# HYDROGEN TECHNICAL ANALYSIS Project Progress Report

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### Abstract

Arthur D. Little is finalizing an analysis of small-scale purification technologies suitable for hydrogen fueling stations. In this project, Arthur D. Little identified and analyzed three technologies not currently supported by DOE that have some promise as hydrogen purification methods for hydrogen fueling stations. The technologies selected for detailed evaluation in this study were metal membranes without noble metals, dry fluorinated metal hydrides and the application of this fluorinated metal hydride in a slurry.

The detailed study was performed in the context of hydrogen fueling station hydrogen production and storage technologies. The analysis considered the technology R&D status, current and potential performance and cost, and possible development paths. Alternative fueling station scenarios were developed and compared on the basis of cost, performance and overall system efficiency.

Preliminary findings indicate that the use of metal hydride slurries for hydrogen purification and storage shows the highest overall off-board system efficiency of the three. Membranes without noble metals show the potential for the lowest hydrogen selling price for hydrogen production systems utilizing high-pressure steam reformers (hydrogen selling price results not presented here). Both of these, if proven, would represent significant improvements over the current state-of-the-art in hydrogen purification. However, considerable further development will be required to prove the feasibility and optimize the implementation of either of these options.

#### Introduction

There is increasing interest in the development of small-scale (< 1 million SCFD) hydrogen fueling stations to support direct hydrogen fuel cell vehicles when and if these vehicles capture a significant fraction of the U.S. passenger vehicle fleet. Considerable attention from DOE and others has been focused on small-scale hydrogen generation to minimize the cost of efficient decentralized hydrogen production, thus avoiding the cost of hydrogen transportation from central hydrogen production facilities. However, the critical step of purification and system integration has not received as much attention, although it has considerable impact on the efficiency and cost of hydrogen production. This relatively limited attention to small-scale purification and system integration may eventually result in barriers to broad implementation of hydrogen purification technologies, DOE wants to better understand what other options may be under development that they are not currently funding.

A hydrogen fueling station with on-site reformer-based production will require hydrogen purification, storage, and dispensing, along with the necessary safety and control components. Small, natural gas-based reformers, being developed for distributed fuel cell power systems, could potentially be used to generate hydrogen rich reformate streams at small-scale fueling stations servicing hydrogen fuel cell vehicles. Purification is an essential step to remove impurities in the reformate that may poison the storage unit or fuel cell, and to remove nonhydrogen species that can dramatically increase the size of the on-site and on-board storage systems.

This paper represents a progress report for this project. Some of the analysis is preliminary and the overall system-level analysis of cost and performance is ongoing. The final analysis will be incorporated into a final report shortly.

### Approach

We have reviewed three small-scale purification technologies not being funded by DOE. For the analysis, we considered the integration of these purification technologies for onsite hydrogen production at vehicle refueling stations. Both a larger (300 vehicle per day) and a smaller (30 vehicle per day) refueling station capacity were analyzed. The analysis included assessments of technical maturity and risks, performance, cost, and a comparison to baseline technologies, as well as the identification of key barriers, and an evaluation of possible development paths. For the performance and cost analysis, we developed detailed flowsheet models for each of the options considered, which included steam reformers as well as autothermal reformers. These flowsheet models were used to estimate the conditions, flowrates, power requirements, and heat transfer duties needed for sizing the equipment. Based on the equipment sizes so calculated, we then obtained cost estimates from quotes and from bottom-up cost models developed by Arthur D. Little under other programs.

For the selection of the purification methods to be studied in detail, we first generated a list of potential purification technologies based on literature information and discussions with DOE and others. A rigorous screening was conducted based on the expected applicability of the options to

distributed hydrogen production and on whether DOE already supports the option (options already supported were outside of this scope of the work). The options selected for detailed evaluation were:

- *Non-palladium metal membranes*. Non-palladium metal membranes are a potential low-cost alternative to palladium-based membranes currently in use. Japanese researchers have promising results from amorphous alloy membranes without noble metals (Zr-Ni). Alloys without noble metals may be two orders of magnitude cheaper than palladium-based materials on a weight basis (Hara 2000).
- *Fluorinated metal hydrides (dry).* Fluorinated metal alloys have the potential to simplify system integration by providing purification while storing the hydrogen on-site as a metal hydride. Forming a porous fluoride film on the surface of metal hydride particles is a promising way to protect the metal hydride from poisoning by non-hydrogen species that are less likely to penetrate through the fluoride film than hydrogen molecules (particularly carbon monoxide and water).
- *Metal hydride slurries*. Utilizing fluorinated metal hydrides in slurries could improve system integration even further and possibly allow fast fueling times if the slurry is also carried on the vehicle.

These technologies were also expected to have good potential to reduce cost and improve performance on the system level. On-site production with small-scale pressure swing adsorption (PSA) purification and delivered hydrogen (central production) with on-site dispensing were chosen as the baseline technologies for comparison. In this report, preliminary overall system efficiencies are presented for some scenarios. Hydrogen selling prices for all scenarios are currently being reviewed and are not included in this report. A complete list of scenarios for the final analysis is presented in Table 1.

Overall system efficiencies were determined based on integrated system modeling using thermodynamic software with inputs from developers and prior Arthur D. Little analyses. Optimum system configurations were determined and system models were developed for each scenario. Figure 1 shows the thermodynamic modeling role in determining system efficiency and hydrogen price.

Hydrogen selling prices (not presented here) were determined based on estimated equipment costs, energy costs, and additional conventional economic assumptions. Equipment costs for individual components were estimated based on vendor quotes, bottoms-up manufacturing cost analysis, or prior Arthur D. Little analyses. In some cases, progress ratios were applied to vendor quotes to obtain costs at high manufacturer production volumes. For most equipment, production volumes of 100 units/year were assumed. Higher production volumes were used for estimating low-pressure reformer costs, assuming a synergy between reformers manufactured for distributed fuel cell power systems and those manufactured for hydrogen production. Twenty-four vendors provided input to this analysis to date.

On-site Hydrogen Production Scenarios				Delivered Hydrogen Scenarios (Baseline)			
Purification Technology	Reformer	On-site Storage	On-board Storage		Delivered State	On-site Storage	On-board Storage
Pressure Swing Adsorption (Baseline)	Steam	High Pressure	cH <sub>2</sub> FCV		Liquid Hydrogen	Cryogenic and High Pressure	cH <sub>2</sub> FCV
	Autothermal	High Pressure	cH <sub>2</sub> FCV				
	Steam	Low Pressure	MH FCV			Cryogenic and Low Pressure	MH FCV
	Autothermal	Low Pressure	MH FCV				
Non-Pd Membrane	Steam	High Pressure	cH <sub>2</sub> FCV		Compressed Hydrogen via Tube Trailer	High Pressure	cH <sub>2</sub> FCV
	Autothermal	High Pressure	cH <sub>2</sub> FCV				
	Steam	Low Pressure	MH FCV			Low Pressure	MH FCV
	Autothermal	Low Pressure	MH FCV				
Fluorinated Metal Hydride (dry)	Steam	Low Pressure	MH FCV			None	cH <sub>2</sub> FCV
	Autothermal	Low Pressure	MH FCV		Compressed		
Fluorinated Metal Hydride Slurry	Steam	Low Pressure	MH Slurry FCV		Hydrogen via Pipeline	None	MH FCV
	Autothermal	Low Pressure	MH Slurry FCV			None	

# Table 1. Scenarios for Detailed Analysis

Notes: It was determined that the metal hydride options would not provide benefits for use with compressed hydrogen vehicles.  $CH_2 FCV = Compressed hydrogen fuel cell vehicle utilizing high-pressure on-board storage at 5000 psia.$ 

MH FCV = Metal hydride fuel cell vehicle utilizing low-pressure metal hydride storage at 100-150 psia.

MH Slurry FCV = Metal hydride fuel cell vehicle utilizing a low-pressure metal hydride slurry storage (oil plus metal hydride).

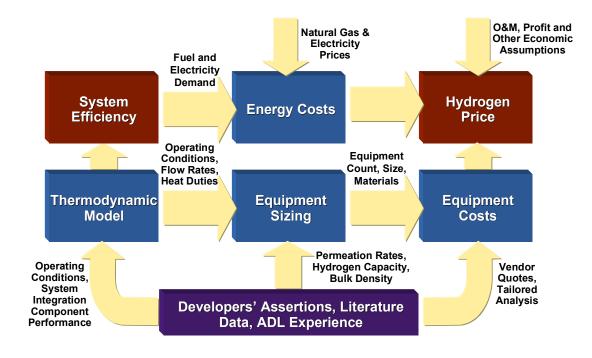


Figure 1. Approach for Determining Efficiency and Price

## Discussion

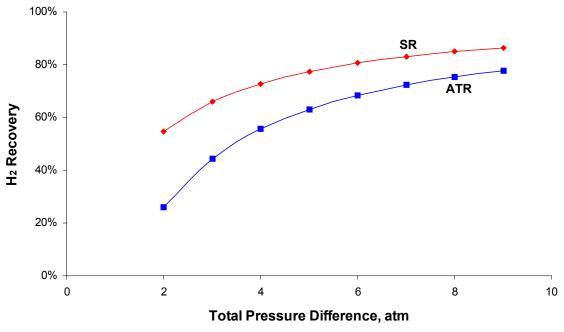
## **Amorphous Membranes without Noble Metals**

Amorphous membranes without noble metals are still in the very early research and development stage (Hara 2000). Japanese researchers were contacted to obtain the current performance of their amorphous  $Zr_{36}Ni_{64}$  alloy membrane for hydrogen separation. We developed long-term performance assumptions based on discussions with these developers (personal communication with Pr. S. Hara, National Institute of Materials and Chemical Research, Tsukuba, Japan) and Arthur D. Little comparative analysis. The long-term assumptions were made considering the membranes' intrinsic material properties such as mechanical properties and metal hydride formation conditions. Table 2 shows the current and long-term performance assumptions along with the current performance of a conventional palladium-based membrane.

Key Membrane Design Assumptions		Units	Zr-Ni Current	Zr-Ni Long-Term	Pd-Ag Current (Baseline)
Operating Temperature		°C	350	350	350
Pressure	Feed	atm	3	10	15
	Permeate	atm	1	1	5
Membrane Thickness		μm	30	15	30
Permeability at 350°C		10 <sup>₋9</sup> mol/m s Pa <sup>₀.₅</sup>	1.2	2.4	15
Permeability Reduction due to CO		%	10	10	5
H <sub>2</sub> Concentration in Permeate		%	100	100	100
Hydrogen Recovery for SR		%	53	86	92

# Table 2. Membrane Performance Assumptions

In order to achieve these long-term performance goals, a significant improvement in operating pressure difference across the membrane is necessary (see Figure 2). Note that the hydrogen recovery is also a function of reformate composition. Reformate from steam reforming (SR) natural gas gives higher hydrogen recovery than reformate from autothermal reforming (ATR) natural gas. Such improvements in performance would greatly improve the performance of the membranes, but there is still considerable technology development risk involved in the development and commercialization of purification systems based on these membrane materials (as compared to the risks associated even with palladium-containing metal membranes).



\* Maximum achievable membrane hydrogen recovery assuming equilibrium conditions.

# Figure 2. Membrane Recovery\* as a Function of Pressure

It is believed that operating pressure for Zr-Ni amorphous membranes is limited by crystallization process conditions and not by mechanical properties of the material (Ritchie 2000; Ismail 2001). Amorphous Zr-Ni-based membranes show mechanical properties, such as tensile strength, hardness and Young's modulus, comparable or superior to those of conventional metal-based membrane materials (Davis 1999). The current low operating pressure difference for Zr-Ni membranes is due to crystallization of the membrane material at assumed temperature and pressure. Crystallization of amorphous materials in the presence of hydrogen is caused by metal hydride formation at certain conditions and leads to a dramatic loss in hydrogen permeability. Metal hydride formation conditions strongly depend on the alloy composition and, thus, can be tailored by modifying membrane material composition such as optimizing component ratios and/or adding small amounts of other metals.

# Fluorinated Metal Hydrides (Dry)

If successful, the use of metal hydride-based purification technology could significantly reduce the pressure requirements for the raw hydrogen stream, as well as the compression requirements for the purified hydrogen stream. Hydride-based systems could work at low-pressure and possibly even be used to pressurize the hydrogen partially, prior to the final compression stages (Golben 1999; Vanhanen 1999). Conventional purification technologies (PSA, membranes) require high reformate pressures (> 10atm) to obtain acceptable hydrogen recovery at reasonable cost. This high-pressure requirement significantly increases cost and compressor parasitic power; especially if low-pressure reformers, developed and produced for distributed fuel cell power systems, are to be used. Such reformers will likely deliver reformate at between 1-3 atm.

The advantage to using these reformers in a fueling station is that their costs are likely to be much lower due to the relatively high production volumes projected for distributed fuel cell power systems. However, operating low-pressure reformers with conventional purification systems will require very large reformate compressors, adding significant cost and parasitic power (decreasing system efficiency). For example, a reformate compressor can use as much as 5% of the hydrogen energy content for electric power to increase reformate pressure from 3 to 10 atm (assuming reformate from an ATR and 70% efficient compressor). When typical power plant efficiencies are taken into account, this electric load can significantly reduce overall system efficiency.

If protected from certain impurities, metal hydriding alloys could provide efficient hydrogen purification at low-pressure, permitting the use of potentially low-cost reformers without expensive and power intensive reformate compressors. Japanese researchers claim that fluorination permits hydriding alloys to tolerate high levels of impurities, and have carried out promising initial feasibility experiments (Liu 1995; Wang 1995). The working hypothesis is that hydrogen molecules could easily penetrate a porous fluoride top layer through microcracks, while larger molecules (impurities) could not. However, the fluoride coating is not infinitely selective to hydrogen, so the hydriding alloy is still poisoned by impurities even if treated. The four basic intermetallic impurity interactions and their effects are listed in Table 3. The quantitative effects on metal hydride performance depends on the impurity nature, fluorination treatment method, and impurity concentration in the bulk gas.

Impurity Interactions	Effect	Compounds	
Poisoning	Rapid loss of hydrogen capacity with cycling, caused by impurities strongly or irreversibly adsorbed on the surface active sites	H <sub>2</sub> S, Organic Sulfur Compounds, CO	
Retardation	Reduction in adsorption /desorption kinetics without significant loss in the ultimate capacity, caused by impurities reversibly adsorbed on the surface active sites	$CO, CO_2, NH_3$	
Reaction	Bulk corrosion leading to irreversible capacity loss	O <sub>2</sub> , H <sub>2</sub> O	
Innocuous	Loss in adsorption kinetics due to surface blanketing	N <sub>2</sub> , CH <sub>4</sub>	

# Table 3. Metal Hydride Impurity Interactions

Assuming sufficient sulfur removal prior to reforming, the main impurities of concern in reformate streams are carbon dioxide (CO<sub>2</sub>), carbon monoxide (CO), and water (H<sub>2</sub>O). We assumed the levels of these impurities found in reformate would not significantly affect hydride performance even after thousands of cycles. This assumption is based on developers' claims of

respective impurity tolerances of 20% and 3,000 ppm (dry volume) for carbon dioxide and carbon monoxide, and no effect of water on the fluorinated metal hydride (personal communication with Pr. S Suda, Kogakuin University, Tokyo, Japan; personal communication with K. Kobayashi, Japan Metals and Chemicals Co., Ltd., Japan). No conclusive tests have been carried out to demonstrate that fluorination alone can provide this level of tolerance but no information to the contrary is available either. Table 4 shows the performance assumptions for the metal hydride purification and storage system.

Key FI MH Design Assumptions		Units	LmNi <sub>4.6</sub> Mn <sub>0.4</sub>
Absolute H <sub>2</sub> Capacity		%	1.34
Reversible H <sub>2</sub> Capacity		%	1.21
Heat of Hydrogenation Reaction		kJ/mol	-39.4
Hysteresis		$ln(P_a/P_d)$	0.05
Hydrogen Recovery for SR		%	90
Inlet Conditions	Temperature	°C	40
	Pressure	atm	3 and 10 *
Outlet Conditions	Temperature	°C	126
	Pressure	atm	10

## Table 4. Metal Hydride Performance Assumptions

Lm is a lanthanum (La) rich mesch metal.

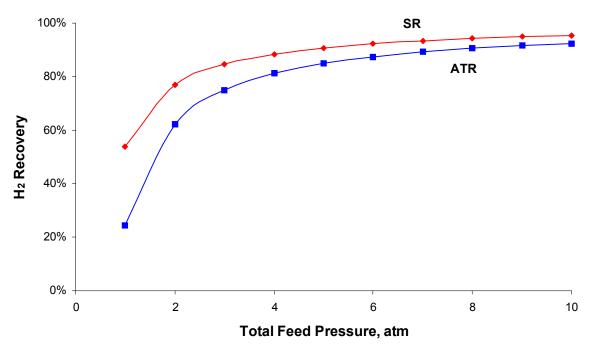
\* 3 atm inlet pressure assumes low-pressure reformers, 10 atm assumes high-pressure reformers

manufactured specifically for hydrogen production. Hydrogen recovery assumes 10 atm operation.

Metal hydrides are being considered for on-site and on-board storage, due in large part to their potential safety advantage over compressed and liquid hydrogen storage. The disadvantage to metal hydrides is their high cost and low storage density (1-5% wt). Weight is not a major concern for on-site storage, but cost is.

Fluorinating the metal hydrides could provide purification at a relatively small additional cost, making the metal hydride system more cost competitive overall. Japanese academic researchers developed a metal hydride fluorination technique involving metal hydride treatment with fluorine salt in aqueous media (Sun 1999; Liu 1995). A Japanese materials company is developing a fluorination process that does not require use of an aqueous system or, consequently, expensive waste-water treatment system (personal communication with F. Liu, former Kogakuin University, Tokyo, Japan; personal communication with K. Kobayashi, Japan Metals and Chemicals, Japan). The proposed technology employs a fluoro-treatment in the gas phase, similar to other fluorination processes currently employed in the chemical industry.

Combining storage and purification has the additional advantage of long residence times. Innocuous and other non-hydrogen species may significantly reduce the hydrogen absorption kinetics. However, assuming very long residence times during storage, we have estimated the hydrogen recovery to be limited by system equilibrium only. Figure 3 shows the hydrogen recovery versus system pressure.



\* Maximum achievable LaNi<sub>4.6</sub>Mn<sub>0.4</sub> hydrogen recovery assuming equilibrium conditions.

# Figure 3. Fluorinated Metal Hydride Recovery\* as a Function of Pressure

### **Fluorinated Metal Hydride Slurries**

Metal hydride slurries consist of metal hydrides suspended in a liquid material, such as an oil. These slurries can be stored and pumped like a viscous liquid, simplifying and speeding handling and heat transfer. However, slurries suffer from the same high cost as dry metal hydrides and have even lower weight density. Still, with improvements in metal hydride technology, slurries could become a very attractive option, at least for on-site storage.

The performance assumptions for the fluorinated metal hydride slurries are essentially the same as for the dry fluorinated metal hydrides. Experimental and pilot scale work with slurries in the Netherlands has shown that the oil has no noticeable effect on various hydrides (Holstvoogd 1989). As continuous adsorption and desorption processes can be used in this case, higher recoveries are possible with reasonable cost. A higher hydrogen recovery, 94% at 10 atm, was assumed on the basis of a continuous process.

Metal hydride slurry hydrogen purification technology was developed for large-scale applications, such as hydrogen for ammonia plants (Zwart 1989). The technology did not find an industrial application due to high product cost in comparison with large-scale membranes and PSA hydrogen separation. However, metal hydride slurries may present significant advantages

over other processes as it combines hydrogen separation with storage and allows easier and faster vehicle fueling. No fuel cell vehicles have demonstrated this technology for on-board storage. Even if the application for on-board storage would be un-attractive, its use for off-board storage could offer considerable handling, thermal integration, and control benefits over dry hydride systems.

#### Results

## System Efficiency

Individual sub-system performance was determined based on the appropriate integration. For example, purification off-gas (containing un-recovered hydrogen) is recycled for use in the reformer to drive the steam reforming reaction and/or preheat gases. Table 5 shows two examples of the sub-system efficiencies that have been calculated from thermodynamic modeling of high-pressure (~10 atm operating pressure) steam reformer systems.

# Table 5. Sub-system Efficiencies Utilizing High-pressure Steam Reformers

Assuming high-pressure storage for compressed H<sub>2</sub> fuel cell vehicles

System Perfo	ormance, % (LHV)	PSA (baseline)	Non-Pd Membrane*
Gas Efficiency = <u>LHV Fuel out</u>	Production <sup>1</sup>	100	93
	Purification	76	86
LHV Fuel in	Storage/Compression	97 <sup>2</sup>	97 <sup>2</sup>
Parasitic Loss =	Production	1.6	1.5
Parasitic Power <sup>3</sup>	Purification	0.6	0.6
LHV H <sub>2</sub> out	Storage/Compression	7.9 <sup>4</sup>	12.3 <sup>4</sup>
System Efficiency including power plan	nt efficiency penalty⁵	62	59

\* Based on long term performance assumptions (see Table 2).

<sup>1</sup> Reformer efficiency calculation does not include the purification and storage recycle gases as fuel inputs (i.e. they are considered "free" inputs). Combined reformer and purification gas efficiencies range from 77-80%.

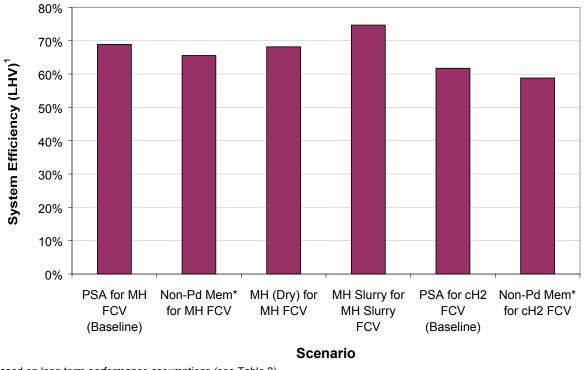
<sup>2</sup> Includes 3% loss of hydrogen from hydrogen compressors that is recovered and used in the reformer.

<sup>3</sup> Includes water pumps, compressors, blowers, and cooling fans.

<sup>4</sup> Over 90% of this power requirement comes from the compressors required to pressurize the hydrogen to storage pressure (3600 psia) and on-board vehicle pressure (assumed to be 5000 psia).

<sup>5</sup> Assumes power plant efficiency is 35% LHV based on the average power plant efficiency of a conventional natural gas steam power plant (a combined cycle natural gas power plant average efficiency is 45% LHV). System Efficiency = (Outlet H<sub>2</sub> LHV)/(Input Natural Gas LHV + Total Parasitic Power/35%).

Overall system efficiencies plotted in Figure 4 show efficiencies are higher for all low-pressure storage options (for MH FCV) versus high-pressure storage options (for  $CH_2$  FCV). This doesn't take into account the on-board efficiencies that should be lower for the low-pressure storage options due to the increased weight of the on-board metal hydride storage. It is more useful to compare separately the low and high-pressure storage options.



\* Based on long term performance assumptions (see Table 2).

<sup>1</sup> Includes a power plant efficiency penalty. Assumes power plant efficiency is 35% LHV based on the average power plant efficiency of a conventional natural gas steam power plant (a combined cycle natural gas power plant average efficiency is 45% LHV). System Efficiency = (Outlet H<sub>2</sub> LHV)/(Input Natural Gas LHV + Total Parasitic Power/35%).

<sup>2</sup> 10 atm operating pressure

Note: Does not include vehicle fuel efficiency. On-board MH storage options will likely be heavier and have lower fuel economy.

# Figure 4. System Efficiencies Utilizing High-pressure<sup>2</sup> Steam Reformers

The metal hydride slurry option has the highest efficiency due to the high hydrogen recovery for purification and the very low parasitic loads. The metal hydride (dry) is less efficient because the on-site metal hydride storage was assumed to be separate from the on-board storage. This requires the use of an auxiliary burner to generate heat for hydrogen desorption during fueling. While the reformer exhaust provides some heat, most heat is supplied by burning natural gas in the burner (alternatively, additional heat could be supplied by electric heaters). On-site and on-board storage could be combined to increase efficiency, but switching these heavy containers could be unwieldy and would introduce considerable complexity into the fueling and charging process. Slurries, on the other hand, are easily transported from on-site to on-board storage so that on-site hydrogen desorption is not required.

Despite higher assumed hydrogen recovery from purification, the non-palladium metal membrane option has slightly lower efficiency than the pressure swing adsorption baseline. With a steam reformer, not much is gained by increasing purification hydrogen recovery alone because this reformer can efficiently use the un-recovered hydrogen to provide heat for the steam reforming reaction. Results are much different when an autothermal reformer is assumed (results not presented here). The non-palladium option has slightly lower efficiency than the pressure swing adsorption baseline due to additional power required for compressing hydrogen at the membrane exit pressure ( $\sim 1$  atm) compared to adsorber exit pressure ( $\sim 9$  atm).

It should be noted that the results presented here are for high-pressure ( $\sim 10$  atm) steam reformer systems only. Modeling low-pressure ( $\sim 3$  atm) steam reformers or autothermal reformers has much different results.

# **Preliminary Conclusions**

Our analysis to date indicates that the use of fluorinated metal hydrides in slurry form could provide significant benefits in terms of off-board system efficiency, especially if the slurry could also be used for on-board storage. Even if only used for purification and on-site storage in decentralized hydrogen production facilities, the use of metal hydride slurries should provide significant efficiency benefits over conventional decentralized hydrogen purification and storage options. However, some technology development will be required to optimize the slurry system, and significant development will be required to develop a stable and effective fluorinated metal hydride that can be produced cost-effectively.

If non-palladium metal membranes could be further developed to operate with reasonable fluxes and at higher pressures, they could provide a very competitive alternative to PSA-based separation and purification systems, though they would not likely yield significant improvements in efficiency.

Based on these results, combined with our ongoing cost analysis, we will make recommendations to DOE regarding the viability of each option, and regarding appropriate R&D activities to further their development.

# Acknowledgments

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