

# Purdue Hydrogen Systems Laboratory

## Part I: Anaerobic Biological Production of Hydrogen

R. Kramer, L. Pelter, E. Ting  
Energy Efficiency and Reliability Center  
Purdue University, Calumet, IN

J. Patterson, J. Gore  
Purdue University, West Lafayette, IN

May 19, 2009

*This presentation does not contain any proprietary, confidential, or otherwise restricted information*

# Overview

## Timeline

**Start – September 2006**  
**End – September 2009**  
**70% complete**

## Budget

- \$2,470,006\*
  - \$1,924,000 (DOE)
  - \$ 50,000 (to NERL/subcontractor)
  - \$496,006 (Purdue)
- Funding received in FY09  
 \$ 984,000

\* This is the overall budget for both hydrogen production and storage research. This presentation only covers the production part

## Barriers

### Barriers addressed

- AL. H<sub>2</sub> molar yield
- AK. Feedstock cost
- AL. Systems engineering

### Targets

	2007	2010	2015
Hydrogen yield percentage (%)	20	25	> 30

## Partners

- Cargill
- Griffith Labs
- Advanced Power Technologies
- Innovene
- BP
- NREL

## Project Objectives - Relevance

- Increase the production of hydrogen from the anaerobic fermentation of organic waste.
- Develop methods and techniques to maximize hydrogen production for a modular energy system for local energy production.
- Develop methods to optimize the value of the produced hydrogen for use in a modular system for local energy production.
- Develop a solar thermal energy system to pre and post process associated waste streams thereby reducing ancillary energy requirements and reducing potential environmental contamination issues for the final product.
- Identify methods to separate hydrogen from bio gas and investigate feasibility of using catalysis to produce a marketable chemical product from the produced carbon dioxide.

## Approach

- Investigate methods to increase hydrogen production from organic waste streams using microbial fermentation through the use of an automated statistical experimental design that employs variations in Ph, temperature, and waste concentration to determine the optimal operating condition.
- Utilize statistical experimental design.
- Develop automated testing device to speed the determination of parameters that maximize hydrogen production.
- Investigate various microbial consortia.
- Investigate various waste materials.
- Utilize a vacuum tube solar collector system for the pre and post treatment of organic waste material.
- Minimize ancillary energy requirements.
- Develop designs for implementation of the technology as a local electric and thermal energy source.

## Milestones

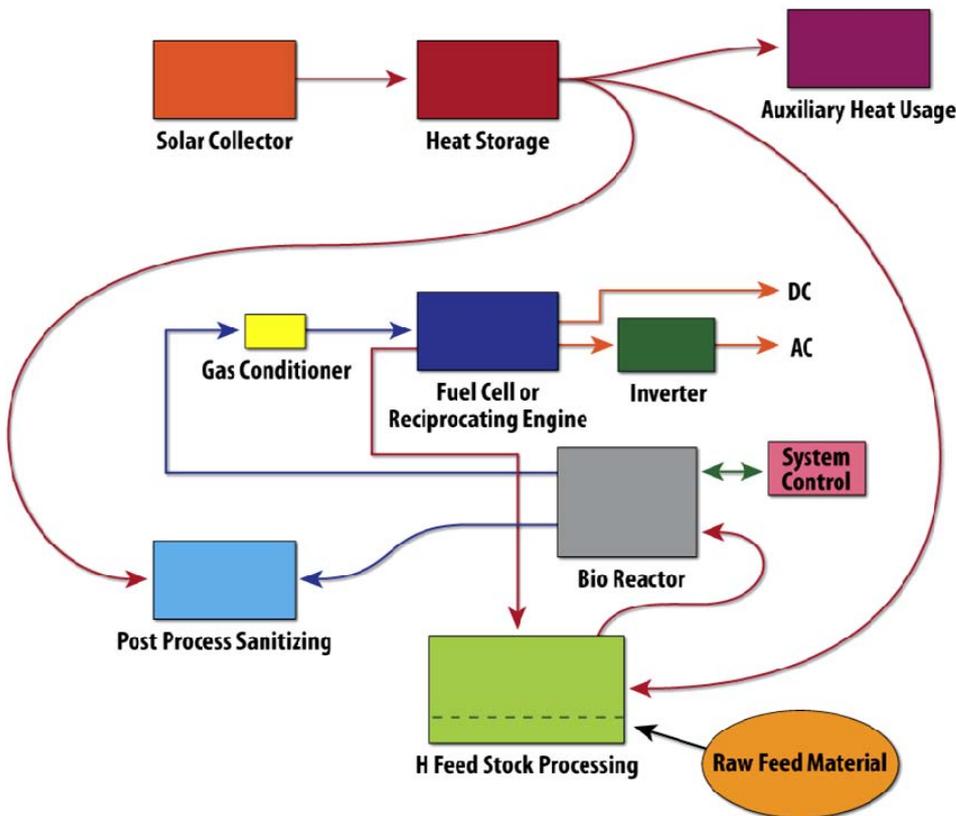
Month/Year	Milestone or Go/No-Go Decision
Nov-08 Apr-09	Make contacts with industrial advisors and hold industry advisor meetings. 100% complete
Aug-09	Milestone: Complete development and initial testing of automated testing device to determine optimal conditions for anaerobic hydrogen production. This device accelerates the determination of optimal hydrogen production conditions. Consider batch vs continuous methods. 70% complete
Sep-09	Milestone: Consider hydrogen production from low water content substrates. 35% complete
Dec-08 Jul-09	Milestone: Construct solar system, including a processing test vessel. Evaluate effectiveness for processing waste. 70% complete

## Previous Technical Accomplishments

- Initial testing of various combinations of microbial consortia was done with organic waste materials to determine parameters that influence production. It was determined that pH, temperature and waste material concentration had the greatest influence on hydrogen production for the test conditions considered.
- An automated testing system was designed and constructed as a tool to assist in determining optimal operating parameters for the process.
- An energy balance model was developed to assess the value of the technology as a local energy source.
- Tests of continuous production system conducted at NREL for Purdue food waste and inoculum samples.

# Technical Accomplishments and Progress

## Development of a Modular System for Local Energy Production

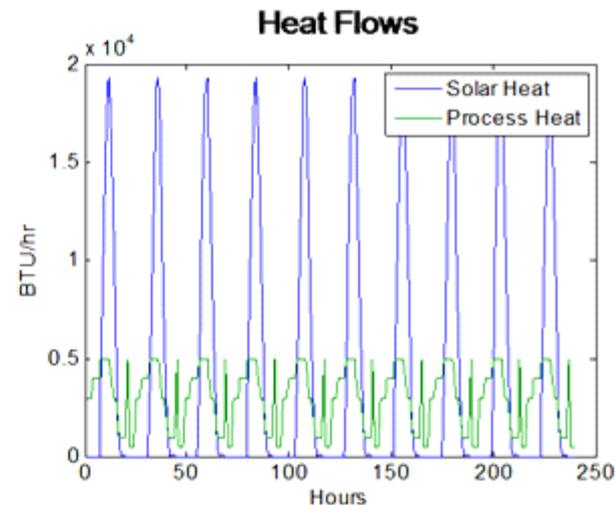
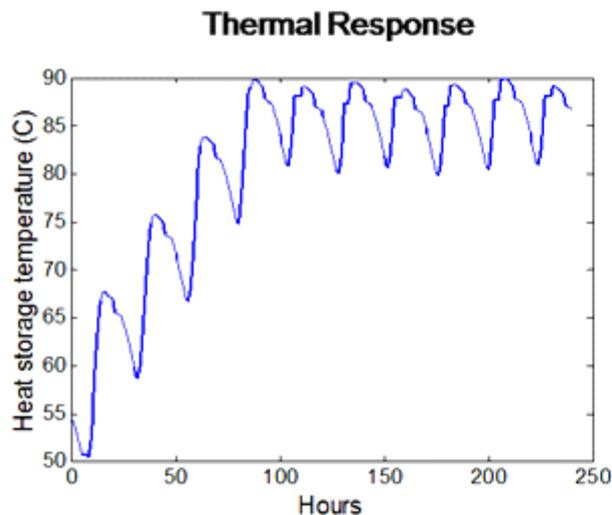


- The initial design of a modular energy system for local energy production has been completed.
- Organic waste material is used in an anaerobic process to produce hydrogen.
- Research to maximize hydrogen production through the use of a statistical experimental design is ongoing.
- Solar thermal system is used for pre and post waste processing and other ancillary uses.

# Technical Accomplishments and Progress

## Development of a Modular System for Local Energy Production

- Energy Balance for Modular Energy System was developed.
- Initial experimental hydrogen production rates were used to determine the size and timing for a prototype design.
- Electric output was used as a modeling parameter for the basic energy balance.
- The solar system was sized to provide heating for pre and post processing and limited ancillary purposes.
- Heat storage is used to store excess heat for purposes such as building heating.
- Use of excess heat to produce potable water is being considered.



# Technical Accomplishments and Progress

## Determination of Initial Optimal Hydrogen Production Parameters



- Waste material is statistically sampled from entire stock of processed food waste.
- Homogenized food waste is placed in 120 ml bottles, buffer is added, bottles are purged with nitrogen, bottles are sealed and autoclaved.
- Bottles are placed in constant temperature bath.
- Gas collection, pressure measuring, and purging needles are inserted into bottles.
- Bottles are purged with nitrogen. GC is used to verify that no oxygen is present.
- Bottles are pressurized slightly with nitrogen and gas purge needle is removed.
- Pressure is continuously monitored by transducers attached to each bottle.
- Gas composition (GC) and pressure (gas volume produced) is automatically measured for each bottle over time.

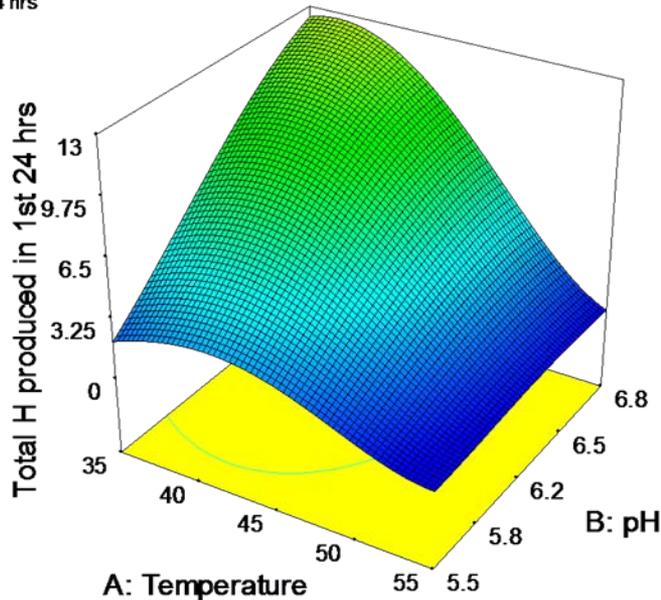
# Technical Accomplishments and Progress

## Determination of Initial Optimal Parameters

Design-Expert® Software  
Original Scale  
Total H produced in 1st 24 hrs  
18.7255  
0.0001

X1 = A: Temperature  
X2 = B: pH

Actual Factor  
C: Concentration = 0.93



- Central Composite Design is used to determine operating values that maximize anaerobic hydrogen production for waste samples.
- For each type of waste and inoculum, operating parameters are varied to determine what combination of conditions maximizes production.
- Sensitivities to differences in substrate and inoculum are initially considered.
- pH values are at start of fermentation process after autoclave.
- Comprehensive tests in process using automated testing device.

# Technical Accomplishments and Progress

## Determination of Initial Optimal Parameters

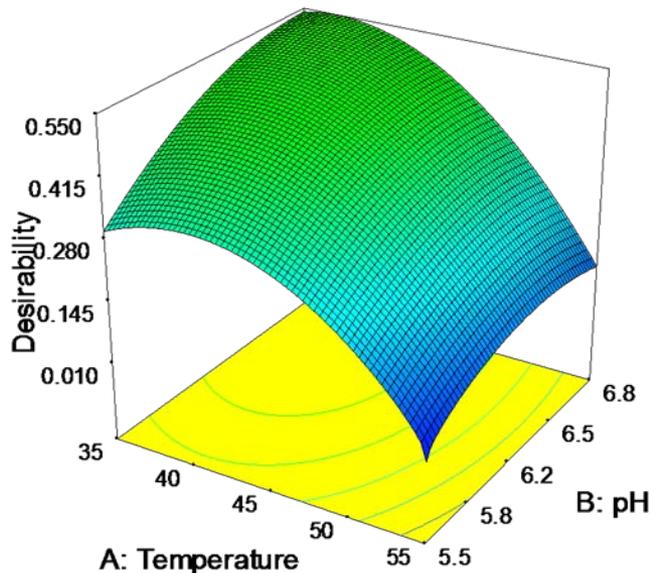
Design-Expert® Software

Desirability



X1 = A: Temperature  
X2 = B: pH

Actual Factor  
C: Concentration = 0.93



- Volume and hydrogen concentration are maximized for the Central Composite design using the Simplex Method to determine the most desirable operating conditions.
- Sensitivity tests for the predicted operating values are done to determine the validity of the prediction.
- Sensitivity to different substrates and inoculum are being considered.
- Influence of water concentration is being considered as another optimization parameter.

# Technical Accomplishments and Progress

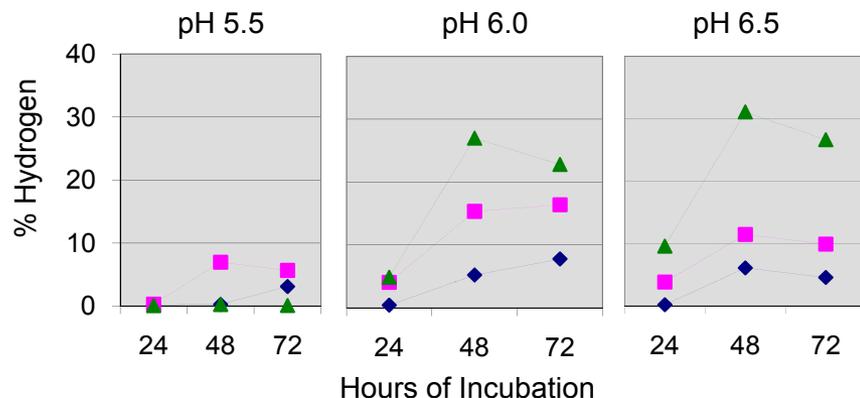
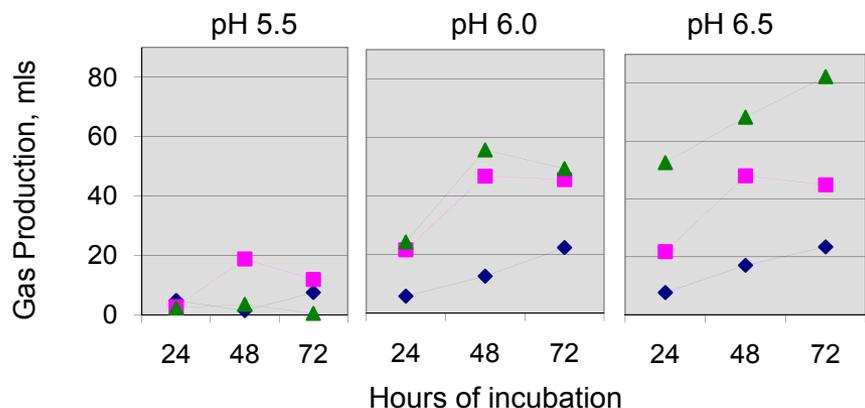
## Initial Food Waste Nutrient Analysis, Ford Hall Food Waste 1

Component	As Sent	Dry Wt.
Moisture (%)	06.30	////
Dry Matter (%)	93.70	////
Crude Protein (%)	17.40	18.60
Crude Fat (%)	12.60	13.40
Acid Detergent Fiber (%)	33.20	35.40
Ash (%)	03.35	03.58
Total Digestible Nutrients (%)	81.50	87.00
Net Energy-lactation (Mcal/lb)	00.85	00.91
Net Energy-maint. (Mcal/lb)	00.90	00.96
Net Energy-gain (Mcal/lb)	00.59	00.63
Digestible Energy (Mcal/lb)	01.63	01.74
Metabolizable Energy (Mcal/lb)	01.51	01.61
Sulfur (%)	00.26	00.28
Phosphorus (%)	00.25	00.27
Potassium (%)	00.37	00.40
Magnesium (%)	00.06	00.07
Calcium (%)	00.90	00.96
Sodium (%)	00.50	00.53
Iron (ppm)	66.00	70.00
Manganese (ppm)	09.00	10.00
Copper (ppm)	06.00	06.00
Zinc (ppm)	28.00	30.00

# Technical Accomplishments and Progress

## Use of Distiller's Grain From Ethanol Production

### Results for Distiller's Grain



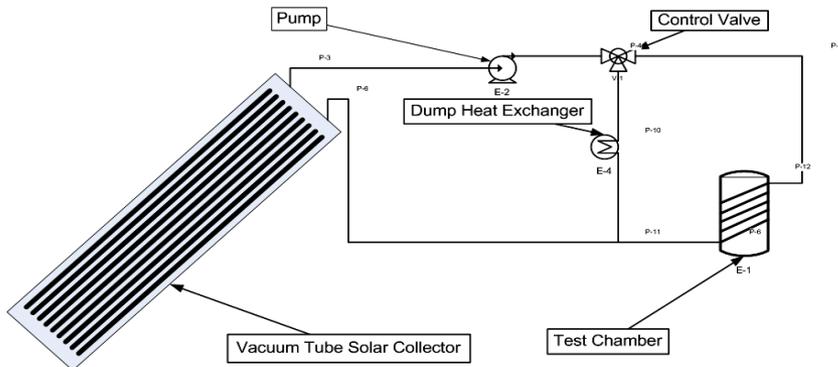
1.6% Dry Material (DM)    3.3% DM    6.7% DM

- Use of Distiller's Grain for hydrogen production is being investigated.
- With increased levels of ethanol production from corn there is an increasing amount of Distiller's Grain (residue remaining after ethanol production).
- Cattle will tolerate < 40% concentration of Distiller's grain mixed with feed.
- In tests, yeast inoculum produced more hydrogen than anaerobic sludge.
- Currently considering effect of inoculum type and concentration, pH, and substrate concentration.
- 120 ml serum bottles used for gas production.

# Technical Accomplishments and Progress

## Solar Thermal System Designed, Constructed and Tested

Solar Thermal  
Test System



- Vacuum tube solar collector used to produce heat for pre and post processing.
- Control system developed to produce high temperatures for processing waste material.
- Routinely obtain temperatures + 115 °C.
- Waste material is routinely batch processed.
- Further optimization of process controls to occur in spring 2009.
- Considering steam generator test for summer 2009.

# Technical Accomplishments and Progress

## Effectiveness of Solar Thermal System Tested

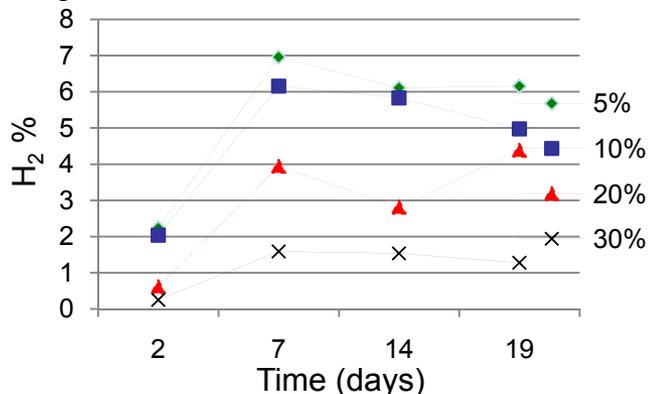


- The developed solar thermal approach effectively eliminates methanogens in the feed material and inoculum.
- Material is heated at 90-100 °C for 10 minutes in the processing chamber of the solar collector system.
- Solar thermal system sanitizes waste after it is used for hydrogen production.
- Without solar thermal treatment plates (upper) show significant growth.
- After solar thermal treatment plates (lower) show no growth.

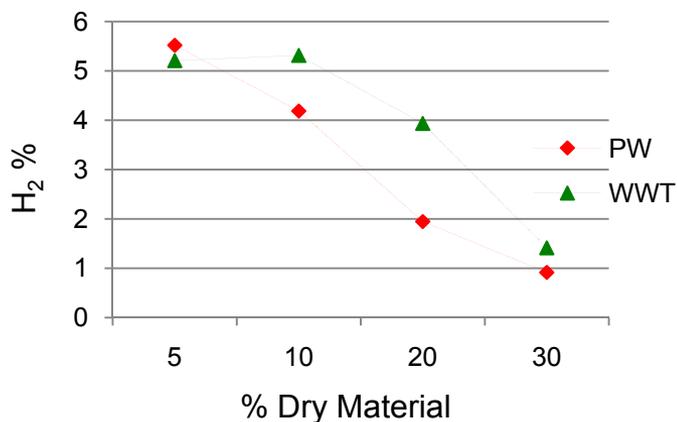
# Technical Accomplishments and Progress

## Reduce Water Concentration

Hydrogen % for Various Substrate Concentrations



H % vs Water concentration and Inoculum



- Reducing water content allows for a decrease in size and weight of modular hydrogen energy system.
- Waste was prepared by drying at 50 °C and then passing through a 1 mm screen in a hammer mill with dry ice.
- Food waste, (1.0 g ± 0.05 g) was added to vials and appropriate amounts of anaerobic buffer was added to give final concentrations of 5%, 10%, 20% and 30%.
- The vials were autoclaved and cooled.
- Vials were then inoculated with 1.0 ml of diluted pig waste (PW) or waste water anaerobic sludge (WWT) and incubated at 37 °C.
- Currently investigating use of support structures to further reduce water concentration.

## Collaborations

- Communications and collaboration with National Renewable Energy Laboratory have continued.
- Periodic advisory board meetings are held to gain input from industry.
- Advisory board members include: Cargill, Griffith Labs, BP, Advanced Power Technologies, and Innovene.
- Collaborative efforts with researchers at the Purdue Calumet and Lafayette campuses continue.

## Future Work

- Continue efforts to increase hydrogen production by optimizing operating parameters.
- Continue efforts to reduce the water concentration of waste material during processing.
- Obtain and test new inoculum samples.
- Obtain and test new waste materials including additional testing of distillers grain.
- Initiate plans for design of bench top test reactor.

## Project Summary

- Operating parameters that maximize hydrogen production for initial inoculum and waste material samples have been identified.
- The solar thermal system has been effective in pre and post processing of waste material and expanding the value to include production of a sanitized fertilizer product.
- Initial results indicate this approach can be used for local energy production after further development and optimization.
- Other uses for excess solar heat including building heating and potable water production are under investigation.
- Possibility of conversion of carbon dioxide into a marketable chemical product is being considered.