Investigation of Pressurized Entrained-Flow Kraft Black Liquor Gasification in an Industrially Relevant Environment

DOE Cooperative Agreement DE-FC26-04NT42261

Quarterly Technical Progress Report, Year 1 Quarter 2

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Investigation of Pressurized Entrained-Flow Kraft Black Liquor Gasification in an Industrially Relevant Environment (DE-FC26-04NT42261)

Quarterly Report for Project Budget Period 1, Quarter 2

Principal Author:
Kevin Whitty
University of Utah

OBJECTIVES

The overall objective of this project is to acquire critical data necessary for successful commercialization of entrained-flow black liquor gasification through a combination of laboratory-scale experiments and operation of a small-scale gasifier operating under conditions representative of those in a commercial system. The project focuses on the gasifier reactor itself, and takes into consideration the entire range of conversion, from the initial black liquor droplet to the final smelt and gas products. Specifically, the project has the following four technical objectives: (1) to characterize black liquor droplet and particle formation in a pressurized, high temperature environment, (2) to generate data on the chemical and physical aspects of black liquor conversion in a high temperature gasifier, (3) to characterize major and minor components in the synthesis gas produced during high temperature kraft black liquor gasification, and (4) to use the information acquired to develop tools useful for design, optimization and troubleshooting of entrained-flow back liquor gasifiers.

BACKGROUND

Black liquor gasification has long been recognized as a promising technology for the pulp and paper industry. Not only does it have the potential to increase energy efficiency and environmental performance of the black liquor recovery system, but gasification opens up opportunities for advanced pulping technologies that can increase pulp yield. Recently, it has been recognized that the syngas from a black liquor gasification system could be used as feedstock for production of chemicals or automotive fuels, making a pulp mill a natural site for a forest biorefinery system. This additional product stream would give the pulp and paper industry more flexibility in challenging market conditions, and could displace petroleum otherwise imported with a domestically-produced, renewable fuel.

Despite the advantages of black liquor gasification and many efforts over the past few decades to develop an effective gasification technology, black liquor gasification has not yet achieved commercial success. Several technical hurdles have yet to be overcome, and there is a notable lack of information about the processes that take place inside an entrained-flow gasifier. Having detailed information on the conversion behavior of black liquor in high temperature gasification systems, from droplet formation through to properties of the smelt and syngas products, would greatly improve the understanding of the process and aid successful commercialization of the technology. This project aims to provide this needed information.
STATEMENT OF WORK

This project is broken down into five technical tasks, described in the sections that follow.

Task 1: Design and construction of an entrained-flow gasifier

A new entrained-flow gasifier will be designed, built and installed in the existing Black Liquor Gasification Research System at the University of Utah. This reactor will be capable of operating either at atmospheric pressure or under pressurized conditions (up to 10 atm) and wall temperatures as high as 1200°C. The design of the gasifier will be unique. It will not simply be a scaled-down version of an industrial-scale gasifier. Rather, it will be designed to meet the specific objectives of this project, namely to obtain data on chemical and physical properties of the liquor and products throughout the entire range of conversion. Compared to an industrial unit, the gasifier will have a higher length-to-diameter (L/D) ratio and several ports for sampling along the length of the unit. However, the gasifier should be able to produce a syngas quite representative of syngas from an industrial system.

Deliverables from this task include a report detailing the design of the gasifier and at least one follow-up report on the performance of the system.

Task 2: Evaluation of droplet formation and burner performance

The purpose of this task is to characterize and model the process of droplet formation under pressurized, high temperature conditions, and to correlate observed droplet characteristics with the performance of the burner and gasifier. There are three components to the task, described below.

Subtask 2a: Spray and droplet formation characterization. Imaging will be performed in two complementary systems. The proposed gasifier is designed to allow optical access of the liquor injector tip and the region just downstream of the burner. The University of Utah will use its high-speed camera to video the spray where optical access is possible within the high pressure gasification chamber. More controlled, complementary tests will be performed in an existing atmospheric pressure optical access drop tube furnace at University of Utah. Small black liquor droplets from an injector will be videoed as they undergo drying and pyrolysis to identify swelling behavior under high heating rate conditions.

Subtask 2b: Computational modeling of black liquor droplet formation. Under this subtask, the data from the imaging tests described above will be used to improve and validate an existing computational model describing black liquor droplet formation. Data from Subtask 2a will be used to validate that the fundamental mechanics of the computational model are correct. This results in a higher level of confidence in the model when it is applied to an industrial burner.

Subtask 2c: Computational model validation. The third component of this task involves comparison of model predictions versus unique droplet formation scenarios not already characterized in Task 2a. Since the model is optimized/calibrated with the data collected in Task 2a, reproducing these findings is not a true validation of model accuracy. Rather, a unique scenario of inputs shall be tested following the same criteria specified in Task 2a for model validation. The imaging results will then be compared to the model’s predictions for both scenarios with and without atomizing gas and model validation will be determined. If this comparison proves to be unacceptable, the model may be refined and Subtask 2c will be repeated with another unique droplet formation scenario.

Deliverables from this task include data and photographs from the imaging studies, a description of the computational model, results of simulations performed with the model, model predictions for a commercial burner, and a model validation analysis.
**Task 3: Detailed analysis of black liquor conversion**

This task takes over where Task 2 ends – when droplets have been formed but have not yet begun to convert. The objective of this task is to characterize the progression of physical (subtask 2a) and chemical (subtask 2b) properties of black liquor as it is converted from droplet to char to fully converted smelt bead. This will be achieved through a combination of sampling in the Utah gasifier operating under pressurized, industrially relevant conditions and more fundamental laboratory experiments. Particular attention will be paid to the near-burner region, where the liquor undergoes its most dynamic changes.

**Subtask 3a: Physical characterization of black liquor during conversion.** This subtask will provide new information on physical properties of the liquor such as density, porosity, structure, internal surface area and sticking propensity. Properties throughout the entire range of conversion will be measured.

**Subtask 3b: Chemistry of black liquor conversion.** This task aims to characterize chemical transformations that occur as black liquor progresses through the various stages of conversion in the gasifier. Both gas and condensed-phase material will be analyzed. By comparing gas and condensed-phase samples throughout the reactor, it will be possible to generate a map of the environment, and to identify rates and mechanisms of conversion of the liquor to gas.

Deliverables from this task include reports on the progression of chemical and physical characteristics of black liquor as it undergoes conversion under a variety of conditions. Submodels describing liquor conversion, suitable for incorporation into CFD models, will be made available.

**Task 4: Transport and radiative properties of black liquor smelt**

The aim of this task is to provide quantitative data on transport properties (viscosity, thermal conductivity) and emissivity of molten kraft black liquor smelt. These properties impact gasifier performance, and are essential for understanding and modeling the behavior of the gasifier. Smelt viscosity will be measured using a slag viscometer, essentially a viscometer immersed into a bath of molten smelt at a given temperature. Approximate thermal conductivity measurements will be made by heating smelt in a 1-D heat pipe and measuring heat flux and temperature difference. Radiative properties and smelt emissivity will be analyzed by optical methods including visible and IR emission measurements. Total emissivity will be obtained by measuring total emissive power with a radiometer and smelt surface temperature. Efforts will also be made to measure spectral emissivity. The smelt will be doped with different concentrations of carbon, since carbon has a broad-band and relatively high emissivity.

Deliverables will include data on viscosity, thermal conductivity, and radiative properties of smelt and carbon-smelt mixtures.

**Task 5: Characterization of synthesis gas from an entrained-flow gasifier**

This task involves detailed analysis of the product gas produced in the Utah entrained-flow gasifier. The product gas will be analyzed by several techniques. Major species will be monitored using existing online continuous emission monitors. Minor species will be measured through a combination of existing on-line gas chromatography and GC/MS of extracted samples. Particular attention will be paid to reduced sulfur species and tars, and efforts will be made to characterize particulate matter in the quenched gas. In addition to sampling at the exit to the quench, sampling at the gasifier reactor exit will also be attempted. The impact of system disturbances on the quality of the syngas will be identified by intentionally upsetting operation of the gasifier.

Deliverables from this task include detailed reports of measured concentration ranges during stable and upset conditions.
SUMMARY OF TECHNICAL PROGRESS THIS QUARTER

Accomplishments for the various technical tasks during this quarter are presented in the sections that follow.

**Task 1: Design and construction of an entrained-flow gasifier**

During this quarter, the design of the entrained-flow gasifier was finalized, and bids for the pressure vessel have been requested. Metallurgy in the lower (quench) proved to be a challenge. The green liquor produced once the smelt is dissolved has the potential to be corrosive even for austenitic materials such as stainless 316. A duplex steel, 2205, was recommended for this service. Unfortunately, this material is roughly three times the cost of stainless. The chosen solution involves welding a sealed liner of 2205 into a carbon steel pressure vessel for the bottom section of the system.

The final design comprises three main sections. The upper reactor section is the heart of the system, and is where the gasification will take place. This will be lined with two layers of refractory. The short middle section provides a transition between the reactor and quench systems. A short water-cooled channel directly below the reactor outlet leads to four spray nozzles that will partly cool the gas as it exits the reactor. The transition section also supports a dip tube that extends into the quench section. The quench section itself will have a water bath through which the product gas will bubble and cool before exiting the system.

Most of the auxiliary systems required to operate the gasifier have been specified, and bids for major items have been requested. Other components have already been ordered.

**Task 2: Evaluation of droplet formation and burner performance**

Two complementary approaches to shed light on black liquor droplet formation and spray characteristics are being undertaken. Brigham Young University will characterize sprays of black liquor in a hot, non-pressurized environment. The University of Utah is considering size and shape development of a single droplet within a spray.

**BYU Spray Chamber**

**Construction of Spray Chamber**

An electrically heated, atmospheric spray chamber has been designed, constructed and tested to 1000°C. Testing indicates higher temperatures are possible, in excess of 1200°C. The heated section of the chamber is a 0.610 m long 0.152 m inside diameter Silicon Carbide tube. The tube is surrounded by four resistance heaters, and insulated with refractory material. Optical access to the spray will be obtained by moving the spray nozzle to different heights inside the tube and imaging the spray as it exits the bottom end of the tube into an induced draft exhaust duct. Unburned material and particulate will be collected in the exhaust system.

The exhaust system has been designed and should be installed, along with other required infrastructure during the second quarter. The fuel delivery system is detailed in the next section.

This spray chamber is also one section of a new multi-fuel entrained flow combustion reactor that may be used for task 3: Detailed Analysis of black liquor conversion during high temperature gasification. The reactor is made by stacking seven to eight chambers identical to that in Figure 1.
Design of Fuel Delivery System

The fuel delivery system is shown conceptually in Figure 2. The pump with motor and control system is already purchased. It is a Seepex, progressive cavity, positive displacement pump that will allow precise and repeatable control of the fuel flow rate. The fuel hopper, fuel lines and nozzles, and heat trace system are still in the design phase. This work will be the focus of the next quarter.

Nozzle Selection

For work in the above mentioned spray chamber, a narrow angle spray is required (~15°). The target drop size is around 300µm with a narrow size distribution. Initial experiments will be performed with an internal mix air atomizing nozzle supplied by Spraying Systems Co. (Spray Set-up No. SU11).

![Figure 1. Diagram of the electrically heated spray chamber. Multiple chambers may be stacked to form an entrained flow reactor.](image)

Experimental Approach

Droplet formation will be studied using high speed imaging, and a phase Doppler particle anemometer (PDPA) laser techniques if applicable. The first objective is to determine the effect, if any, of black liquor
flashing in an internal mix atomizer. The nozzle will first be characterized using unheated water, and then heated black liquor. Spray cone angle, droplet shape and size, and qualitative imaging will be used to compare the water and black liquor sprays. The size and shape of the black liquor jet should be predictable based on the measurements from the water unless flash boiling is occurring.

**Figure 2.** Conceptual design of the fuel delivery system.

**Single Droplet Studies**

Progress was made on developing a system to generate single droplets of black liquor which can then be fed into an optical access drop tube furnace. The droplet generator is based on the principle of a vibrating orifice. Black liquor on one side of an orifice is pressurized just to the point where the liquor is about to be pushed through the orifice. An oscillating electric signal is then fed to a piezoelectric ceramic to which the orifice is attached. The small vibrations that result break the surface tension of the liquor in a controlled manner, allowing a stream of monodisperse droplets to be ejected, one for each vibration. The challenge lies in feeding the black liquor. The system must be kept hot enough to allow the liquor to flow, yet not so hot that it flashes or dries. During this quarter a system for pressurizing and feeding black liquor was set up, and a head containing the vibrating orifice was plumbed together. So far, generating a stream of concentrated liquor has not been successful, though it is possible to create single droplets.

**Task 3: Detailed analysis of black liquor conversion**

Experimental activity in this task must wait until the gasifier has been constructed. In the meantime, however, efforts are aimed at acquiring as much relevant information as possible through a literature review. Gasification by partial oxidation is essentially sub-stoichiometric, oxygen-rich combustion. So, much of the information that has been published regarding pyrolysis and combustion of black liquor is relevant to this type of gasification. Information on atmospheric black liquor pyrolysis and combustion is readily available. Articles regarding conversion under pressure are limited.

**Task 4: Transport and radiative properties of black liquor smelt**

Going into this quarter, we were optimistic that we would be able to acquire an available used slag viscometer that could be used for measuring the viscosity of black liquor smelt. There are only a few slag viscometers in existence, so this seemed like a good opportunity. The lab that has the unit is closing
down and offered the equipment to the university for a good price. The equipment is somewhat old, so we contacted the company that bought out the original manufacturer and which provides service for that brand of equipment. It turns out that they provide no technical support for the slag viscometer in question. In particular, they do not manufacture the high temperature molybdenum spindles that would be necessary for smelt viscosity measurements. Alternatives are now being explored.

**Task 5: Characterization of synthesis gas from an entrained-flow gasifier**

No activity for this task will take place until after Task 1 is complete.

**PLANS FOR NEXT QUARTER**

For the period April-June 2005, efforts will focus on the issues outlined below:

**Entrained-flow gasifier construction.** Bids for the reactor system will be received and a fabricator will be selected. Discussions with the fabricator and fine-tuning the design to make sure the vessel complies with all ASME code will ensue. Once a final design has been established, the fabricator will develop fabrication drawings and run the necessary stress calculations. It is hoped that this procedure will not take long, and that procurement of parts and fabrication of the vessel can be nearly done by the end of the quarter. Procurement of other parts of the system will continue, and installation of those parts that can be put in place in the absence of the gasifier itself will commence.

**Droplet imaging and modeling studies.** For BYU's spray characterization tests, plans for the next quarter include installation of the experiment infrastructure, including the exhaust system, design and ordering of parts for the fuel system, assembly of the spray chamber and fuel system and preliminary tests with water. The cost of the reactor and fuel injection system have been supported internally by Brigham Young University and have not been acquired from funds for this project. These tests will establish the ability to characterize nozzles to be used in the entrained-flow gasifier at the University of Utah. Once this is established, the facilities will be used to characterize additional nozzles. Also of interest is to perform a fundamental study on the effects of drying, devolatilization and swelling on secondary spray breakup. This can be done with the same facilities but by using a nozzle which does not atomize the droplets prior to injection. The balance between work load after the first year (3rd Quarter 2005) will depend on the success of producing a desirable spray for the entrained flow facility.

Development of the system to generate a stream of monodisperse droplets will continue. An alternative black liquor feeding approach will be tested, in which black liquor of lower concentrations will be pumped, rather than pressure fed, to the system. Starting with lower concentrations will ensure that the system can function. Higher concentrations will be successively fed.

**Black liquor conversion studies.** Currently, there are no accepted submodels to describe the chemistry liquor conversion in an entrained-flow gasifier. Development of such models is one of the goals of this program, and requires experimentation and analysis of conversion in the yet-to-be-built gasifier. That said, the current literature search has revealed that there are models and data available on black liquor combustion that have parallels with gasification by partial oxidation. Over the next half year, the most suitable of these data will be compiled and a model or series of models to describe conversion during gasification will be developed.

**Smelt properties studies.** Alternatives for measuring the viscosity of smelt will be identified. One possibility is to construct a new such system, and cost estimates for such a system will be explored.
Another, less attractive possibility would be to establish collaboration with an institution that has a suitable device, and arrange to have the tests done at that institution. Development of a system to measure thermal conductivity of black liquor smelt will commence during this quarter as well, and an initial design for a system to measure smelt radiative properties will be constructed.

**SCHEDULE AND PROJECT STATUS**

The major milestones for the project and the planned actual dates of completion are listed in Table 1.

<table>
<thead>
<tr>
<th>ID No.</th>
<th>Task/Milestone Description</th>
<th>Planned Completion</th>
<th>Actual Completion</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Design and construction of entrained-flow gasifier</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.1</td>
<td>Basic design finalized</td>
<td>2</td>
<td>3</td>
<td></td>
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<tr>
<td>1.2</td>
<td>Design finalized, components specified, request bids</td>
<td>3</td>
<td>3</td>
<td>In progress</td>
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<tr>
<td>1.3</td>
<td>All components ordered</td>
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<td></td>
<td></td>
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<td>1.4</td>
<td>Construction of gasifier complete</td>
<td>9</td>
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<tr>
<td>1.5</td>
<td>Installation of system complete</td>
<td>10</td>
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<tr>
<td>1.6</td>
<td>Report on gasifier design/operation</td>
<td>15</td>
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<td></td>
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<tr>
<td>2</td>
<td>Evaluation of droplet formation, burner performance</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.1</td>
<td>Foundation for computational model complete</td>
<td>12</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.2</td>
<td>Initial droplet imaging/LDA tests complete</td>
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<td>2.3</td>
<td>Follow-up imaging/LDA tests complete</td>
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<td>2.4</td>
<td>Computational model of droplet formation complete</td>
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<tr>
<td>2.5</td>
<td>Report on droplet formation/burner performance</td>
<td>36</td>
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<td>3</td>
<td>Detailed analysis of black liquor conversion</td>
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<td>Literature review complete</td>
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<td>Sampling procedure established</td>
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<td>Acquisition of data complete</td>
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<td>Development of models describing conversion complete</td>
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<td>3.5</td>
<td>Report of physical properties vs. conversion</td>
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<td>3.6</td>
<td>Report on conversion chemistry</td>
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<td>4</td>
<td>Transport and radiative properties of smelt</td>
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<td>4.1</td>
<td>Construction of molten smelt expf device complete</td>
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<td>4.2</td>
<td>Acquisition of viscosity data complete</td>
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<td>Report on transport/radiative properties</td>
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<td>5</td>
<td>Characterization of syngas</td>
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<td>Initial syngas characterization complete</td>
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<td>Measurement of trace species complete</td>
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<td>5.3</td>
<td>Evaluation of syngas during upsets complete</td>
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<tr>
<td>5.4</td>
<td>Report on syngas characteristics</td>
<td>36</td>
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</table>
BUDGET DATA

As of the end of the quarter, approximately 8% of the overall project budget had been spent. As the project progresses and parts for the gasifier are purchased and that system is operated, spending will pick up. Table 2 shows the estimated expenditures for the current quarter, as well projected expenditures for the next quarter. As construction of the entrained-flow gasifier progresses, spending in that category will pick up notably. Estimates for subcontracts assume that the subcontractors will spend the difference between what was budgeted and what was spent to date proportionally over the remainder of the budget period.

<table>
<thead>
<tr>
<th></th>
<th>Expenditures 01/01/05 - 03/31/05</th>
<th>Budget Category</th>
<th>Next Quarter Projection 04/01/05 - 06/30/05</th>
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<td><strong>TOTAL</strong></td>
<td><strong>148,990</strong></td>
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<td>DOE (80%)</td>
<td>33,833</td>
<td>DOE (80%)</td>
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<tr>
<td>Cost Share (20%)</td>
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<td>Cost Share (20%)</td>
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<td><strong>TOTAL</strong></td>
<td><strong>42,292</strong></td>
<td><strong>TOTAL</strong></td>
<td><strong>148,990</strong></td>
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ACKNOWLEDGEMENTS

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