

II.I.1 Development of Solar-Powered Thermochemical Production of Hydrogen from Water*

Robert Perret (Primary Contact)
 Alan Weimer, University of Colorado
 Bunsen Wong and Lloyd Brown, General
 Atomics Corporation Richard Diver and
 Nathan Siegel, Sandia National Laboratories
 Michele Lewis, Argonne National Laboratory
 Yitung Chen, University of Nevada, Las Vegas
 (UNLV)

The UNLV Research Foundation
 8311 W. Sunset Road, Suite 200
 Las Vegas, NV 89113
 Phone: (702) 895-2833; Fax: (702) 895-3877
 E-mail: ntsllc@rperret.com

DOE Technology Development Manager:
 Rick Farmer

Phone: (202) 586-1623; Fax: (202) 586-9811
 E-mail: Richard.Farmer@ee.doe.gov

DOE Project Officer: Paul Bakke
 Phone: (303) 275-4916; Fax: (303) 275-4753
 E-mail: Paul.Bakke@go.doe.gov

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Subcontractors:

- General Atomics Corporation, San Diego, CA
- University of Nevada, Las Vegas, Las Vegas, NV
- University of Colorado, Boulder, CO
- Sandia National Laboratories (SNL), Albuquerque, NM
- National Renewable Energy Laboratory (NREL), Golden, CO
- Argonne National Laboratory (ANL), DuPage County, IL

Project Start Date: June 25, 2003
 Project End Date: November 30, 2008

*Congressionally directed project

Objectives

- Quantify cycle thermodynamics, reactant/product equilibrium quantities for each cycle step, cycle step reaction kinetics for current thermochemical cycles:
 - Zinc oxide
 - Cadmium oxide
 - Copper chloride
 - Cobalt ferrite
 - Sodium manganese (addressed under separate funding and reporting)

- Optimize designs, processes and flow sheet performance to estimate minimum hydrogen cost for selected cycles.
- Test and evaluate a solid particle receiver concept for implementation with solar-powered thermochemical cycles.
- Design and construct a secondary concentrator and integrate with multiple reactor tubes for on-sun zinc oxide testing.
- Evaluate benefits of advanced heliostat research and development to reduce costs of solar-powered hydrogen.
- Establish comparative cost effectiveness of feasible thermochemical cycles using the H2A spreadsheet.
- Ultimately, demonstrate integrated pilot plant designs, including on-sun testing for up to three competitive cycles.

Technical Barriers

This project addresses the following technical barriers from the Hydrogen Production section of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

- (AU) High-Temperature Thermochemical Technology
- (AV) High-Temperature Robust Materials
- (AW) Concentrated Solar Energy Capital Cost
- (AX) Coupling Concentrated Solar Energy and Thermochemical Cycles

Technical Targets

Solar-Driven High-Temperature Thermochemical Hydrogen Production

Characteristics	Units	2008 Target	2012 Target	2017 Target
Solar Thermochemical Hydrogen Cost	\$/gge H ₂	10	6	3
Installed Heliostat Capital Cost	\$/m ²	180	140	80
Process Energy Efficiency	%	25	30	>35

gge: gasoline gallon equivalent

Process Energy Efficiency

- All cycles being studied presently have estimated process thermal efficiencies higher than 30%

(lower heating value, LHV). One process under investigation is estimated to provide process thermal efficiency of 59% (LHV).

Solar Thermal Capital Installed Cost

- The primary contributor to solar thermal capital cost is in installed heliostats. The project is assessing current state of the technology to identify research and development to provide significant cost reduction in heliostat installed capital cost.

Solar Thermochemical Hydrogen Cost

- Estimated 2015 costs of hydrogen from the Hybrid Sulfur cycle are \$3.90-4.90/gge in 2015.
- Estimated 2015 costs of hydrogen from the Zinc Oxide cycle are \$4.60-6.60/gge.
- Estimated 2015 costs of hydrogen from the Copper Chloride cycle are \$4.00-5.10/gge.
- Additional cycle research, process and plant optimization are necessary and are being pursued to establish confidence in these cost estimates.

Table 1 summarizes the most recent projections for the hydrogen production costs of several cycles (at the plant gate) relative to the DOE cost target of \$3.00/gge:

TABLE 1. Current Hydrogen Production Cost Estimates (based on H2A Central Production Tool)

Cycle	2015 Cost*	2025 Cost*	Comments/Key Issues
Cd/CdO	Under revision	Not available	Cycle undergoing major revisions
CuCl	\$4.30 (\$3.98 - \$5.07)	\$2.82 (\$2.65 - \$3.35)	Electrolyzer cost highly uncertain
Ferrite	\$5.52 (No sensitivity)	Not available	Very preliminary design and cost analysis
Hybrid Sulfur	\$4.37 (\$3.86 - \$4.89)	\$2.91 (\$2.46 - \$3.21)	Solar electric cost important
Zn /ZnO	\$5.07 (\$4.58 - \$6.53)	\$3.62 (\$3.12 - \$4.87)	Solar field + receiver cost and performance

*Sensitivity range shown in parentheses

Accomplishments

- H2A cost estimates have been generated for all but one solar thermochemical to hydrogen (STCH) cycles.
- With one cycle yet to report, cost estimates for all current STCH cycles under consideration are less than the DOE 2008 target cost.
- Participated in the International Energy Agency Task 25 meetings in Germany and San Diego, CA.

Zinc Oxide

- An experimental investigation of the thermal dissociation of ZnO particles (1st step) in aerosol flow was completed.
- The hydrolysis of zinc powder (2nd step) was studied in an aerosol to determine if high conversions are feasible at short residence times and high dispersions.
- Construction and testing of a new multi-tube reflective wall solar reactor. Proceeded with validation and testing of the power and flux profile using the high flux solar furnace (HFSF), capable of producing 2,500 suns concentration, at NREL.
- A new secondary concentrator has been constructed that produces up to 3,000 suns.
- Both ray trace and multi-physics models have been developed to simulate performance of the HFSF test facility.
- Development and testing of a 3-dimensional computational fluid dynamics (CFD) model of the multi-tube aerosol flow reactor using finite volume schemes in the commercial software Fluent is on track.
- Simulations have been completed for both reacting and non-reacting small particles entrained in either a single inert gas or a mixture of gas species and indicated that the aerosol mixture within the flow tubes reached temperatures up to about 1,377-1,477°C.

Ferrites

- Began experiments to evaluate ferrite transport mechanisms and reaction rates.
- Began investigating techniques for fabricating high surface area reactive ferrite structures.
- Completed the construction of the CR5 solar interface for metal oxide cycles.
- Completed an initial H2A assessment of the ferrite cycles based on the CR5 interface.

Cadmium Oxide

- A rotary kiln reactor was designed and constructed to improve the hydrolysis reaction of molten cadmium.
- Design modifications for a thermogravimetric analysis (TGA) were completed to permit measurements of recombination rates during the dissociation of CdO; first measurements show a recombination of 30-40% for temperature in the range 1,000°C to 1,475°C.
- A window-enclosed reactor design was completed for CdO dissociation to prevent loss of Cd vapor; modeling studies addressing effect of negative pressure differential showed that the decomposition

temperature could be reduced from 1,500°C to 1,255°C for operating pressures around 0.1 bar.

- A CdO flowsheet for a 24-hr plant has been developed; the thermal efficiency of the initial flowsheet was found to be 48.3% (LHV); further optimization is expected to increase this predicted efficiency.
- A CdO central plant concept solar field has been developed to make use of a beam-down collector/concentrator to permit ground-level operation of all chemical processes.

Copper Chloride

- The Cu-Cl cycle was selected as the most promising alternative thermochemical cycle under the Nuclear Hydrogen Initiative.
- Designed, fabricated and successfully tested the nebulizer hydrolysis reactor.
- Completed a conceptual process design for the three reaction Cu-Cl cycle.
- Developed an Aspen Plus flowsheet, calculated efficiency and capital costs and completed a preliminary H2A cost analysis.
- Participated in an International Nuclear Energy Research Initiative with the Atomic Energy of Canada Ltd. and four Canadian universities and an informal information exchange with Commissariat à l'Énergie Atomique in France and a Nuclear Energy Research Initiative-Consortium (NERI-C) with three U.S. universities.

Solid Particle Receiver

- Completed the experimental evaluation of the solid particle receiver prototype.
- A new solar ray tracing algorithm has predicted the maximum temperature at the rear inner wall of the solid particle receiver (SPR) in agreement with the experimental results.

Data Base Management

- Maintained the Web-based Solar Hydrogen Generation Research (SHGR) resource management system up-to-date with high availability status and updated quarterly progress reports into the SHGR resource system.
- Generated new cycle ranking tool by integrating Microsoft Excel-based H2A tool with the SHGR resource management system.



Introduction

Hydrogen production by thermo-chemical water splitting is a chemical process that accomplishes the decomposition of water into hydrogen and oxygen using only heat or a combination of heat and electrolysis instead of pure electrolysis. A comparative assessment has evaluated 351 unique cycles and identified 12 potential thermochemical cycles grouped in about four chemical reaction classes for which additional reaction information is required. Detailed engineering analysis of the most competitive system concepts, using literature and laboratory data, will be applied to system design for testing and evaluation. A small-scale demonstration and a pilot plant design for the most competitive system concept will meet the project objective.

Approach

Thermo-chemical cycles were identified through literature search and contributions from cycle originators within and outside the project. Chemical reactions analysis provides the theoretical chemical feasibility of individual cycle steps. An objective scoring process was developed and implemented to select the best, most cost-effective cycles for further study (Figure 1). Laboratory studies of reactant and product concentrations and reaction kinetics validate earlier theoretical predictions and provide necessary data for improved performance assessments and down-selection to a few cycles for demonstration, test and evaluation.

Solar thermal energy for high temperature processes (<1,300°C) is being studied using a SPR concept that accumulates directed solar energy from a field of heliostats. Solar thermal energy for ultra-high temperature processes ($T > 1,300^\circ\text{C}$) is being studied using cavity receiver concepts equipped with a secondary concentrator design. An engineering assessment of current heliostat cost and performance has defined the benefits and research and development options to reduce significantly the costs of heliostat fields and associated towers that represent the primary cost drivers for a solar thermal energy processing system.

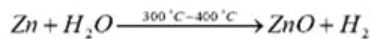
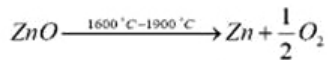
Results

Zinc Oxide

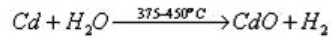
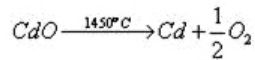
Experimental investigation of the thermal dissociation of ZnO particles (1st step) in aerosol flow was completed. Experiments were performed at temperatures between 1600 and 1,750°C and residence times between 1.11 and 1.78 s. The net conversions after recombination had a maximum value of 18%, with a mean value of 8%. Product particle sizes ranged between 5 nm and 70 nm.

Volatile Metal Oxides

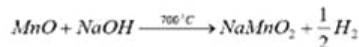
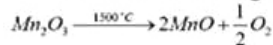
•Zinc oxide



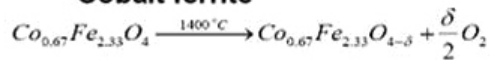
•Cadmium Oxide

Non-volatile Metal Oxides

•Sodium manganese



•Cobalt ferrite

Other

•Hybrid copper chloride

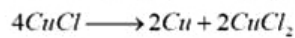
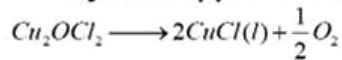
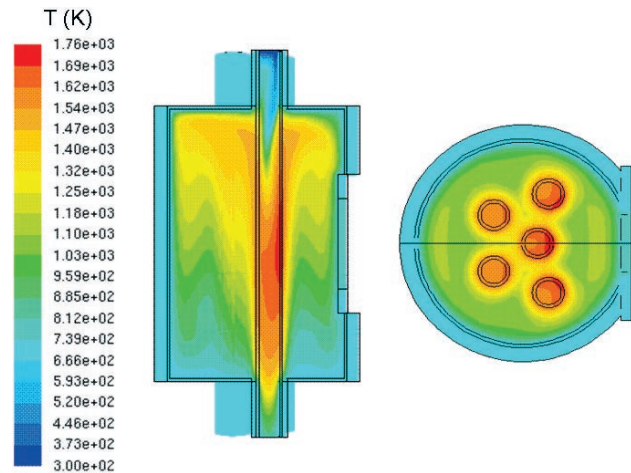


FIGURE 1. STCH Cycles under Investigation

Hydrolysis of zinc powder (2nd step) was studied in an aerosol to determine if high conversions are feasible for short residence times and high dispersions were reacted with water vapor to form hydrogen and zinc oxide in an aerosol flow tube reactor at ambient pressure (82 kPa) between 380 and 540°C and a water concentration of 3%. The highest conversion of 158 nm zinc particles observed in the flow system was about 24% at 540°C and a gas residence time of ~0.6 s. Non-isothermal TGA indicated that complete conversion of zinc to zinc oxide could be achieved with longer residence times.

Solar Cavity Reactor System Experimentation:

Significant progress has been made toward constructing and testing a new multi-tube reflective wall solar reactor. Validation and testing of the power and flux profile from the High Flux Solar Furnace (HFSF) at NREL was done to ensure accurate values for our reactor testing and modeling. The reactor is presently installed at NREL and is being used with an older secondary concentrator capable of producing 2,500 suns concentration. A new secondary has been constructed to provide

FIGURE 2. Temperature Profiles for 1 SLPM N₂ and 1 g/min Alumina Particles per Tube with a Total Solar Input of 14.4 kW

concentrations up to 3,000 suns. Efficiency comparisons will be made between the new and old concentrators.

Solar Receiver Modeling: A three dimensional computational fluid dynamics model of the multi-tube aerosol flow reactor is being developed using finite volume schemes in the commercial software Fluent. Simulations of inert alumina particles entrained in nitrogen gas with a total diffuse solar input of 14.4kW and an assumed external reactor wall temperature of 427°C indicated that the aerosol mixture within the flow tubes reached temperatures up to about 1,377-1,477°C (Figure 2).

Ferrite

Ferrite feasibility work is continuing and is focused in two areas: The evaluation of transport mechanisms and reaction rates for the water oxidation reaction and in the fabrication of high surface area reactive structures. The goal is to use a specialized reactor along with reactive samples prepared with atomic layer deposition to investigate how the water oxidation proceeds and to identify transport limitations due to the development of a passivating layer or other solid-state diffusion mechanism.

CR5 Development

The construction of the CR5 has been completed. The system is currently set for initial on-sun testing in Sandia's solar furnace facility beginning in early August. A recent image of the CR5 system is shown in Figure 3.

Cadmium Oxide

The feasibility of using molten cadmium for hydrolysis was demonstrated in a flow through

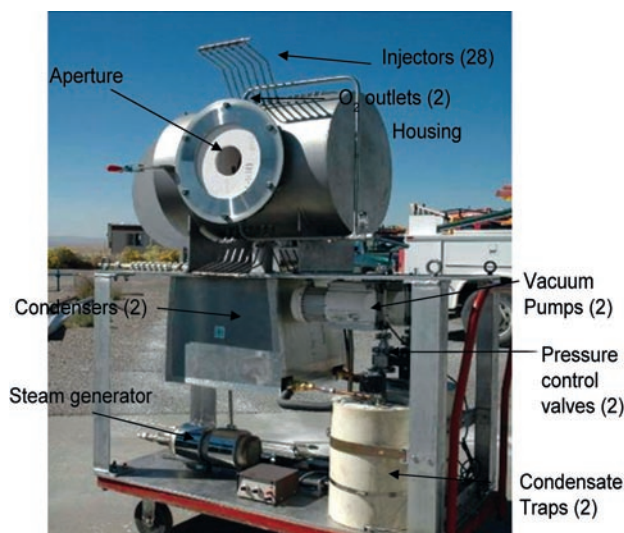


FIGURE 3. The CR5 System

experimental set up in which steam bubbles through a static pool of molten cadmium (Figure 4(a)). The maximum steam to hydrogen conversion rate was measured at 0.7% at around 470°C (Figure 4(b)) which is significantly less than the calculated equilibrium conversion of 5%. A rotary kiln reactor was designed and constructed to enhance the residence time and conversion. Early results show the system can continuously generate hydrogen using molten cadmium.

Post decomposition cadmium vapor quenching is required to limit oxygen recombination. A TGA at UNLV was modified to measure recombination rates at various temperatures. A reaction rate of 30-40% was measured for both zinc and cadmium for in the temperature range between 1,000 and 1,475°C.

A design study concluded that a window-enclosed decomposer using “beam-down” optics is preferred for the cadmium cycle. The window decomposer mixes molten cadmium with the decomposed cadmium and oxygen to carry out the quench (Figure 5). As large windows are not practical, an array of smaller hexagonal window concentrators was conceived. The window enables the use of inert gas and sub-atmospheric pressure to reduce the decomposition temperature. Modeling results shows a mark reduction in CdO decomposition temperature, from 1,500 to 1,255°C at 0.1 bar. Furthermore, using air (10:1 air: Cd) to reduce the cadmium vapor pressure in the decomposed products reduces the decomposition temperature to 1,300°C.

Process flowsheets for the Cd/CdO hydrogen cycle have been developed using Aspen Plus modeling and simulation software along with data from the hydrolysis and recombination experiments. A 24-hour hydrogen plant concept incorporates thermal heat storage using three different media: cadmium at 325°C, cadmium

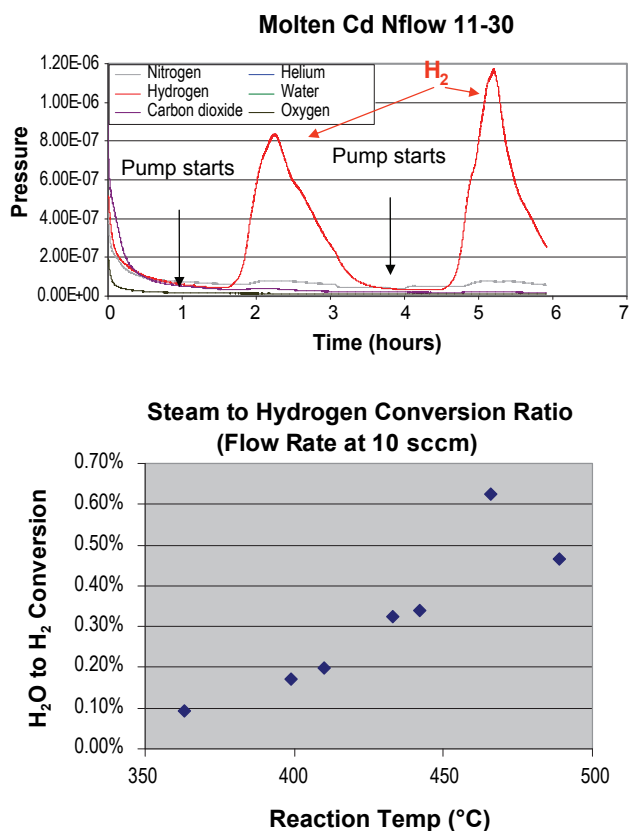


FIGURE 4. Molten Cd Hydrolysis Results

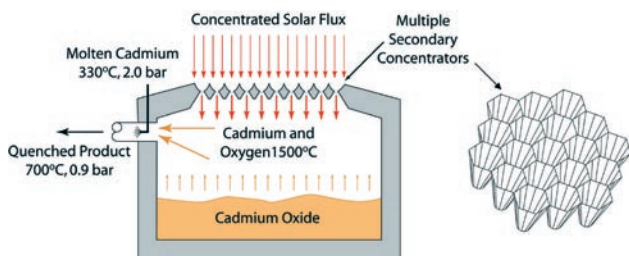


FIGURE 5. Schematic of a Beam-Down Multi-Concentrator Cadmium Decomposer Design

oxide at 325°C or cadmium at 700°C. One part of the plant operates under solar power, and the second part operates during solar off-hours with thermally stored energy. Preliminary calculations predict a thermal efficiency of 48.3% (LHV). The hydrogen plant components have been sized for 100,000 kg/day and the gate-cost of hydrogen was calculated to be around \$4.50/kg based on a 2015 case.

Copper Chloride

Conceptual Process Design: The conceptual process design consists of four unit operations: electrolysis, the crystallizer, hydrolysis, and the oxychloride decomposition reactor.

Electrolyzer: The electrolyzer is based on proton exchange membrane-type fuel cell technology. An anion exchange membrane separates the anode and cathode allowing transfer of chloride ion from the cathode to the anode at 80-100°C and 24 bar. Hydrogen is produced at the cathode from HCl solution at 24 bar. The cathode is flashed to produce a vapor stream consisting of steam and <1 mole% HCl and a liquid stream containing 87 mole% HCl. The flashed acid-rich liquid stream is recycled back to the cathode feed tank. The flashed acid-poor steam stream is used to feed the hydrolysis reactor. CuCl is oxidized at the anode to CuCl₂. The electrolyzer is being developed by the Atomic Energy of Canada Limited and proof-of-concept experiments have been completed.

Crystallizer: The anode effluent is chilled to precipitate the CuCl₂. It may be necessary to use a scrapped surface crystallizer for the cooling step similar to that manufactured by Armstrong Engineering.

Hydrolysis Reactor: CuCl₂ is transferred as slurry at 22 bar to the hydrolysis reactor where it is sprayed into a superheated (400°C) steam environment at 1 bar. The CuCl₂ free jet expands and aspirates the superheated steam into the jet to provide high mass and heat transfer between the CuCl₂ and the steam. The CuCl₂ is converted to Cu₂OCl₂ and HCl. The HCl and unreacted steam exit the hydrolysis reactor to be cooled and are fed to the cathode of the electrolyzer.

One hydrolysis reaction study showed that the HCl content of the feed solution should be as low as possible to obtain high yields of Cu₂OCl₂ and HCl. A second study showed that high steam to CuCl₂ ratios are

required for high yields. This latter result is observed experimentally and is one of the larger inefficiencies in the cycle. Promising results have been obtained with a nebulizer reactor in which very small droplets of CuCl₂ are formed which promote high heat and mass transfer.

Oxychloride Decomposition Reactor: Dry, free flowing solid Cu₂OCl₂ accumulates at the bottom of the hydrolysis reactors and is then transferred to the oxychloride decomposition reactor at 550°C where it decomposes into molten CuCl and oxygen. The heat is recovered from the molten CuCl in a direct heat exchanger where the CuCl is granulated while raising steam. The solidified CuCl is then transferred to the anode feed tank where it is dissolved in an HCl-CuCl₂ solution.

Aspen Plus™ Flowsheet: The current Aspen flowsheet is shown in Figure 6. A CuCl cycle plant producing 125 MT H₂/day requires 210 MW of thermal energy and 87.8 MW of electrical energy and results in an efficiency of about 40-42% (LHV). In conjunction with the Aspen results, a pinch analysis was used to develop an integrated heat exchange network which will effectively recover waste process heat. As with any conceptual process that is proposed with little experimental results and data, a number of key assumptions had to be made. These were:

- Electrolysis reactor operates at:
 - 0.5 volts
 - 500 milliamps/cm²
- The material of construction is porcelain coated carbon steel.

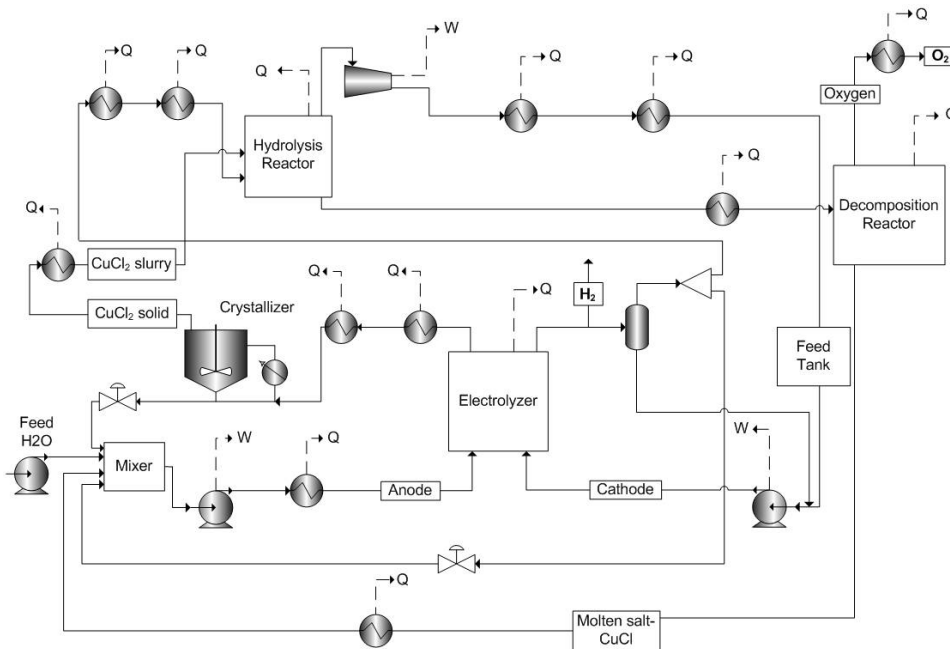


FIGURE 6. Aspen Plus™ Flow Sheet for the Cu-Cl Cycle

- The crystallizer operates with minimal water, HCl and CuCl entrainment.
- In the hydrolysis reactor, copper oxychloride forms rapidly for residence time ~5 sec.
- The sublimation of CuCl in the oxy-decomposition reactor is negligible.
- CuCl_2 will flow from the crystallizer to the hydrolysis reactor under the pressure gradient.
- Ideal separators are used for the entire process.

Key Economic Assumptions: In completing the economic analysis a number of assumptions were made:

- The base year for the economic analysis was 2005 so the Chemical Engineering's Plant Cost Index = 468.
- Plant capacity: 125 MT/day of hydrogen.
- Electrolysis cell cost (vessel and electrodes) was assumed to be \$2,500/m².
- Bulk HCl cost was assumed to be \$241/metric ton.

The preliminary efficiency and hydrogen production costs for 2015 and 2025 are shown in Table 2. For 2025, the hydrogen production costs are \$3.01 per kg and the efficiency is about 42%. We are currently optimizing the process to identify how production costs can be reduced.

TABLE 2. H2A Results for 125,000 kg of Hydrogen/Day Using the Cu-Cl Cycle

	2015	2025
Capital, \$MM		
-Solar	223.4	183.2
-Chemical Plant	134.2	77.3
Utilities, MW	92	82
Efficiency, %	40	42
Cost of Hydrogen, \$/kg	4.38	3.01

Solid Particle Receiver

The SPR is a direct absorption receiver in which solar energy heats a curtain of falling ceramic particle to a temperature in excess of 1,000°C. On-sun testing was conducted in early 2008 at SNL. The test plan included three mass flow rates and three input power levels. Some tests were conducted under conditions where ambient winds exceeded 30 mph.

Tests measured a maximum particle temperature change of 247°C and a maximum receiver efficiency of about 57%. Additional data were also taken for the interior air and wall temperatures of the cavity as well as for particle velocity. The ultimate goal of the SPR test program is to produce a device that is capable of a 400–600°C particle temperature change (perhaps

including recirculation) while exceeding a receiver efficiency of 70%.

Conclusions and Future Directions

Zinc Oxide

Projected costs for the solar-powered zinc oxide thermochemical cycle exceed the 2025 target of \$3.00/gge. Ultra-high temperature for this cycle causes significant radiation loss and accordingly increased solar plant costs. Since the solar plant cost represents the most significant cost for all solar thermochemical cycles, it is concluded that the zinc cycle is unlikely to compete favorably with other options.

Ferrites

The CR5 ferrite reactor is the first of the thermochemical cycles to be tested on-sun in semi-integrated form. Feasibility has been demonstrated, but operational issues and cost characteristics for the CR5 remain to be investigated thoroughly. Effort during the coming year will address these issues by using higher surface area reactive elements by a atomic layer deposition concept.

Cadmium Oxide

Flowsheet models of the CdO thermochemical process continue to promise the highest thermal efficiency of all current cycles under study. Kinetics of the hydrolysis step remain uncertain and this step will continue under investigation during the coming year. Quench studies will continue in attempts to both quantify and resolve the dissociation recombination rate.

A “beam-down” solar field design has been developed, permitting ground-level operation of all chemical processes. More complete analysis of this beam down field and its likely cost will be undertaken during the coming year.

Copper Chloride

The CuCl thermochemical cycle shows lowest estimated cost in 2025 of those solar-powered thermochemical cycles that have reported 2025 estimates. This cycle has the advantage of lowest required temperature for the high temperature step, leading to cost savings for the solar field. It has the disadvantage of being the most complicated cycle of those presently under investigation. Continued development is necessary to resolve whether electrolyzer costs will cancel the advantages of lower temperature operation. Activities will include:

- Optimization of the Cu-Cl cycle through modeling and experiments.

- Development of energy-efficient methods, e.g., membrane distillation for facilitating separations of any entrained HCl or CuCl in the CuCl₂ that is to be transferred to the hydrolysis reactor.
- Replacement of the current nebulizer with an ultrasonic nozzle.
- Scale-up the design for the oxychloride decomposition reactor.

Solid Particle Receiver

Experimental data from the SPR testing will be used to develop and validate computational models to support a conceptual design for a Phase 2 SPR capable of meeting the particle temperature and receiver efficiency goals. It is likely that future SPR development will be sponsored by the Solar Energy Technology Program.

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5. Perkins, C., P.R. Lichty, C. Bingham, and A.W. Weimer, "Effectiveness of a Fluid-wall for Preventing Oxidation in Solar-thermal Dissociation of ZnO," *AIChE Journal*, **53** (7), 1830 (2007).
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7. Lewis, M.A., J.G. Masin, and P.A. O'Hare, Evaluation of alternative thermochemical cycles-Part I The methodology. *Int. J. Hydrogen Energy*, to be published (2008).
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10. Ferrandon, M. S., M., Lewis, M.A., Tatterson, D.F., Nankani, R.V., Kumar, M., Wedgewood, L.E., Nitsche, L.C., *The hybrid Cu-Cl thermochemical cycle. Part I. Conceptual process design and H2A cost analysis. Part II. Limiting the formation of CuCl during hydrolysis*, Extended Abstract submitted for the NHA Annual Hydrogen Conference 2008, Sacramento, CA, March 31-April 4, 2008.
11. Taide Tan, Yitung Chen, Zhuoqi Chen, Nathan Siegel and Gregory J. Kolb, "Wind Effect on the Performance of Solid Particle SOLAR Receivers: Part I- The Models and the Performance without an Aerowindow Protection", *Solar Energy*, (submitted and under reviewing).
12. Taide Tan, Yitung Chen, Zhuoqi Chen, Nathan Siegel and Gregory J. Kolb, "Wind Effect on the Performance of Solid Particle SOLAR Receivers: Part II-The Performance with an Aerowindow Protection," *Solar Energy*, (submitted and under reviewing).
13. Huajun Chen, Yitung Chen, Hsuan-Tsung Hsieh, and Nate Siegel, 2007, "CFD Modeling of Gas Particle Flow within a Solid Particle Solar Receiver," *Journal of Solar Energy Engineering*, Vol. 129, pp. 160-170, May 2007.
14. Taide Tan and Yitung Chen, "Review of Study on Solid Particle Solar Receivers," *Proceedings of the 2nd International Forum on Heat Transfer (IFHT2008-129)*, September 17-19, 2008, Tokyo, Japan (submitted and under reviewing).

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1. Weimer, A.W., "Renewable Fuels via Solar-thermal Processing," Keynote Address presented at the 2008 Solar PACES Biennial Meeting, Las Vegas, NV (March 7, 2008).
2. Weimer, A.W., "Solar-thermal Processing to Split Water," presented at the Department of Chemical and Biological Engineering, Colorado State University, Fort Collins, CO (April 20, 2007).
3. Weimer, A.W., "Solar-thermal Water Splitting Using a Zn/ZnO Thermochemical Cycle," 2007 Inaugural Energy symposium UNLV, Las Vegas, NV (August 16, 2007).
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