EXTENDED OPERATIONS OF THE PRATT & WHITNEY ROCKETDYNE PILOT-SCALE COMPACT REFORMER

Year 6 – Activity 3.2 – Development of a National Center for Hydrogen Technology

Topical Report

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National Energy Technology Laboratory
U.S. Department of Energy
626 Cochran Mill Road
PO Box 10940, MS 921-107
Pittsburgh, PA 15236-0940

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Performance Monitor: Darryl Shockley

Prepared by:

Jay C. Almlie

Energy & Environmental Research Center
University of North Dakota
15 North 23rd Street, Stop 9018
Grand Forks, ND 58202-9018

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ABSTRACT

U.S. and global demand for hydrogen is large and growing for use in the production of chemicals, materials, foods, pharmaceuticals, and fuels (including some low-carbon biofuels). Conventional hydrogen production technologies are expensive, have sizeable space requirements, and are large carbon dioxide emitters. A novel sorbent-based hydrogen production technology is being developed and advanced toward field demonstration that promises smaller size, greater efficiency, lower costs, and reduced to no net carbon dioxide emissions compared to conventional hydrogen production technology. Development efforts at the pilot scale have addressed materials compatibility, hot-gas filtration, and high-temperature solids transport and metering, among other issues, and have provided the basis for a preliminary process design with associated economics. The process was able to achieve a 93% hydrogen purity on a purge gas-free basis directly out of the pilot unit prior to downstream purification.

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EXECUTIVE SUMMARY

Hydrogen is a key feedstock and chemical component for many industrial processes. Currently, 40–45 million tonnes a year of hydrogen is produced worldwide, with about a third of that attributed to the United States. Hydrogen production is forecast to grow about 3.5% annually for the next 2 years, with the Asia–Pacific countries growing faster than other regions of the world. Most of that growth will be due to existing hydrogen uses. As much as demand growth, increased energy costs and new environmental regulations will likely drive hydrogen producers to install new production technologies and modify existing facilities to increase production capacity, achieve improved efficiencies, and reduce emission levels to assure their competitiveness and compliance.

Of the hydrogen production technologies that have been commercialized, conventional steam methane reforming (SMR) is the most prevalent, producing 80%–95% of the hydrogen generated in the United States today and about 45% worldwide. While conventional SMR is the preferred hydrogen production technology, it has several issues, which include equipment size, efficiency, lack of integration, capital cost, and cost of incorporating carbon dioxide capture. SMR-based hydrogen units, to a great extent, are designed individually, constructed on-site, and consist of a large SMR (the size of a multistory building) unit, one or more water–gas shift reactors, and a pressure swing absorption (PSA) hydrogen purifier, along with associated desulfurizing, heat recovery, and other equipment.

Pratt & Whitney Rocketdyne (PWR) is developing a potentially “disruptive” hydrogen production technology. The term “disruptive” is used because PWR believes this technology could disrupt the economics of today’s hydrogen production market. PWR’s hydrogen production technology represents an alternative technology to conventional SMR. PWR’s technology applies process intensification concepts to arrive at a proprietary regenerative sorbent process that is capable of producing industrially useful quantities of hydrogen and that possesses significant advantages over conventional SMR technology. These advantages include the following:

- Compact, modular design
- Improved efficiency
- Less product gas cleanup
- Reduced greenhouse gas emissions at less cost
- Lower equipment cost

The PWR hydrogen generator design is expected to provide sorbent-enhanced performance while avoiding issues experienced by some other sorbent-enhanced reforming technologies.

Additionally, PWR's hydrogen generator will provide improved performance without the need for expensive air separation units or oxygen carriers and will convert feeds other than natural gas without the addition of a prereformer. The design also promises to be flexible with respect to scale. Thus the PWR hydrogen generator concept overcomes many of the deficiencies of SMR and other hydrogen production technologies.

The goal of the current scope of work was to advance the state of operability of the pilot-scale unit to the point of being able to generate hydrogen continuously for more than 30 hours. The technical team deemed the goal attainable based upon successes in achieving continuous solids flows and operational procedure advances achieved during the past year of development effort under National Center for Hydrogen Technology (NCHT) funding. This was a continuation of previously proposed work.

The team proposed the following set of tasks to complete this scope of work:

- Prepare the pilot-scale test hardware for extended, continuous test operation with hydrogen production occurring during a majority of the duration of the operations.
- Modify operational and start-up procedures, based upon knowledge accumulated during the prior year of development and test efforts.
- Conduct extended, continuous test operations with hydrogen production.

This scope of work is a continuation of work performed prior to NCHT Year 6 (No. DE-FE0003466). Therefore, extensive reporting on prior work is presented in the final topical report for this project for NCHT Years 3–5 (Contract No. DE-FC26-05NT42465). A new U.S. Department of Energy contract dictated separate reporting for Year 6 alone.

The pilot-scale test unit was successfully prepared for test operations, according to plan. As part of these preparations, a significant rebuilding of the rotary valve below the standpipe needed to be completed. Significant effort was also put into formalizing start-up procedures, operating procedures, and system performance-monitoring procedures. This was necessitated by the fact that the pilot-scale system has proven much more temperamental to operate than originally expected at the inception of the project.

Finally, two extended test operations were completed in December 2010. These test runs were completed during the periods of 11/29/10–12/3/10 and 12/4/10–12/8/10. These runs served to prove a repeatable degradation of catalyst performance. The test runs resulted in collection of some of the most meaningful process data to date in this development program. The usefulness of the analysis of the results from the tests funded by Year 6 funding was to provide multiple data sets that lend insight into the causes of the catalyst performance degradation.

The team had an objective of achieving 30 hours of continuous hydrogen production was not achieved because of the repeatable catalyst deactivation. Although the objective was not reached, the team viewed this as a partial success because the process itself is now being

evaluated, rather than the process support mechanisms. Additional work is now being carried out under separate funding sources.

EXTENDED OPERATIONS OF THE PRATT & WHITNEY ROCKETDYNE PILOT-SCALE COMPACT REFORMER

Year 6 – Activity 3.2 – Development of a National Center for Hydrogen Technology

APPROACH

Hydrogen is a key feedstock and chemical component for many industrial processes, which include the following:

- Ammonia production – The production of ammonia involves reaction of nitrogen from the air with hydrogen to produce ammonia using the Haber process. Ammonia itself is then used in fertilizer manufacture, industrial refrigeration systems, and the manufacture of a variety of industrial chemicals.
- Oil refining – In oil refineries, hydrogen is used to upgrade viscous oil fractions to produce products such as gasoline and diesel and for removing contaminants such as sulfur. Demand for hydrogen in refineries is increasing dramatically as standards for fuels tighten in the United States and Europe and as sulfur content is more tightly regulated.
- Chemicals – Hydrogen is used as a raw material in the chemical synthesis of hydrogen peroxide, polymers, and solvents. Hydrogen is also used to purify gases (e.g., argon) that contain trace amounts of oxygen, using a catalytic combination of the oxygen and hydrogen followed by removal of the resulting water.
- Pharmaceuticals – The pharmaceutical industry uses hydrogen to manufacture vitamins and other pharmaceutical products.
- Food and beverages – Hydrogen is used to hydrogenate unsaturated fatty acids in animal and vegetable oils, producing solid fats for margarine and other food products.
- Semiconductor manufacture – The semiconductor industry uses hydrogen to stabilize amorphous silicon and carbon materials.
- Miscellaneous – Generators in large power plants are often cooled with hydrogen since the gas possesses high thermal conductivity and offers low friction resistance. The nuclear fuel industry also uses hydrogen as a protective atmosphere in the fabrication of fuel rods.

Currently, 40–45 million tonnes a year of hydrogen is produced worldwide, with about a third of that attributed to the United States. Hydrogen production is forecast to grow about 3.5% annually for the next 2 years, with the Asia–Pacific countries growing faster than other regions of the world. Most of that growth will be because of existing hydrogen uses, with only a small amount to support new uses, such as biofuel hydrotreatment and fuel for stationary power units.

As much as demand growth, increased energy costs and new environmental regulations will likely drive hydrogen producers to install new production technologies and modify existing facilities to increase production capacity, achieve improved efficiencies, and reduce emission levels to assure their competitiveness and compliance.

Hydrogen can be derived from water and various carbonaceous sources by way of reactions such as steam methane reforming (SMR), partial oxidation of methane, and carbon dioxide reforming:

Partial oxidation of carbon and Boudouard reactions:

As well as water–gas shift:

Conventional hydrogen production is carbon dioxide-intensive. In many refineries, carbon dioxide emissions from reforming (hydrogen production) units are second only to the large fluidized catalytic cracking (FCC) units. Refineries that have hydrocrackers instead of FCCs depend even more on hydrogen production. Hydrogen is used both to produce fuels and to remove environmentally harmful contaminants from fuels. Increased demand for refined products and greater environmental regulations in the United States will increase refinery hydrogen needs and, thus, carbon dioxide emissions. Switching to environmentally friendly biofuels will not necessarily alter this situation because many biofuels require hydrogen in their production. Thus there is an acute need for an inexpensive hydrogen production technology that is less carbon dioxide-intensive.

Many technologies have been and are being developed that employ these reactions in various ways to manufacture hydrogen. These technologies differ not only by the reactions that they incorporate and feedstocks that they consume, but also by production costs, process efficiency, level of technical maturity, readiness for commercialization, oxidant, physical size, emissions, and compatibility with emission treatment, such as carbon dioxide capture, technologies. Additionally, unit costs (cost per unit of production) vary with factors such as scale both in absolute terms (economies of scale often reduce unit costs with increased size) and in relative terms by comparisons amongst technologies (technologies have different scales at which their unit costs are minimal). Table 1 highlights some of these differences.

Table 1. Hydrogen Production Technology Comparison

Technology	Maturity	CO ₂ Capture ¹	Feedstock ²	Oxidant Source	Optimal size, tonne/day	Application
Gasification	Commercial	++	C	O ₂ , H ₂ O		
SMR						
Conventional	Commercial	+	NG	H ₂ O	<500	
Membrane ³	R&D	+++	NG	H ₂ O		
Sorbent ³	R&D	+++	NG	H ₂ O		
Partial Oxidation	Commercial	++	NG	Air, O ₂ , H ₂ O		
Methane Autothermal Reformer	Commercial	++	NG	Air, O ₂ , H ₂ O	Very large	Flexible CO:H ₂ ratio in product
Hydro Hydrogen Membrane- Reforming	R&D	+++	NG	H ₂ O		Power generation
Chemical Looping	R&D	+++	C, NG	Metal oxide carrier		

¹ CO₂ capture compatibility: increasing “+” symbols implies increasing compatibility.

² Feedstock: C – coal, petcoke; NG – natural gas.

³ “Membrane” and “sorbent” refer to reformer-integrated H₂ and CO₂ separation devices.

Of the hydrogen production technologies that have been commercialized, conventional SMR is the most prevalent, producing 80%–95% of the hydrogen generated in the United States today and about 45% worldwide. Commercial partial oxidation (POX) and autothermal reformer (ATR) installations are far less numerous: in 2007 Sandia National Laboratory estimated on the order of 50 POX and a handful of ATR commercial units were operating globally. The situation is similar for gasification installations.

Despite the prevalence of SMR, research continues into new gasification and reforming technologies, which is motivated by improved efficiencies, increased operability and reliability, reduced costs and, especially, evolving emission regulations. Alstom has been developing processes that circulate oxygen carriers between two reactors: one in which the carrier is oxidized by exposure to air and the other in which it is reduced by exposure to a carbonaceous fuel so as to produce syngas. By using the carrier to extract oxygen from air and circulating the carrier at a substoichiometric rate, the carbonaceous fuel is partially oxidized without nitrogen dilution or expensive air separation. The research has studied the use of natural ores as oxygen carriers for coal conversion and supported metal particles for natural gas conversion. Hydro, now Statoil, has researched a hydrogen membrane-reforming (HMR) concept in which multiple membrane reactors in series would produce a carbon-free hydrogen stream for a gas turbine power generation application (1). Natural gas and steam were fed to one side of the first reactor where reforming and shifting would occur to produce hydrogen that would transfer to the other side of the membrane. Heat for the reaction was provided by feeding air to the other side of the reactor and combusting the hydrogen received from the reformer side. Both streams progressed

to the next membrane reactor where further reforming, shifting, and transfer would occur on one side of the membrane and some limited combustion to the other. The reformer product stream contained all the carbon (now capture-ready), and the hydrogen product stream contained the hydrogen as well as nitrogen and water from the combustion stream. The diluents in the hydrogen stream were at acceptable levels for a gas turbine fuel (see Figure 1).

Membrane- and sorbent-enhanced SMR are two concepts that have been investigated that focus on carbon dioxide capture. For example, BOC (now part of the Linde Group) has studied placing a hydrogen separation membrane in a fluidized-bed reformer to extract hydrogen and enhance conversion, and Chevron has researched integration of a sorbent bed into a reformer to capture carbon dioxide during reforming. As with the membrane, removal of a reaction product shifts the equilibrium to enhance conversion, so that the reaction can progress further at lower temperatures than in conventional reforming. Once saturated, the sorbent was regenerated off-line, thus making the carbon dioxide product available for capture and sequestration. Two significant concerns regarding these technologies are the mechanical strength of the membrane to withstand abrasion by catalyst in the reformer and the mechanical stability of the sorbent to tolerate the cyclic expansion and contraction of the crystal lattice because of carbon dioxide adsorption and desorption. Developers have reported sorbent pulverization in fixed-bed designs.

In the absence of membrane and sorbent enhancement, gasification and reforming technologies produce product streams that contain significant amounts of carbon monoxide. It is common practice to improve process efficiency and hydrogen yields and to reduce carbon monoxide concentrations by adding one or more stages of water–gas shift downstream of gasifiers and reformers. The first stage tends to be run at higher temperatures to promote conversion rate (kinetics) over equilibrium concentration (thermodynamics). A second stage, if adopted, tends to be run at lower temperatures to shift the equilibrium to the products and, thereby, produce more hydrogen.

While conventional SMR is the preferred hydrogen production technology, it has several issues, which include equipment size, efficiency, lack of integration, capital cost, and cost of incorporating carbon dioxide capture. SMR-based hydrogen units, to a great extent, are designed individually, constructed on-site, and consist of a large SMR (the size of a multistory building), one or more water–gas shift reactors, and a pressure swing absorption (PSA) hydrogen purifier, along with associated desulfurizing, heat recovery, and other equipment. Efficiencies of

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Figure 1. Hydro's HMR.

commercial-scale equipment run 60%–70% (lower heating value [LHV]), which is much reduced when carbon dioxide capture is included. Improved efficiencies (above 65%) and carbon dioxide capture come with significant additional equipment complexity and cost.

BACKGROUND

Pratt & Whitney Rocketdyne (PWR) is a company with a long history in the development of hydrogen-based technologies, primarily with respect to its use as a rocket engine fuel. A recent effort at PWR has resulted in the development of a potentially “disruptive” hydrogen production technology. The term “disruptive” is used because PWR believes this technology could disrupt the economics of today’s hydrogen production market.

PWR approached the Energy & Environmental Research Center (EERC) National Center for Hydrogen Technology[®] (NCHT[®]) to assist in the development, testing, demonstration, and commercialization of this technology because of the EERC’s history of working with hydrogen technologies and bringing new technologies to the commercialization stage.

PWR’s hydrogen production technology represents an alternative technology to conventional SMR. PWR’s technology applies process intensification concepts to arrive at a proprietary regenerative sorbent process that is capable of producing industrially useful quantities of hydrogen and that possesses significant advantages over conventional SMR technology. These advantages include the following:

- Compact design – The PWR technology provides a 90% size reduction compared to a conventional SMR-fired box.
- Process integration/multifunctional reactor – The PWR hydrogen generator eliminates the complexity and costs of separate shift reactors.
- Modular – PWR generator components can be fabricated remotely from the construction site and then assembled on-site, which offers potential cost reduction and quality improvement.
- Improved efficiency – The PWR technology provides 5%–20% higher hydrogen yields than commercial SMR technology and accomplishes this without the large volume of export steam that is characteristic of conventional SMR technology.
- Less product cleanup – The PWR hydrogen generator potentially produces hydrogen at a higher concentration in the product stream than conventional SMR technology, which substantially reduces purification requirements for the product and, in some applications, might eliminate the need for purification. The effect would be to reduce PSA size or cycle frequency, thus decreasing PSA capital cost or increasing its reliability, or possibly eliminate the PSA completely (PSA elimination would increase hydrogen recovery by a minimum of 10%).

- Reduced greenhouse gas emissions at less cost – The PWR hydrogen generator has the potential to favorably affect carbon dioxide emissions in four ways: 1) its potentially greater efficiency means less energy, hence, fewer carbon dioxide emissions per volume of hydrogen product than conventional SMR; 2) it has the ability to process biologically based feedstocks, which results in a near-neutral carbon dioxide emission situation; 3) the carbon dioxide content of the flue gas is higher than conventional SMR technology and, thus, takes less energy to capture (which reduces capture-related carbon dioxide emissions); and 4) its ability to process biofeedstocks and reduced carbon dioxide capture requirements introduce the opportunity for net negative carbon dioxide emissions. That is, capture and sequestration of biofeedstocks, in effect, transfers carbon dioxide from the atmosphere into geologic storage.
- Lower equipment cost – The above advantages translate into an estimated 30%–40% reduced equipment cost for the PWR hydrogen generator compared to conventional SMR technology.

The PWR hydrogen generator design is expected to provide sorbent-enhanced performance while avoiding issues experienced by some other sorbent-enhanced reforming technologies. Additionally, PWR's hydrogen generator will provide improved performance without the need for expensive air separation units or oxygen carriers and will convert feeds other than natural gas without the addition of a prereformer. The design also promises to be flexible with respect to scale. Thus the PWR hydrogen generator concept overcomes many of the deficiencies of SMR and other hydrogen production technologies.

Year 6 – Objective

The goal of this scope of work was to advance the state of operability of the pilot-scale unit to the point of being able to generate hydrogen continuously for more than 30 hours. The technical team deemed the goal attainable based upon successes in achieving continuous solids flows and operational procedure advances achieved during the past year of development effort under NCHT funding. This was a continuation of previously proposed work.

Year 6 – Tasks

The team was to prepare the pilot-scale test hardware for extended, continuous test operation, with hydrogen production occurring during a majority of the duration of the operations. During previous test efforts, prior to this scope of work, a rotary valve was damaged because of sorbent build-up and scaling. To accomplish the current scope of work, the team proposed to resurface a rotary valve damaged during previous off-nominal operating periods.

Additionally, the team set out to modify operational and start-up procedures, based upon knowledge accumulated during the prior year of development and test efforts. These modified procedures were to be formalized in a detailed operations handbook designed to provide the team with guidance required to start up the unit, engage the unit in hydrogen generation modes, and monitor the unit performance.

Finally, the team proposed to conduct extended, continuous test operations with hydrogen production. Following successful testing, results would be presented at a national hydrogen conference, provided one was available.

This scope of work is a continuation of work performed prior to NCHT Year 6. Therefore, extensive reporting on prior work is presented in the final topical report for this project for NCHT Years 3–5. A new U.S. Department of Energy contract dictated separate reporting for Year 6 alone.

RESULTS AND DISCUSSION

Preparation for Extended Tests

The pilot-scale test unit was successfully prepared for test operations, according to plan. As part of these preparations, a significant rebuilding of the rotary valve below the standpipe needed to be completed. This valve was sent to a trusted machine shop, where the valve was reground to specified dimensions. Sufficient hardfacing on the rotor and the body of the valve existed after the resurfacing to allow its use without a reapplication of hardfacing. Hence, this task proved simpler than planned and allowed the team to proceed into test operations.

Modification of Start-Up and Operational Procedures

Significant effort was put into formalizing start-up procedures, operating procedures, and system performance-monitoring procedures. This is worth noting in this topical report not only because of the effort put into it, but also because the exercise forced the technical team to fully debate details of operations not focused on previously. The pilot-scale system has proven much more temperamental to operate than originally expected at the inception of the project in 2005. Therefore, strict procedures needed to be written and followed to enable tests to be conducted long enough to explore process phenomena without tripping on hurdles associated with simple mechanical operation.

It is fair to state that prior to this extensive effort, adequate knowledge of pilot unit operation did not exist within the team. It was only through repeated exploration of system performance that this knowledge can now be captured in detailed operations documents. These scripts were critical in achieving the repeatable results experienced in pilot unit test operations during this year of the project.

The end result of this activity is a 120-page document that summarizes all operations knowledge gained to date in the form of a script to follow to effectively and efficiently put the unit into hydrogen production mode and maintain operation there for extended periods of time. The team will continue to modify this document as needed, but it now is in a state that could allow an operator with relatively little knowledge of the unit to operate it successfully.

Extended Test Operations

Two extended test operations were completed in December 2010. These test runs were completed during the periods of 11/29/10–12/3/10 and 12/4/10–12/8/10. These runs served to prove a repeatable degradation of catalyst performance. The test runs resulted in the collection of some of the most meaningful process data to date in this development program. The usefulness of the analysis of the results from the tests funded by Year 6 funding was to provide multiple data sets that lend insight into the causes of catalyst performance degradation.

Figure 2 shows a summary of the 13-hr hydrogen production period from the first December 2010 test (12/5/10). In this graph, it is evident that both CO₂ and H₂ production fell off near the end of the hydrogen production period. This is indicative of a progressive catalyst failure that causes the reactions in Equations 1 and 6 to yield progressively less product gases with time.

This degradation of performance was also noted previously in July 2010. Subsequent testing in the 12/4/10–12/8/10 timeframe yielded very similar results within a slightly shorter period of time (5 hr). It is currently believed that the root causes of catalyst degradation are much more sensitive to temperature than previously understood. In fact, as a result of these tests, the operational procedure for the tests has been refined to narrow the temperature range of the hydrogen reactor and decrease the mean temperature of the hydrogen reactor.

Figure 2. Hydrogen production, 12/4/10–12/5/10 (nitrogen-free basis).

Figure 3 summarizes the range of temperatures experienced in the hydrogen reactor over the course of several tests. These data have helped the team to define the temperatures at which the hydrogen reactor will perform best. It was once believed that more hydrogen yield would be observed near 1300°F, but the team now believes that the unit will produce more hydrogen at approximately 1150°F.

Additional work performed after Year 6 focused on discovering the exact root causes of the catalyst deactivation. The data generated during the Year 6 tests simply helped the team understand at which conditions the reaction proceeds better.

Presentation of Results

In the originally proposed scope of work, it was stated that results from this project would be presented at a national hydrogen conference or other suitable venue. A number of circumstances prevented this. First, a clear defocusing on the topic of hydrogen occurred at the federal level. This resulted in fewer hydrogen conferences being available, compared to previous years.

A conference to present results was identified in early 2011. This was the 2011 Council for Hydrogen Energy Implementation Conference (planned by the organization that was formerly known as the Mountain States Hydrogen Business Council), to be held at the Kennedy Space Center in Florida. In May 2011, this conference was cancelled, leaving the team again looking

Figure 3. Tests with catalyst failures.

for another pertinent conference at which it could present results before the contract expired at the end of October 2011. Another conference possibility that looked promising was the California Hydrogen Business Council event to be held on October 13 in Oakland, California. As of the date of this report, that conference has not yet been fully planned. No other conference target was found, so this objective was not completed.

CONCLUSIONS

Year 6 NCHT funding allowed the project team to conduct two additional pilot-scale hydrogen production tests to verify and explore a previous observation of decreasing hydrogen yield with time. This behavior was repeated twice with similar shapes in the decreasing yield curve. These data will provide the team with information necessary to determine the root causes of the catalyst deactivation and to redefine pilot-scale unit design and operational procedures to preclude this catalyst deactivation.

The team had an objective of achieving 30 hours of continuous hydrogen production was not achieved because of the repeatable catalyst deactivation. This is the first point in the development program at which pilot-scale unit operations have advanced to the point where this behavior could be observed in a focused, exploratory method. Although the objective was not reached, the team viewed this as a partial success because the process itself is now being evaluated, rather than the process support mechanisms. Additional work is now being carried out under separate funding sources.

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