
MCM-41	MCM-41 silica	N/A	1033.7	0	0	0	--
SBA-15	SBA-15 silica	N/A	896.9	0	0	0	--
13976-31		Grafting	19.4	14.66	6.55	2.61	--
13976-32		Grafting	465.4	9.69	4.22	2.68	--
13976-36		Grafting	28.0	14.20	6.57	2.52	--
13976-37		Grafting	193.4	8.86	4.00	2.59	--
13976-38		Grafting	320.1	11.18	5.11	2.55	--
13976-43		Grafting	418.8	6.47	1.80	4.19	--
13976-44		Grafting	570.4	4.77	1.25	4.46	--
13976-49		Grafting	182.3	9.01	3.14	3.35	0.95
13976-50		Grafting	324.4	6.07	2.02	3.50	0.82
13976-51		Grafting	307.5	7.17	2.41	3.47	--
13976-52		Grafting	348.9	5.59	1.85	3.51	--
13976-66		Co-cond.	394.2	5.98	1.18	5.92	1.76
13976-67		Co-cond.	306.9	5.19	1.01	5.97	1.70
13976-68		Grafting	130.6	7.38	0.75	11.53	1.33
13976-69		Grafting	230.0	5.22	0.61	10.02	1.47
13976-74		Grafting	305.7	6.27	1.15	6.39	1.33
13976-75		Grafting	160.0	7.44	1.99	4.37	0.92
13976-76		Grafting	282.0	5.71	0.90	7.38	1.08

O_2 Adsorption-Desorption Cycle Tests by TG-MS

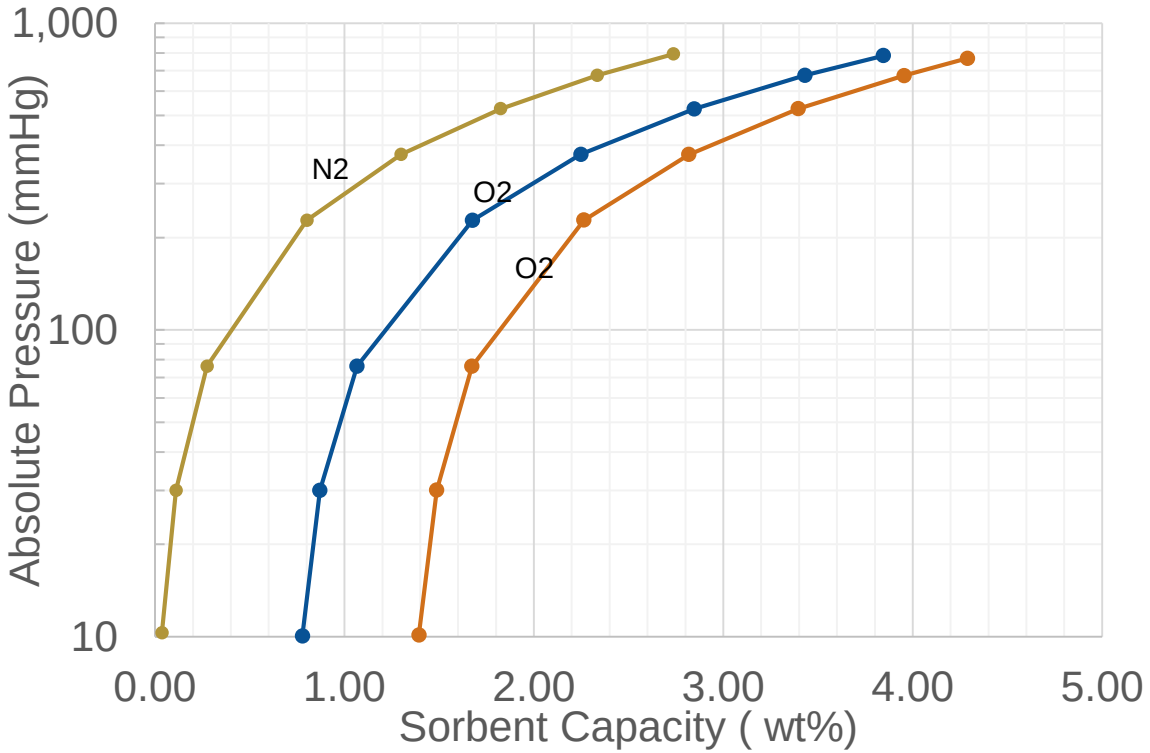


N_2 and O_2 Isothermal Curves@ RT





Isothermal Curves (Desorption) of N_2 and O_2 @ RT



Milestones to be Achieved

Task No.	Milestone Description	Planned Completion	Actual Completion	Verification Method
6	Selected O_2 binding materials further optimized for capacity, selectivity and kinetics	6/30/18		Quarterly Report
7	Selected O_2 binding materials' thermal, chemical and performance stability established	7/31/18		Quarterly Report
7	Achieve ≥95% O_2 purity in O_2 separation processes	7/31/18		Quarterly Report
1	Submit Annual Report	10/31/17		Annual Report
6	Selected O_2 binding materials further optimized for capacity, selectivity and kinetics	6/30/18		Quarterly Report
8	5 TPD modular O_2 production system designed	8/31/8		
9	Complete techno-economic analysis and issue TEA report	10/31/18		Final Technical Report
1	Submit Final Technical Report	10/31/18		Final Technical Report

Summary of Progress to Date

- Developed multiple synthesis routes for O_2 binding solid materials
- Synthesis optimization in progress for:
    1. High O_2 adsorption capacity (already reached 0.5 wt% BP1 target)
    2. Rapid O_2 adsorption
    3. Enhanced O_2 binding reversibility
    4. Improved material stability
    5. Efficient and economic synthesis routes

Modular Oxygen Production – Technology Roadmap

Previous Work	Current Project	Current Project	Future Deployment / Sustainment	
2016	2017 - 2018	2018 – 2020	2021 – 2023	2023+
Proof of Concept / Feasibility				Commercial Roll-out

Laboratory Validation

- Synthesis and characterization of O_2 binding materials
- Formation into sorbents and membranes
- Assessment of materials for air separation process
- Initial design of modular air separation unit
- Preliminary techno-economic assessment

Pilot Demonstration

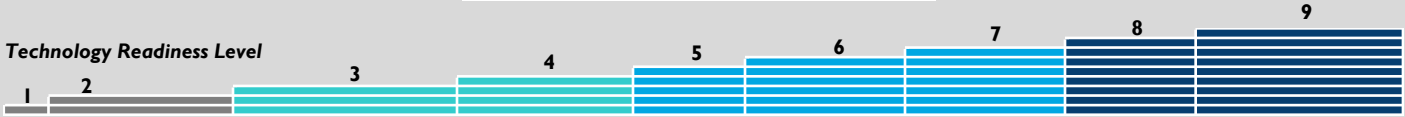
- Development and scale-up of materials into commercially producible form factor (commercial scale membrane module, sorbent quantities)
- Design, engineering and construction of modular pilot system
- Pilot operation
- Development of full process model
- Detailed TEA
- Development of process design package for full scale modular demonstration system (5-50 TPD O_2)
- Business arrangement with commercial partner, transfer in preparation of commercial offering.

Commercial Offering / Manufacturing 1st Adopter Demonstration

Potential Technology Owners:

- Sorbent manufacturing
- Design, engineering and construction of modular system (5-50 TPD O_2)
- Commercial scale demonstration at 1st adopter site

Ongoing sorbent & process refinements to increase market relevance and economic competitiveness



Acknowledgments

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Questions?

Thank you!