

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

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- University of Colorado, Boulder, CO
- Sandia National Laboratories, Albuquerque, NM
- National Renewable Energy Laboratory, Golden, CO
- Argonne National Laboratory, Argonne, IL
- ETH-Zurich, Switzerland
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*Congressionally directed project

- 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:

- (U) High-Temperature Thermochemical Technology
- (V) High-Temperature Robust Materials
- (W) Concentrated Solar Energy Capital Cost
- (X) Coupling Concentrated Solar Energy and Thermochemical Cycles

Technical Targets

TABLE 1. Solar-Driven High-Temperature Thermochemical Hydrogen Production

Characteristics	Units	2006 Target	2012 Target	2017 Target
Solar Thermochemical Hydrogen Cost	\$/gge H ₂	10	6	3
Solar Thermal Capital Installed Cost	\$/m ²	180	140	80
Process Energy Efficiency	%	25	30	>35

Objectives

- Quantify cycle thermodynamics and 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)

Process Energy Efficiency

All cycles being studied presently have estimated process thermal efficiencies higher than 30%. One process under investigation is estimated to provide process thermal efficiency of 59%.

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

Our current estimate for the cost of hydrogen from a solar driven hybrid sulfur cycle is \$3.90-\$4.90/gasoline gallon equivalent (gge). Our current estimate for the cost of hydrogen from the zinc oxide cycle is \$4.60-\$6.85/gge. Additional research as well as process and plant optimization are being done to confirm these early cost estimates and to develop improved technology to meet the DOE targets.

Accomplishments

- Zinc oxide
 - Determined that thermal ZnO dissociation follows L'vov diffusion controlled kinetic expression.
 - Developed global rate expression for Zn hydrolysis.
 - Achieved record Zn yield of 36% (after recombination) in aerosol configuration.
 - Validated computational fluid dynamics (CFD) computer simulations of reactors and showed rapid forward reaction is possible
- Cadmium oxide
 - Developed understanding of the reaction path for hydrolysis with solid cadmium using carbon dioxide.
 - Demonstrated hydrolysis using molten cadmium and steam in a flow system.
 - Measured the effects of different gas environments on cadmium oxide decomposition temperature.
 - Constructed a system to study the recombination kinetics of metal vapor and oxygen after decomposition.
- Copper chloride
 - Developed process flow diagrams and the physical properties database for the Aspen Plus™ simulation.

- Measured thermodynamic data, shown to be critically needed from sensitivity analyses.
- Initiated collaborations with Atomic Energy of Canada, Ltd. (AECL) and the University of Ontario - Institute of Technology.

- Cobalt ferrite
 - Performed several consecutive hydrogen production cycles on-sun using cobalt-ferrite reactive materials.
 - Began construction of the CR5 solar interface for metal oxide cycles.
- Solid particle receiver
 - Began construction (nearing completion) of the solid particle receiver prototype.
 - Conducted initial cold flow stability testing of the solid particle receiver.
- Advanced heliostat
 - Hosted heliostat cost reduction workshop and identified a research and development plan to achieve substantial cost reductions.



Introduction

Hydrogen production by thermo-chemical water splitting, 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, meets the goals for hydrogen production using only water and clean renewable solar energy. A comparative assessment has assessed 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. 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. For hybrid cycles, electro-chemical cell designs are reviewed and modeled for ideal reactions, equilibrium reactions and for reactions incorporating process kinetics. Various electrolyte solutions are explored in the laboratory to optimize the electro-chemical process. Chemical engineering system modeling software is applied to quantify thermal efficiency of conceptual flow charting of the most competitive thermo-chemical cycles, and plant design software is applied to provide capital cost estimates for the engineered thermo-chemical concept design. These systems studies provide the process thermal efficiency for later use in determining system efficiency, as well as early estimates of cycles specific hydrogen costs.

Solar thermal energy for high temperature processes ($T < 1,300^{\circ}\text{C}$) is being studied using a solid particle receiver (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 will define the benefits and research and development options for reducing significantly the costs of heliostat fields and associated towers that represent the primary cost drivers for a solar thermal energy processing system. Capital and operating cost estimates developed from study products are used to identify the most attractive thermo-chemical cycles for test and evaluation in integrated closed-loop, bench-scale tests. Subsequent to successful integrated bench-scale, closed-loop demonstrations, earlier capital and operating costs will be refined and a “go/no-go” decision, based on economic potential, will be made regarding whether or not to proceed to pilot plant design and implementation planning.

Results

Zinc Oxide

Carefully designed experiments were performed to determine the rates of thermal ZnO dissociation and Zn hydrolysis. Thermogravimetric experiments were used to establish a rate law for each reaction; aerosol experiments tested the applied model in an environment where diffusion controls were relatively unimportant. CFD simulations were performed with the commercial finite element code COMSOL Multiphysics. The simulations incorporated the kinetic models found from experimentation as well as a participating media approach for radiation heat transfer to the particulate phase.

ZnO dissociation was shown to follow a L'vov style rate model, which includes an Arrhenius temperature

dependence coupled with rate control by diffusion away from the reacting surface. The activation energy for this reaction was $356 \pm 25.9 \text{ kJ mol}^{-1}$; the pre-exponential factor depended on diffusion resistance for transport away from the particle surface. This led to the prediction that reaction in a dispersed aerosol reactor would be much faster than in a stationary (e.g. thermogravimetric analyzer) configuration. This was borne out by experiment, where aerosol rates were three orders of magnitude greater than in the stationary cases. In the aerosol case, quenching was more rapid, leading to a lower degree of recombination than has yet been reported in the literature. The net yield of Zn was ~36%, very high when compared to the previous record of 1%. It was postulated that better dispersion of the feed powder may be able to increase the yield. The aerosol reactor configuration also generated extremely small particles (10-50 nm) due to limited opportunity for growth during quenching (Figure 1). This is important when considered in light of the results of the Zn hydrolysis kinetics experiments, where it was shown that the reaction only occurs rapidly on the Zn particle surface. Small particles have a high specific surface area, and thus are advantageous to use in the hydrogen generation step of the Zn/ZnO cycle. The Zn hydrolysis kinetics experiments also showed that reaction rates were faster in aerosol configurations than in stationary configurations, indicating the importance of diffusion control for that reaction. The results of the aerosol ZnO dissociation experiments were incorporated into a CFD model for this reaction. The model predicted rapid heating of the particles and rapid reaction, but

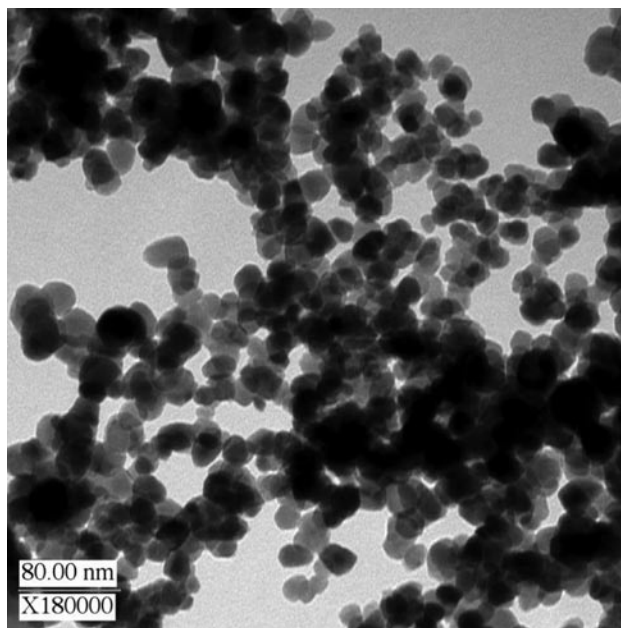


FIGURE 1. Transmission Electron Micrograph of Zn Particles Collected from Aerosol Reactor Operating at $1,750^{\circ}\text{C}$ (180,000X)

showed that oxidation sensitive reactor walls would quickly corrode due to exposure to reaction products. A “fluid-wall” diffusion barrier was unable to prevent wall corrosion leading to emphasis on discovery of high temperature oxidation resistant materials.

Cadmium Oxide

Hydrolysis of solid cadmium requires continuous refreshment of clean metal surface because of the formation of a passivating hydroxide layer. A combination of milling and use of carbon dioxide for chemical removal of the hydroxide layer was found effective. The carbon dioxide generates a porous cadmium carbonate interface, exposing underlying fresh cadmium for hydrolysis as shown in Figure 2a. Cadmium carbonate crystals were found to break off the surface as the layer grows (Figure 2b).

Hydrolysis of molten cadmium was accomplished by passing a steam saturated carrier gas through the melt. This approach has the advantage of self-generated fresh

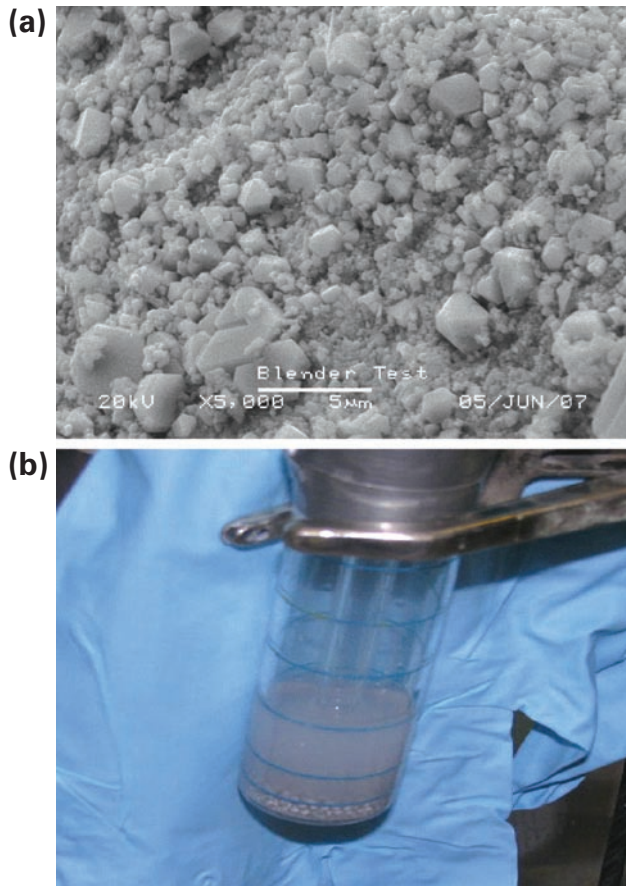


FIGURE 2. (a) Surface of cadmium pellet after sitting in a water bath with carbon dioxide bubbling through it. It is decorated with cadmium carbonate crystals. (b) Cloudy suspension in the bubble bath due to cadmium carbonate crystals that have broken off the cadmium pellet surface.

molten metal surface and is being studied for optimum operating conditions to maximize hydrogen production rates. Preliminary results found that the amount of hydrogen generated was greater than predicted. Results to date show (Figure 3) a steady state conversion efficiency of water vapor passing through the melt to hydrogen between 15% and 20%, but it is anticipated that the rate will improve upon maximizing the contact area between the metal and steam. New experiments are in progress in which pure steam is used, dispensing with the carrier gas. Rates of CdO formation will be measured. Present results show that the cadmium oxide particles formed by the molten hydrolysis process are about 0.5 µm in diameter.

Decomposition of cadmium oxide will be quantified using a thermal gravimetric analyzer. Preliminary experiments have been used to test system modifications to permit quantification of decomposition kinetics as influenced by particle size, residence time, and the thermal and chemical environment. This work is also useful for designing a prototype decomposer for testing. Preliminary tests show that the reaction completion temperature varies from 1,300°C in air to 1,640°C in oxygen (Figure 4).

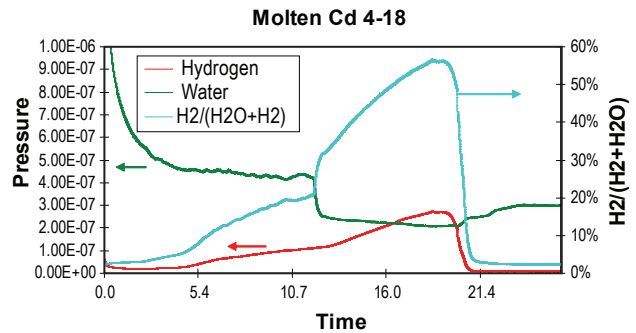


FIGURE 3. Residual Gas Analysis Spectrum of the Water and Hydrogen Profile from a Molten Cadmium Hydrolysis Experiment (The Maximum Ratio Between the Two is 56%).

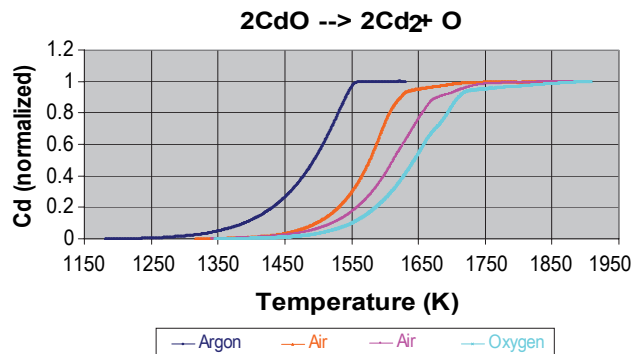
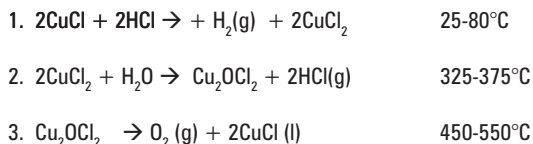


FIGURE 4. Decomposition of Cadmium Oxide in Different Gas Environments

Hybrid Copper Chloride

The Cu-Cl cycle consists of two thermal reactions and one electrochemical reaction in which hydrogen is produced. The three-reactions in the Cu-Cl cycle are shown in the following.



A preliminary process flow diagram was prepared using Aspen Plus with data from completed proof-of-principle experiment for the two thermal reactions (2 and 3 above). The decomposition of the Cu_2OCl_2 (3) was shown in the laboratory to be a simple thermal decomposition. A mechanism was hypothesized and kinetic data were obtained.

Work is currently focused on understanding the hydrolysis reaction (2). Sensitivity analyses of the model showed a critical need for accurate thermodynamic data for Cu_2OCl_2 , which were subsequently measured and added to the physical properties database. The updated model predicted that the presence of HCl in the steam stream in the hydrolysis reaction could inhibit the formation of Cu_2OCl_2 . Engineering methods to minimize HCl content were determined. Thermodynamics predicted 98% yield of Cu_2OCl_2 at 375°C with no significant formation of CuCl when using a steam-to-copper molar ratio of 17:1 (Figure 5a). Experiments showed about 85% yield with significant CuCl formation (Figure 5b). Work is proceeding to reduce the CuCl formation.

Proof-of-principle experiments for the electrochemical reactions showed hydrogen production. AECL designed and tested a flow-through electrochemical cell using various membrane and electrode materials in the 3-reaction model that avoids the necessity of Cu formation from CuCl in the 4-reaction option. This 3-step option is expected to experience lower capital costs than the 4-step option but might require more electrical work. Selection of the option for continued development requires work on both to determine their comparative hydrogen “gate costs”. The Gas Technology Institute (GTI) has developed a plastic body electrode with conducting graphite channels for electrodeposition of copper from CuCl. It found that coating the graphite channels with carbon black permits complete release of the deposited copper with little or no damage to the underlying electrode (Figure 6).

Cobalt Ferrite

Ferrite feasibility tests were conducted using cobalt ferrite ($\text{Co}_{0.67}\text{Fe}_{2.033}\text{O}_4$) supported on yttria stabilized zirconia in a 1:4 ratio by wt. The samples were cast as

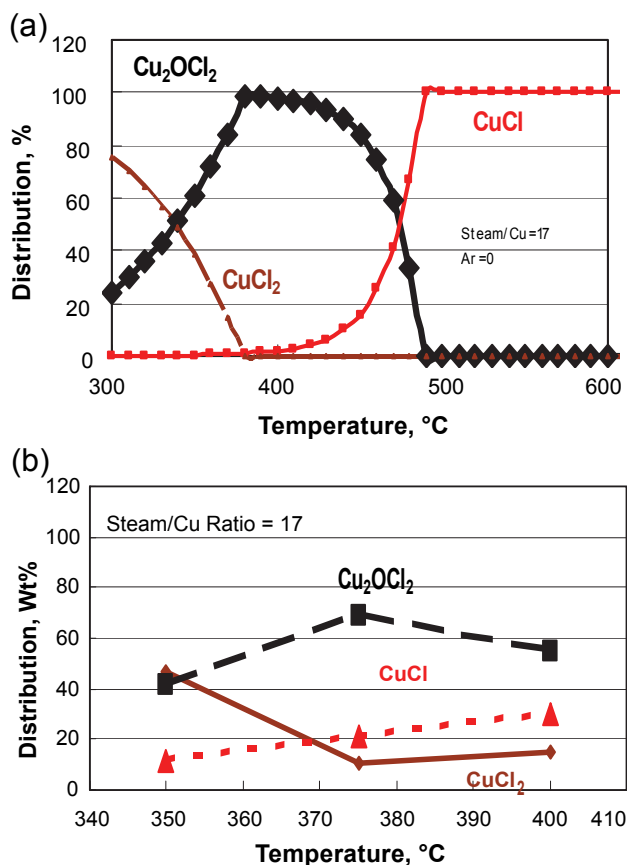


FIGURE 5. (a) Predicted Cu_2OCl_2 Yield and CuCl Formation; (b) Laboratory Results of Cu_2OCl_2 Yield and CuCl Formation

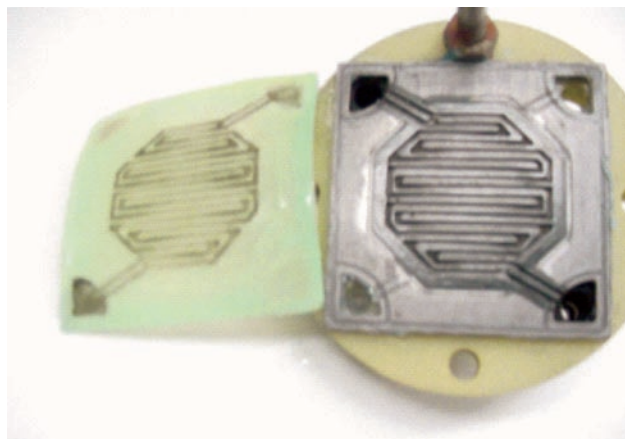


FIGURE 6. GTI-Produced Electrode Using Conducting Graphite Channels Coated with Carbon Black to Facilitate Copper Release

three dimensional lattice structures and tested on-sun at the Sandia National Laboratories (SNL) solar furnace. Hydrogen production with these materials involves two chemical reactions: a high temperature ($1,550^\circ\text{C}$) thermal reduction to produce oxygen followed by a lower temperature ($1,100^\circ\text{C}$) water oxidation to produce

hydrogen. The cobalt ferrite material was run for 10 consecutive cycles with no noticeable loss in chemical performance or structural integrity. In fact, the hydrogen yield increased with the number of cycles. These results are promising in that these materials must maintain reactivity and structural integrity over thousands of cycles in a practical device. A data set from the on-sun testing is shown in Figure 7.

The CR5, shown in Figure 8, is under construction and scheduled for completion in early August, 2007. The completed prototype will be used for on-sun testing at a power level of near 9 kW_{th} .

Solid Particle Receiver

The solid particle receiver is a direct absorption receiver in which solar energy heats a curtain of falling ceramic particle to a temperature in excess of $1,000^\circ\text{C}$. A small scale test platform was built to investigate particle flow properties. Tests were conducted to experimentally determine the distribution of velocity, curtain thickness, and curtain opacity along a drop length of approximately 3 m. Velocity data were measured using a high speed digital camera to image

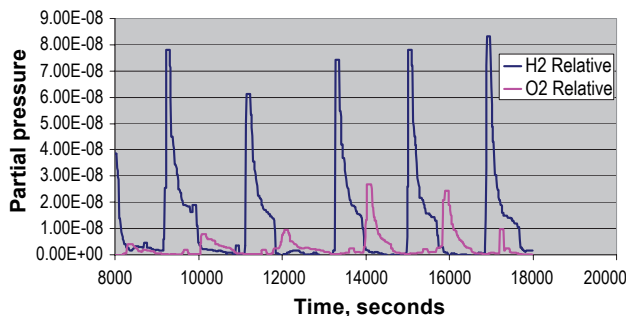


FIGURE 7. Hydrogen Production During On-Sun Testing of Cobalt Ferrite ($\text{Co}_{0.67}\text{Fe}_{2.033}\text{O}_4$; YSZ – 1:4 by Weight) and Oxygen Production at $1,500^\circ\text{C}$, Hydrogen Production at $1,100^\circ\text{C}$

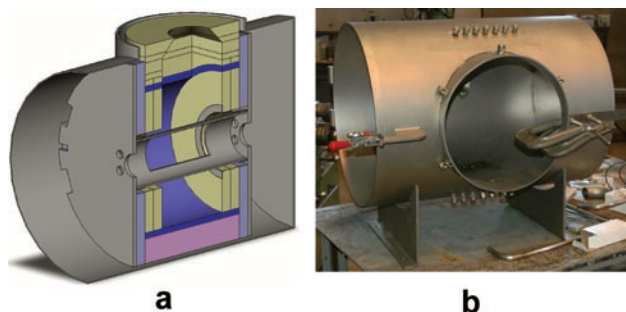


FIGURE 8. (a) Cross Section of the CR5 Device; (b) Picture of the Reactor Housing under Construction at SNL

the particle flow at 1,000 frames per second with an exposure time of 100 microseconds. Five mass flow rates ranging from 1 kg/s-m to 22 kg/s-m were tested and it was found that all flows approached a terminal velocity of about $6\text{-}7 \text{ m/s}$ in a drop distance of 3 m.

Curtain opacity was measured by taking backlit images of the flow from the front. The opacity was calculated by calculating the ratio of the area covered by particles to the total frontal area in the images. In general, the flow is fairly opaque just downstream of the discharge point and then becomes progressively more diffuse as it drops. Eventually the opacity reaches a constant value near the terminal velocity. For the case of flow at 4.5 kg/s-m , the opacity ranges from 0.65 at discharge to 0.45 at terminal velocity. Opacity increases with mass flow rate.

Construction of the on-sun receiver is nearing completion. The prototype is now located within the solar power tower at SNL and will be lifted to the roof in October or November of 2007. We will test at the 1-2 MW level in batch mode. The particle inventory is sufficient to allow batch tests in excess of five minutes.

Advanced Heliostat R&D

Power towers are a preferred method of producing solar hydrogen and solar electricity on a large scale. Heliostats are the most important cost element of a solar power tower plant. Since they constitute $\sim 50\%$ of the capital cost of the plant, it is important to reduce heliostat cost as much as possible to improve the economic performance of power towers. We performed a study [1] to evaluate current heliostat technology and estimated a price of $\$126/\text{m}^2$ given year-2006 materials and labor costs for a deployment of $\sim 600 \text{ MW}$ of power towers per year. This 2006 price yields electricity at $\$0.067/\text{kWh}$ and hydrogen at $\$3.20/\text{kg}$. We proposed research and development that should ultimately lead to a price as low as $\$90/\text{m}^2$, which equates to $\$0.056/\text{kWh}$ and $\$2.75/\text{kg H}_2$. Approximately 30 heliostat and manufacturing experts from the United States, Europe, and Australia contributed to the study content during two separate workshops conducted at the National Solar Thermal Test Facility.

Conclusions and Future Directions

Zinc Oxide

- Zn hydrolysis is surface transport limited; faster reaction rates and higher net conversions can be achieved with higher specific surface area particles.
- Aerosol reactors can be highly advantageous configurations due to rapid reaction rates, relatively low levels of product recombination, and the possibility of forming very small product particles.

- Due to the rapid rate of surface oxidation at high temperatures, materials development should focus on making high temperature materials oxidation resistant.

Future work will focus on improving feed dispersion and Zn yield in the decomposition step, demonstrating the dissociation reaction “on-sun,” and finding suitable materials for high temperature operation of the cycle.

Cadmium Oxide

- Molten cadmium hydrolysis is preferred over the solid process because no mechanical work is required and the reaction rate appears to be favorable.
- Recombination will play a critical role in CdO reduction; methods for quenching the high temperature vapor stream while minimizing recombination will have to be developed.

Future work will optimize the hydrolysis step and define the quench process. These processes will be quantified in laboratory experiments so that overall efficiency of the cadmium cycle can be established with confidence.

Hybrid Copper Chloride

- The process flow diagram has been completed by treating the electrochemical step as a “black box.”
- Electrochemical cell designs have been developed and tested with final selection awaiting completion of the separations study for the 4-reaction cycle option and preliminary cost analysis of the options.

Cost analysis of this cycle will be completed while treating the electrochemical step parametrically. Selected electrochemical cell design will be optimized and updated cost analysis will be completed.

Cobalt Ferrite

- Yttria-stabilized zirconia provided a durable support for cobalt ferrite multi-cycle testing.
- The CR5 receiver/reactor design has been completed. Construction is in process and the receiver is expected to be ready for on-sun testing around August-September 2007.

Cobalt ferrite testing in a refined on-sun reactor will provide data to support system performance and economic (H₂A) models. The results of continued ferrite studies and the CR5 performance will support a go/no-go decision for ferrite cycles in general.

Solid Particle Receiver

- Based on model studies and cold-flow testing, a test version of a solid particle receiver has been designed and construction is nearing completion.

Data and analysis of on-sun testing of the test version of a solid particle receiver will support a firm conclusion regarding the utility of the solid particle receiver as well as the range of operating parameters for its use.

Advanced Heliostat R&D

- A broad study of research and development options has resulted in the definition of a development plan that promises significant reduction in heliostat costs.

Efforts will continue to seek funding to implement the plan identified by the study.

Special Recognitions & Awards/Patents Issued

Four papers presented at the ASME Energy and Sustainability conference in Denver, 2006, were awarded best paper honors:

1. James, D.L., Siegel, N.P., Diver, R.B., Boughton, B.D., Hogan, R.E., “Numerical Modeling of Solar Thermo-Chemical Water-Splitting Reactor,” Proceedings of ISEC2006, Denver, CO, July 8–13, 2006.
2. Allendorf, M.D., Diver, R.B., Miller, J.E., Siegel, N.P., “Thermodynamic Analysis of Mixed-Metal Ferrites for Hydrogen Production by Two-Step Water Splitting,” Proceedings of ISEC2006, Denver, CO, July 8–13, 2006.
3. Miller, J.E., Evans, L.R., Stuecker, J.N., Allendorf, M.D., Siegel, N.P., Diver, R.B., “Material Development for the CR5 Solar Thermochemical Heat Engine,” Proceedings of ISEC2006, Denver, CO, July 8–13, 2006.
4. Diver, R.B., Miller, J.E., Allendorf, M.D., Siegel, N.P., Hogan, R.E., “Solar Thermochemical Water-Splitting Ferrite-Cycle Heat Engines,” Proceedings of ISEC2006, Denver, CO, July 8–13, 2006.

FY 2007 Publications/Presentations

Publications

1. Perkins, C., P. Lichty, A.W. Weimer, and Carl Bingham. “Fluid-Wall Effectiveness for Preventing Oxidation in Solar Thermal ZnO Reactors,” *AIChE Journal*, **53**, pp. 1830-1844, 2007.
2. Perkins, C. and A.W. Weimer, “Computational Fluid Dynamics Simulation of a Tubular Aerosol Reactor for Solar Thermal ZnO Decomposition,” *Journal of Solar Energy Engineering, In Press, Nov. 2007.*

3. Perkins, C. “Solar Thermal Decomposition of ZnO in Aerosol Flow for Renewable Hydrogen Production,” Ph.D. Thesis, *University of Colorado at Boulder*, 2006.
4. Kolb, G.J., Siegel, N.P., Diver, R.B., “Central-Station Solar Hydrogen Power Plant,” *ASME Journal of Solar Energy Engineering*, vol. 129, pp.179-183, 2007.
5. Kolb, G. J., et. al., “Heliostat Cost Reduction Study,” Sandia National Laboratories internal report, SAND2007-3293, 2007.
6. Huajun Chen, Yitung Chen, Hsuan-Tsung Hsieh, Nathan Siegel, “CFD Modeling of Gas Particle Flow Within a Solid Particle Solar Receiver,” *ASME Journal of Solar Energy Engineering*, vo.129, pp.160-170, 2007.

Presentations

1. Funke, H., C. Perkins, and A.W. Weimer, “Hydrogen Generation by Hydrolysis of Zinc Powder Aerosols,” *Proceedings of Solar 2006*, Denver, CO, July 10–13, 2006.
2. Perkins, C., P. Lichty, and A.W. Weimer, “Reaction Kinetics of the High Temperature ZnO Dissociation Step in a 2-Step Solar Thermochemical Water Splitting Process,” *Proceedings of the AIChE Annual Meeting*, San Francisco, CA, November 12–17, 2006.
3. Funke, H., C. Perkins, and A.W. Weimer, “Investigation of the Water Reduction with Zinc Powder Aerosol to Form Hydrogen Fuel,” *Proceedings of the AIChE Annual Meeting*, San Francisco, CA, November 12–17, 2006.

4. Weimer, A.W., C. Perkins, P. Lichty, D.M. King, H. Funke, T. Francis, and A. Lewandowski, “Critical Challenges in Developing Solar Thermochemical Water Splitting Cycles for Renewable Hydrogen,” *Proceedings of the AIChE Annual Meeting*, San Francisco, CA, November 12–17, 2006.
5. J. G. Masin and M. A. Lewis, Efficiency Calculations for the Hybrid Copper-Chloride Thermochemical Cycle, *Proceedings of the AIChE Annual Meeting*, San Francisco, CA, November 12–17, 2006.
6. M. A. Lewis and J. G. Masin, Cu-Cl Cycle R&D at Argonne National Laboratory, University of Ontario Institute of Technology, Oshawa, Canada, Dec. 8, 2006.
7. M. A. Lewis, “Status of the R&D effort for the Cu-Cl Cycle,” University of Ontario Institute of Technology, Oshawa, Canada, May 28, 2007.
8. James, D.L., Siegel, N.P., Diver, R.B., Boughton, B.D., Hogan, R.E., “Numerical Modeling of Solar Thermo-Chemical Water-Splitting Reactor,” *Proceedings of ISEC2006*, Denver, CO, July 8–13, 2006.

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1. Kolb, G. J., et. al., “Heliostat Cost Reduction Study,” Sandia National Laboratories internal report, SAND2007-3293, 2007.