Integrated Operation of the INL Hytest System and High-Temperature Steam Electrolysis for Synthetic Natural Gas Production

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INTEGRATED OPERATION OF THE INL HYTEST SYSTEM AND HIGH-TEMPERATURE STEAM ELECTROLYSIS FOR SYNTHETIC NATURAL GAS PRODUCTION

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The primary feedstock for synthetic fuel production is syngas, a mixture of carbon monoxide and hydrogen. Current hydrogen production technologies rely upon fossil fuels and produce significant quantities of greenhouse gases as a byproduct. This is not a sustainable means of satisfying future hydrogen demands, given the current projections for conventional world oil production and future targets for carbon emissions. For the past six years, the Idaho National Laboratory has been investigating the use of high-temperature steam electrolysis (HTSE) to produce the hydrogen feedstock required for synthetic fuel production. High-temperature electrolysis water-splitting technology, combined with non-carbon-emitting energy sources, can provide a sustainable, environmentally-friendly means of large-scale hydrogen production. Additionally, laboratory facilities are being developed at the INL for testing hybrid energy systems composed of several tightly-coupled chemical processes (HYTEST program). The first such test involved the coupling of HTSE, CO2 separation membrane, reverse shift reaction, and methanation reaction to demonstrate synthetic natural gas production from a feedstock of water and either CO or a simulated flue gas containing CO2. This paper will introduce the initial HTSE and HYTEST testing facilities, overall coupling of the technologies, testing results, and future plans.

I. INTRODUCTION

Although global oil demand decreased in 2008 and 2009, demand is expected to once again grow in 2010[1]. However, some researchers believe that world oil production capacity may have already peaked in early 2008 and is declining [2]. Also, the use of liquid hydrocarbon fuels results in significant greenhouse gas emissions and contributes to global warming. A hydrogen-based energy economy has been proposed, with goals of reduced oil consumption and reduced greenhouse gas emissions. However, the conversion process to a hydrogen-based energy economy will require decades due to the tremendous infrastructure already in place related to liquid hydrocarbons.

Synthetically-derived hydrocarbon fuels (synfuels) can represent an interim solution and a bridge to a future hydrogen economy. Synfuel production is a mature technology, and requires syngas – a mixture of hydrogen (H2) and carbon monoxide (CO) – as a feedstock. Traditionally, syngas and synfuel have been produced via coal gasification and partial oxidation of natural gas. Synfuel production from coal gasification follows the following simplified series of reactions [3,4]:

Gasification: \( 2C + 1/2O_2 + H_2O \rightarrow 2CO + H_2 \) (1)
Water gas shift \( CO + H_2O \rightarrow H_2 + CO_2 \) (2)
F-T reaction \( CO + 2H_2 \rightarrow CH_4 + H_2O \) (3)
Net reaction \( 2C + H_2O + 1/2O_2 \rightarrow CH_4 + CO_2 \) (4)

As is noted in [3,4], the actual series of reactions is much more complex. Similarly, synfuel production via partial oxidation (POX) of natural gas follows the following simplified path:

POX \( CH_4 + 1/2O_2 \rightarrow CO + 2H_2 \) (5)
F-T reaction \( CO + 2H_2 \rightarrow CH_4 + H_2O \) (6)
Net reaction \( 2C + H_2O + 1/2O_2 \rightarrow CH_4 + CO_2 \) (7)

Steam methane reforming produces a syngas with a H2 / CO ratio much higher than 2 and is therefore not ideally suited for producing synfuels. There are several things to note about the above synfuel production techniques. First, in the case of coal gasification, to obtain the required H2 / CO ratio of 2, additional H2 is required. This is provided via the water gas shift reaction at the expense of CO2 production. Second, both schemes consume fossil fuels. Finally, both processes result in the production of the greenhouse gas CO2.

A sustainable and more environmentally friendly method of synfuel production entails using captured CO2.
as the carbon source and water splitting to supply hydrogen [3,4]:

Water splitting  
$$3H_2O \rightarrow 3H_2 + 3/2O_2$$  (8)

Reverse water gas shift:  
$$CO_2 + H_2 \rightarrow CO + H_2O$$  (9)

F-T reaction  
$$CO + 2H_2 \rightarrow CH_2 + H_2O$$  (10)

Net reaction  
$$CO_2 + H_2O \rightarrow CH_2 + 3/2O_2.$$  (11)

In this reaction scheme, the greenhouse gas CO$_2$ is consumed rather than produced and no fossil fuels are consumed.

The reaction pathway proposed above relies upon a suitable renewable energy powered and/or nuclear powered water-splitting technologies for carbon-free sustainable hydrogen production. The Idaho National Laboratory (INL) has been researching the use of solid-oxide fuel cell technology to electrolyze steam for large-scale nuclear-powered hydrogen production. The scope of activities under the INL High Temperature Steam Electrolysis (HTSE) program includes computational fluid dynamics modeling, process flow sheet analyses, and experimental testing. Scales of experimental testing extend from ~2 watt button cell tests to 15 kW multi-stack tests.

Parallel to the INL high temperature electrolysis activities, the INL Energy Security Initiative has been established to help achieve energy security for the United States. The Energy Security Initiative studies hybrid energy systems that are based upon tightly-coupled chemical processes, more efficient reactor designs, improved or new heat transfer modes, new materials, and new measurement / control systems.

Due to the difficulty of small scale Fischer-Tropsch processing, it was decided to postpone Fischer-Tropsch studies and instead produce synthetic methane as Phase I of the INL Energy Security Initiative:

Water splitting  
$$4H_2O \rightarrow 4H_2 + 2O_2$$  (12)

Reverse water gas shift:  
$$CO_2 + H_2 \rightarrow CO + H_2O$$  (13)

Methanation reaction  
$$CO + 3H_2 \rightarrow CH_4 + H_2O$$  (14)

Net reaction  
$$CO_2 + 2H_2O \rightarrow CH_4 + 2O_2.$$  (15)

An existing high temperature electrolysis testing apparatus was coupled to a new reverse shift reactor and methanation reactor to produce synthetic natural gas via the above reaction pathway (Eqs. 12-15). As part of a 2500 hour long-duration HTSE test, 60 hours of integrated tests were conducted. All hydrogen was provided via HTSE. CO$_2$ was separated from a simulated flue gas stack (circa 15% CO$_2$ in humid nitrogen) using a nitrogen purification membrane and directed to the shift reactor where CO was produced using the hydrogen supplied by HTSE. The resulting syngas mixture continued to a methanation reactor. Measurements of the product gas composition were taken with a four-channel micro-GC.
hydrogen gases while continuing to operate near atmospheric pressure. The flow rates of nitrogen, hydrogen, and air are established by means of precision mass-flow controllers. Air flow to the stack is supplied by the shop air system, after passing through a two-stage extractor / dryer unit.

Downstream of the mass-flow controllers, nitrogen is mixed with hydrogen gas. Hydrogen is included in the inlet flow as a reducing gas in order to prevent oxidation of the Nickel cermet electrode material. The nitrogen / hydrogen gas mixture is mixed with steam by means of a heated water bath. The humidifier water temperature is maintained at a constant setpoint value using computerized feedback control. The dewpoint temperature of the nitrogen / hydrogen / steam gas mixture exiting the humidifier is monitored continuously using a precision dewpoint sensor. Pressure is also measured at the dewpoint measurement stations using absolute pressure transducers. Local stream pressure information is required to determine the mole fraction of steam in the gas mixture at the dew point measurement station. Since the nitrogen and hydrogen flow rates are fixed by the mass flow controllers, and the steam partial pressure is fixed by the bath temperature, the complete inlet gas composition is precisely known at all times. All gas lines located downstream of the humidifier are heat-traced in order to prevent steam condensation. Gas line temperatures are monitored by thermocouples and controlled by means of computer-controlled SCRs.

The electrolysis product stream exiting the furnace is directed towards a second dewpoint sensor upon exiting the furnace and then to a condenser through a heat-traced line. The condenser removes most of the residual steam from the exhaust. The final exhaust stream is vented outside the laboratory through the roof.

The rate of steam electrolysis is measured via two different, independent methods: 1) electrical current through the stack, and 2) the measured change in inlet and outlet steam concentrations as measured by the on-line dew point sensors.

The inlet gas mixture is directed to the high temperature furnace, which heats and maintains the electrolyzer at the appropriate test temperature via computer-based feedback control. The furnace also preheats the inlet gas mixture and the air sweep gas. A photograph of a 10 cm x 10 cm 10 cell stack, mounted on its inconel test fixture and resting on the furnace base, is shown in Fig. 3. The stacks were fabricated by Ceramatec, Inc., of Salt Lake City, UT. The stacks have a per-cell active area of 64 cm² (10 cm x 10 cm cells) for a total active area of 640 cm².

I.B. Electrolysis Stack

Planar stacks used for testing by the INL are fabricated by Ceramatec, Inc., of Salt Lake City, UT. The internal components of a typical stack are shown in Fig. 4 and are comprised as follows. The interconnect plate is fabricated primarily from ferritic stainless steel. It includes an impermeable separator plate (~0.46 mm thick) with edge rails and two corrugated flow fields, one on the sweep-gas side and one on the steam / hydrogen side. The height of the flow fields is 1.0 mm. Each flow field includes 32 perforated flow channels across its width to provide uniform gas-flow distribution. The steam / hydrogen flow fields are fabricated from nickel foil. The air-sweep flow fields are made from ferritic stainless steel. The interconnect plates and flow fields also serve as electrical conductors and current distributors. To
improve performance, the sweep-side separator plates and flow fields treated with a spinel coating. On the steam / hydrogen side of the separator plate, a thin (~10 μm) nickel metal coating is applied.

The electrolyte is partially-scandia-stabilized zirconia, ~140 μm thick. The steam / hydrogen electrode (cathode in the electrolysis mode) is graded, with a nickel cermet layer (~13 μm) immediately adjacent to the electrolyte and a pure nickel outer layer (~40 μm).

The sweep-side or oxygen-side electrode (anode in the electrolysis mode) and bond layer are composed of cobalt ferrite.

I.C. INL Energy Security Initiative Phase I Facility

The HYTEST Phase I setup was constructed adjacent to the HTSE test facility described above. The INL Energy Security Initiative Phase I facility (hereafter referred to as Phase I) was designed to couple the HTSE testing operations with reactor components in order to use the hydrogen produced by ongoing HTSE testing. In this manner, the quality and dynamic production of hydrogen could be investigated concurrently with Phase I activities that address: 1) feedstock conversion, 2) energy integration, 3) energy storage and product synthesis, 4) by-product management (i.e. CO₂ capture, recycle, and reduction to fuels and chemicals), and 5) system simulation and process monitoring and control.

A photograph of the Phase I setup is included in Fig. 5. This setup integrates four reactors: 1) a 10 cm x 10 cm x 10 cell HTSE stack (described above), 2) an heated tubular reactor capable of being heated to 1200°C to perform steam-methane reforming and reverse-shifting of CO₂ with H₂, 3) a high-pressure fixed-bed catalytic synthetic fuels reactor, and 4) a CO₂-separation membrane reactor. The reactors are set up for integrated testing, including recycle of the streams necessary to attain high conversion efficiency and to investigate system dynamics and stability. The entire process setup (not including the HTSE stack) is contained in a transportable enclosure that provides access to components through Lexan-Glass™ windows on each side of the module. Electrical power is supplied through a single power cord (208/120 volt, 100 amp), which can be disconnected and locked-out for electrical installations and maintenance. The module is ventilated with a variable-speed blower mounted on the building roof. Airflow checks indicated that face velocities greater 100 ft/s can be achieved, even when two windows are open at medium fan speeds (about 1,500 scfm volumetric flow). An air damper is installed on the module to allow for up to 2,500 scfm of airflow. The multiple windows and damper provide excellent air draft throughout the enclosure with the ability to achieve 3 to 6 air turnovers per minute.

Figure 5. Photograph of INL Energy Security Initiative Phase I experimental facility.

The Phase I process diagram is found in Fig. 6. The process system is mainly constructed of stainless steel vessels and tubing with typical Swagelok fittings. Flow is established by applying positive pressures of less than 30 psig to electronic gas mass flow controllers, which regulate the total flow through the methanation reactor at approximately 0.25-5.0 slpm.

Compressed gas mixtures supply reactant CO, CO₂, CH₄ and diluent/purge N₂. After system checkout, all of the test hydrogen was supplied by the coupled HTSE reactor. A hydrogen pump is used to compress the HTSE-produced hydrogen to 45 psig. The pump is controlled electronically to maintain the hydrogen tank pressure between 35 psig and 45 psig. Hydrogen make-up gas from compressed gas cylinders can be provided, but was not necessary for the current tests. Spreadsheet calculations were used to determine the flow rates of individual reactants to attain the desired operating conditions and reactant concentrations.

Reactant and recycle gas compositions are achieved by metering gases in the proper ratios with calibrated mass-flow controllers and verified by on-line sample collection and monitoring at the points marked in the process diagram (Fig. 6). Stream flow rates, compositions, pressure, and temperature conditions are continuously monitored and controlled to ensure safe conditions and proper flow balances. Automatic shutoff solenoid-actuated valves are installed in the system in the event that reactor temperatures fall below safe limits. This feature is incorporated to prevent the formation of nickel carbonyls or hazardous gas leaks into the general occupancy area of the lab.

Steam is introduced into the system using a water evaporation flash pot. A metering pump injects a controlled amount of water semi-continuously into the heated steam generator vessel through a ¾” stainless steel tube. The water vaporized in the steam generator discharges into the main gas line downstream of the gas pre-heater. A 2” dip tube directs the water feed flow.
towards the bottom of the vessel. A stainless steel mesh is placed in the vessel to disengage moisture droplets ejected from the boiling film of water. A thermocouple measures the interior temperature of the steam generator.

Carbon dioxide was separated from a simulated flue gas stack (approximately 15% CO₂ in nitrogen) using a nitrogen purification membrane (Fig. 7). The separated CO₂ was diverted to the shift reactor, where it was reverse-shifted to carbon monoxide (CO) using hydrogen supplied by HTSE. The resulting CO-H₂ syngas mixture was fed to the methanation reactor.

The methanation reactor is a High Pressure Equipment Company MS-19 Micro Series Reactor with an effective height of 20 inches and a 9/16" internal diameter. This tubular micro-reactor is fabricated from 316 SST with a design pressure rating of 1200 psi at 450°C. The reactor was charged with approximately 70 g of pelletized catalyst, supported over a stainless steel support screen. Process temperatures are continuously monitored by three evenly-spaced thermocouples near the top, middle, and end of the catalyst bed. Feedback-controlled band heaters are used to maintain the reactor temperatures. Six 1-½" wide, 1" ID Watlow™ mineral-insulted band heaters are evenly spaced along the length of the reactor barrel. For the current tests, the reactor inlet and interior temperatures were maintained above 250°C to prevent possible formation of nickel.
tetracarbonyl, Ni(CO)$_4$, which can form at temperatures below approximately 150°C.

Gas compositions were measured using an Agilent 3000 Micro Gas Chromatograph, configured with four channels. The GC is outfitted with two identical mole sieve columns, one Plot Q column, and one Plot U column. Each of the gas species was detected on two or more columns of the GC, providing a useful cross-check of the data. Argon carrier gas was used for all four of the channels in this test. Various GC methods and programs were developed and tested using calibration gases. This made it possible to cover the wide range of concentrations from ppm to over 90 vol.%. Injection times were also varied to enable greater flexibility of range. Instrument calibration was performed weekly, but retention times and response factors remained relatively constant throughout the testing.

Data logging was accomplished with the control program and gas chromatograph. In addition, manual data collection sheets were developed to keep periodic records of temperatures, pressures, flow rates, and gas species measurements.

II. EXPERIMENTAL RESULTS

Results will be discussed roughly in order by which reactants flow through the integrated system: first, high-temperature steam electrolysis; second, CO$_2$ separation membrane; third, reverse-shift reactor; and finally, methanation reactor.

II.A. Electrolysis Stack

Testing commenced with an inlet hydrogen flow rate of 800 sccm, nitrogen flow rate of 1600 sccm, and furnace temperature of 800°C. The inlet dew point temperature was 82°C. This corresponds to an inlet steam mole fraction of 0.6. The theoretical open cell potential for these inlet conditions was calculated to be 0.87 V. The measured open cell potential was 0.86 V.

Fig. 8 presents the results of an initial polarization curve / voltage sweep performed under the above conditions. The voltage was automatically swept from open cell to the thermal neutral voltage of 1.29 volts / cell for the operating temperature of 800°C. The peak current was measured to be 15.1 A, corresponding to a peak current density of 0.24 A/cm$^2$. Measured per-cell area specific resistance (ASR) values were around 2 Ωcm$^2$.

Long-duration steady-state operations at the thermal neutral voltage of 12.9 V began on May 29 and completed 2500 hours at constant input on September 10. A summary graph of the results of the test is shown in Fig. 9. This graph shows the operating voltage, stack current and ASR with data recorded every five minutes. The stack operating conditions were kept constant at the same values as those for the polarization curve test discussed above.

Several events disrupted the steady-state testing. It is not clear whether these events affected the long-term performance of the stack. Most of these disruptions involved interruptions of inlet gas flows (N$_2$ and H$_2$),
caused by problems with the gas mass flow controller electronics. Later in testing (beyond 1200 hours), the gas lines in the gas humidifier would become periodically clogged, also interrupting gas flows to the stack. Apparently the laboratory water demineralization system was not operating correctly. As a result, several analog pressure gauges were installed to monitor the humidifier back pressure such that lines could be replaced before gas flow was interrupted. If blockage of gas flow occurred, the stack would not receive sufficient steam and electrical current would drop drastically due to steam starvation.

During the first 60 hours, the nickel oxide in the hydrogen electrode is being reduced and all of the components of the cell are sintering. The ASR of the stack reached 2.3 $\Omega$-cm$^2$ at 60 hours elapsed test time, at which point it stabilized. Beyond 60 hours elapsed test time, the average rate of degradation was 8.15 % per 1000 hours.

At the conclusion of 2500 hours the stack had a per-cell ASR of 2.75 $\Omega$-cm$^2$. The flow rate of HTSE products was approximately 1400 cm$^3$/min. The hydrogen production rate as measured by the stack current (Faraday’s Law) was 690 sccm (41.4 N-liters/hour, 3.7 g/h). This corresponds to an outlet composition of approximately 49% H$_2$ and 51% N$_2$ by volume. Subsequent gas chromatograph analysis indicated the product mixture was closer to 45% H$_2$ and 55% N$_2$ by volume. The stack electrical current was 9.89 A.

II.B. CO$_2$ Separation and Methanation

The product stream from the high temperature steam electrolysis stack was captured, pressurized, and stored in a surge tank. A feedback-controlled hydrogen pump pressurized the surge tank to 35 to 45 psig. The CO$_2$ membrane separation system was run for approximately 8 hours of integrated operations with the HTSE experiment and the high-temperature gas-shift reactor. A model flue gas (15% CO$_2$ in N$_2$) was fed to the membrane system. The retentate (mostly N$_2$) was discharged to the exhaust, and the permeate (concentrated CO$_2$ in N$_2$) was mixed with H$_2$/N$_2$ from the HTSE experiment and forwarded to the gas-shift reactor. Test conditions and results from the membrane experiment are displayed in Table 1.

![Figure 10. Comparison of experimental data and model predictions for membrane CO$_2$ / N$_2$ separation.](image)

Table 1. Membrane test conditions / outlet CO$_2$ concentration.

<table>
<thead>
<tr>
<th>Test</th>
<th>$n_{\text{feed}}$ [mol/s]</th>
<th>$P_{\text{feed}}$ [psig]</th>
<th>$P_{\text{retentate}}$ [psig]</th>
<th>$P_{\text{permeate}}$ [psig]</th>
<th>CO$_2$ mol%</th>
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<tr>
<td>1</td>
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<td>120</td>
<td>117</td>
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<td>120</td>
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<td>8.12</td>
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<td>2.59</td>
<td>140</td>
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<td>160</td>
<td>150</td>
<td>12.8</td>
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</table>

Fig. 10 compares the experimental concentration data with predictions from a membrane model. A description of the membrane computational model can be found in [6]. The mole fraction of CO$_2$ in the permeate stream is plotted against the feed flow rate parameter $n_{\text{feed}}/P_{\text{feed}}$. In these coordinates, the model predictions exhibit nearly linear dependence on the feed flow parameter. This agrees somewhat with the data at low flow rates, but the measurements at high flow rates are sporadic and do not agree well with the model. The disparity is likely due to problems with the experimental setup. The rotameter-type flowmeter that was installed upstream of the membrane unit was inadequate to provide consistent quantification of the flow of feed gas to the membrane. These unreliable measurements were difficult to reconcile with the composition data from the gas chromatograph.

Attempts to perform reverse CO$_2$ shift with hydrogen in a tubular reactor were partially successful. The tubular reactor was charged with a steam methane reforming catalyst. Unfortunately, a small, but significant amount of CO$_2$ was converted to condensed carbon (or coke) on the surfaces of the heated tube. This carbon buildup contributed to problematic catalyst coking. Nonetheless, Fig. 11 and 12 compare measured data with equilibrium conditions for the test to determine the extent that CO$_2$ was converted to CO with H$_2$ following the water-gas shift reaction. Excess hydrogen supplied from the HTSE was combined with CO$_2$ in a dry inlet stream. This gave a maximum driving force to convert CO$_2$ to CO. Data were collected as the furnace temperature was increased and then decreased. The flowrate was increased during the temperature descent to investigate the affect of residence time and pressure.

The methanation synfuels reactor was operated in concert with the HTSE experiment for more than 30 hours.
of integrated testing. During methanation testing, a total inlet flow rate of approximately 2 slpm was maintained. Table 2 lists the operating conditions for the methanation parametric studies conducted.

In general, the fractional conversion of CO to CH₄ was greater at higher pressures and lower temperatures. This is consistent with Le Chatelier’s principle for the exothermic methanation reaction where the total number of moles decreases with the extent of reaction. The extent of methanation tended to over 70% conversion regardless of reactor temperature (ranging from 275 to 325 C) or reactor pressure (ranging from 5 to 25 psig). The time to reach steady state, however, did depend on the initial conditions.

### III. CONCLUSIONS

The HYTEST Phase I facility at the INL operated in conjunction with the HTSE experiment for over 60 hours of integrated testing. During operations, the synfuels reactor, CO₂ membrane separator, and high-temperature shift reactor were operated in several configurations to demonstrate various aspects related to hybrid energy systems and synfuels production. This facility represents a major accomplishment within the Energy Security Initiative as it provides a flexible and reconfigurable testbed for laboratory-scale demonstrations of hybrid-energy concepts and technologies.

### ACKNOWLEDGMENTS

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### Table 2. Methanation results

<table>
<thead>
<tr>
<th>Test</th>
<th>T&lt;sub&gt;reactor&lt;/sub&gt; [°C]</th>
<th>P&lt;sub&gt;inlet&lt;/sub&gt; [psig]</th>
<th>9% CO SLPM</th>
<th>45% H₂ SLPM</th>
<th>100% N₂ SLPM</th>
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