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Vendor Test Studies Supporting the Design of a Biomass-to-Ethanol Pilot Plant

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ABSTRACT

In support of the effort to develop the biomass-to-ethanol process, the National Renewable Energy Laboratory (NREL) is building a pilot plant based on the enzymatic conversion of cellulose to ethanol. The plant will incorporate operations for feed handling, size reduction, pretreatment, fermentation, distillation, and solids separation. Pilot plant testing of critical equipment at vendor facilities was undertaken to ensure good and reliable designs. Specifically, vendors tested pumping, agitation, and centrifugation of biomass slurries. Sulfuric acid pretreated wood was successfully pumped at solids concentrations up to 30%. Agitation of pretreated biomass slurries was investigated over a range of solids concentrations from 11 to 18.5%. Pretreated and fermented biomass slurries can be successfully dewatered in a centrifuge to a 30% solids concentration. Additionally, metals were tested for corrosion under conditions likely to be encountered in a dilute sulfuric acid prehydrolysis to identify suitable materials of construction for a pretreatment system. Corrosion rates were found to be highly dependent on temperature. Zirconium was the only material that had low corrosion rates at conditions of 2% sulfuric acid and 200°C.

Index Entries: Biomass; ethanol; pilot plant; pumps; centrifuge.

INTRODUCTION

The US Department of Energy (DOE) and the National Renewable Energy Laboratory (NREL) are installing a Process Development Unit (PDU) for the conversion of biomass to ethanol. It will serve as a user facility where private industry, academia, and government can cooperatively develop biomass conversion technology. The PDU will be used to verify process performance, investigate alternative process options, and develop scale-up data for design of large-scale demonstration or commercial facilities. The plant is designed to process 900 dry kg/d (1.0 dry t/d) of biomass, and will be flexible enough to accommodate a variety of process conditions and feedstocks.

Lignocellulosic biomass includes such materials as agricultural and forestry waste, municipal solid waste, waste paper, and wood and herbaceous energy crops. These materials represent one of the most abundant renewable resources on earth. Both the cellulosic and hemicellulosic fractions of biomass can be converted to simple sugars that can subsequently be fermented to ethanol. The conversion into ethanol of even a small portion of this resource into ethanol could substantially reduce gasoline consumption and dependence on petroleum.

Acid- and enzyme-based processes are the two most common methods of converting cellulose to simple sugars. Various processes based on dilute and concentrated acids hydrolyze the cellulose and hemicellulose to their respective sugars (*1–3*). Enzymatic processes involve a pretreatment step to hydrolyze the hemicellulose or dissolve the lignin, thereby increasing the digestibility of the cellulose. Then, cellulase enzyme is used to hydrolyze the cellulose, and both sugars (glucose and xylose) are subsequently fermented to ethanol (*4*).

A subcontract has been placed with John Brown Engineering and Construction to design and install an enzymatic-based biomass-to-ethanol pilot plant for NREL. Phase I of this effort involved vendor testing of critical equipment, preparing a conceptual process design, ordering long lead-time equipment, and completing preliminary engineering. The conceptual design was completed in March 1993, and preliminary engineering was completed in June 1993. After completion of Phase I, the subcontract was extended to include Phase II—the detailed design and installation of the PDU.

The pilot plant will be designed with the following unit operations: feedstock milling and conveying, pretreatment, fermentation, ethanol recovery, and solids separation. Feedstock is delivered to a mill for size reduction and then is conveyed to the pretreatment reactor. The choice of mills for this plant was discussed in a previous paper (*5*). In the pretreatment step,the feed is treated to increase the susceptibility of the cellulose to enzymatic conversion by cellulase. This treatment also hydrolyzes a large fraction of xylan to the five-carbon sugar xylose. The plant will initially use a dilute sulfuric acid pretreatment. Xylose can then be fermented to ethanol by a variety of microorganisms. A fraction of the pretreated cellulose stream is used as substrate by the fungi *Trichoderma reesei* to produce cellulase. The rest of the pretreated cellulose is sent to the cellulose conversion step, which uses the simultaneous saccharification and fermentation (SSF) process. Yeast, cellulase, and pretreated cellulose are all combined in the same fermenter. The enzyme hydrolyzes the cellulose to glucose, which is simultaneously fermented by the yeast to ethanol. The product stream from SSF is sent to distillation to remove ethanol from the fermentation broth.

VENDOR STUDIES: DESIGN SUPPORT FOR THE PILOT PLANT

The goal of vendor testing done during Phase I of this project was to generate design information for the NREL pilot plant. It is not the intent of this article to present the final process design, but to convey information obtained during the design phase from specific vendor test. Because these activities were early in the design process and owing to limitations at the vendor facilities for choosing operating conditions, the conditions (e.g., solids concentrations, temperatures, and so forth) reported may have little bearing on the final design of the plant. These tests were undertaken strictly to generate some information on certain aspects of the process and to increase the reliability and confidence in the final design. Obviously, there are still many operational parameters and unknowns associated with this technology. Since budget limitations allowed only limited testing, the choice of the most important areas for vendor testing was made by a team of John Brown and NREL engineers.

It was considered important that the plant have the ability to handle pretreated and fermented biomass slurries. Three critical activities whose operation depends on the nature of the biomass slurry and solids concentration are pumping, agitation, and centrifugation. Each of these operations were tested in vendor facilities with biomass slurries of varying solids concentration. Additionally, a corrosion study was performed to determine the effect of sulfuric acid on various metals at different temperatures.

The materials tested were dilute sulfuric acid pretreated wood chips and the same material subjected to the SSF process. Poplar chips were milled to approx 1-mm diameter by 5-mm long particles in a Sprout-Bauer 91-cm (36-in.) Double Disc Pressurized Refiner (Model 418) located in the Sprout-Bauer pilot plant facility in Springfield, OH. Additional information on the milling results (moisture content, particle size, and so on) is provided by Schell and Harwood (*5*). This material was sent to the Tennessee Valley Authority (TVA) pilot plant in Muscle Shoals, AL, where the material was pretreated in a Sunds vertical digester at conditions of approx 1.0% sulfuric acid and 160°C for 10 min. About 1200 kg of pretreated material at a 30% solids concentration were generated for testing. A fraction of this material was sent to NREL and subjected to the SSF process in a 150-L New Brunswick fermenter at 37°C. After removal of the liquid hydrolyzate from the pretreated wood, the wood was

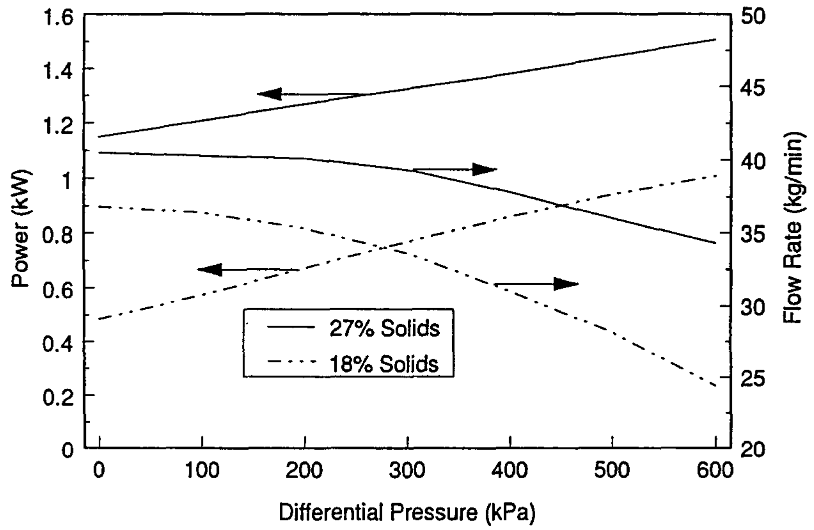

Fig. 1. Pump power and flow rate as a function of pump differential pressure for a treated biomass slurry at 18 and 27% solids concentration at a fixed pump speed of 200 rpm.

diluted with water to a 10% solids concentration in the fermenter. A relatively high cellulase loading of 25 IU/g cellulose and β-glucosidase at 10 IU/g cellulose was used to ensure adequate conversion. This material was then used for subsequent centrifuge testing.

Pump Testing

To test the feasibility of pumping pretreated wood, a pump test was conducted with a progressing cavity pump at the Robbins and Myers, Inc. pilot facility in Springfield, OH using a Model 1FFJ6 SSE AAA Moyno Sanitary Pump. The performance curves generated with this material are shown in Fig. 1 for 18 and 27% solids concentration at a fixed pump speed of 200 rpm. As expected, the power consumption increases with increasing solids concentration. However, the flow rate was lower at the lower solids concentration (18%), probably because of greater slippage within the pump. According to the manufacturer's data, the power requirement at 18% solids concentration is approx double the requirement for pumping water only. A 27% solids concentration appeared to be the maximum concentration that could be handled by this pump.

A pump trial was also conducted on a rotary-lobe pump in the machine shop of C. Doering and Son, Inc. in Westmont, IL. The pump was a Waukesha Model 34 Universal Rotary Lobe Pump fed by a twin-screw auger system. Shaft-power measurements were not available from this vendor, but testing showed that it was possible to pump pretreated wood at a 30% solids concentration. The auger feeder system was necessary to keep the pump supplied with wood; otherwise, the material bridged and dewatered at the pump throat.

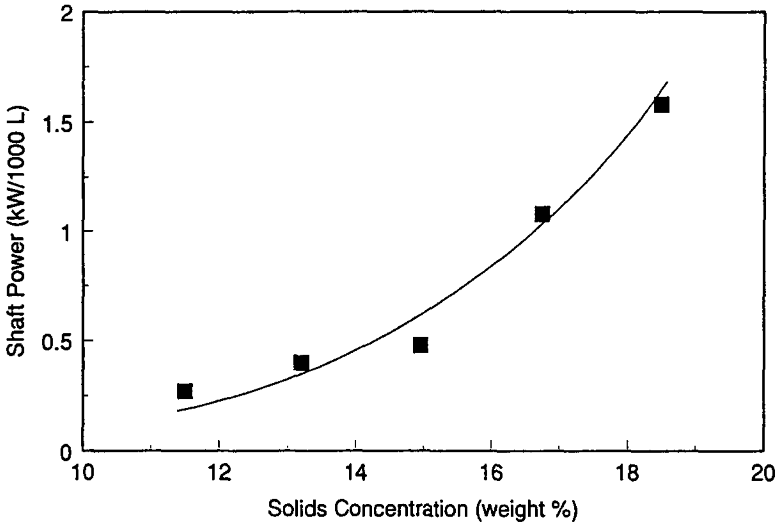

Fig. 2. Agitation power requirements for a pretreated biomass slurry as a function of solids concentration using a Lightnin A315 impeller.

Agitation Testing

Agitation tests were conducted on pretreated material by Lightnin in their Rochester, NY facility. Pretreated material was used to obtain data for the worst-case power consumption. As fermentation proceeds, solids concentrations will decrease because of hydrolysis of the cellulose during the SSF process, which will lower agitation power requirements. Shaft power as a function of solids concentration is shown in Fig. 2. Shaft power was obtained by the vendor from measurements of shaft speed and torque, and was defined as the amount of power required to provide good top-to-bottom turnover (a qualitative estimate of good particle movement from the top of the tank to the bottom). These results were obtained in an unbaffled tank (76 cm in diameter by 91 cm deep with a 61 cm liquid depth) with a single Lightnin 40.6-cm A315 impeller. The A315 impeller is typically supplied by Lightnin for fermentation applications. The power requirement at 18.5% solids concentration (near the upper limit for a mixable pretreated biomass slurry) was 1.58 kW/1000 L (8 hp/1000 gal). At the lowest solids concentration tested (11.5%), the power requirement was 0.27 kW/1000 L (1.35 hp/1000 gal). The addition of baffles to the tank approximately doubled the power requirements. The baffles were 5-cm wide and placed at 90° angles to each other. For a high-solids concentration (18.5%) slurry in an unbaffled tank, an aeration rate of 0.04 vvm was enough to cause flooding of the impeller. Baffles eliminated flooding up to 0.20 vvm, but the high air-flow rate resulted in a deterioration of the flow pattern.

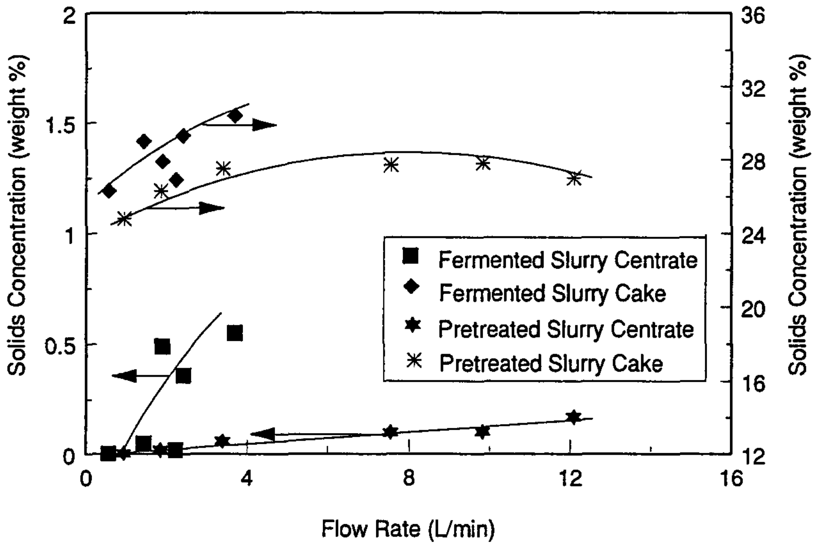

Fig. 3. Centrifuge cake and centrate solids concentration as a function of flow rate for a pretreated and fermented biomass slurry for the P-660.

Centrifugation Testing

Centrifugation tests were conducted on pretreated and fermented wood slurries on pilot-scale continuous decanters in the laboratories of Alfa-Laval Separations Inc., Sharples Division, in Warminster, PA and Centrico, Inc., in Northvale, NJ. Centrifugation would be required for removing xylose from the pretreated biomass stream for separate fermentation or for removal of solids from the fermented material, which could then be sent to a boiler to supply a plant with steam and electricity (*6*). Testing at Sharples was conducted on a Model P-660 Decanting Centrifuge. Pretreated wood was diluted to an 8% solids concentration, neutralized with lime, heated to 66°C, and fed to the centrifuge with a Moyno pump. Testing at Centrico was conducted on a Westfalia Model CA-150 Clarifier Decanter. Pretreated wood was also diluted to an 8% solids concentration, heated to 60°C, and fed by a Tri-Clover Ladish Model PR10 Rotary Positive Displacement Pump. For both machines, fermented material was fed as received from NREL.

The results on pretreated and fermented material for the Sharples P-660 and Centrico CA-150 are shown in Figs. 3 and 4, respectively. As expected, the pretreated wood is easier to separate than fermented wood. This is shown by the higher solids concentration found in the centrate for the fermented wood and is the result of the smaller particle sizes. The decrease in particle size during enzymatic hydrolysis has previously been documented (*7*). The limit of solids concentration in the cake appears to be about 30–32%. A different type of equipment, such as a filter or screw press, would be required to obtain a drier cake.

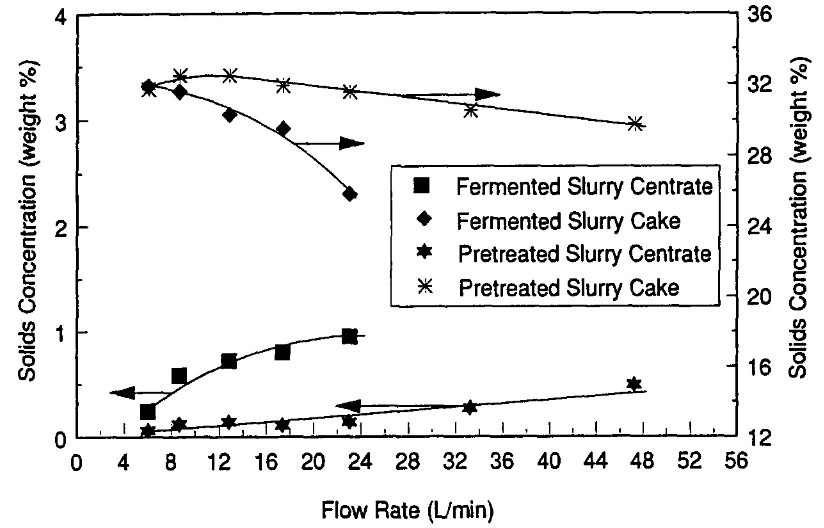

Fig. 4. Centrifuge cake and centrate solids concentration as a function of flow rate for a pretreated and fermented biomass slurry for the CA-150.

Corrosion Testing

Corrosion testing was performed to test various metals at conditions that might be encountered during the dilute sulfuric pretreatment of biomass. This testing was conducted at TVA to conform with the National Association of Corrosion Engineers Test Method TM-01-69. Corrosion rates were measured at four test conditions: 2.0% (w/w) sulfuric acid at 25°C, and 2.0% sulfuric acid and 0.1% formic acid at 104, 180, and 200°C. A minor amount of formic acid was added because it is a degradation product from the acid hydrolysis of cellulose. These conditions are associated with the feed, pretreatment reactor, and flash-tank environments of a pretreatment system. The conditions are conservative and are probably more severe than would normally be encountered using a dilute sulfuric acid pretreatment.

Specimens were welded circular disks (produced by welding two semicircular disks of the same material to each other) 2.54 cm in diameter and 0.635-cm thick. Before testing, specimens were degreased and weighed; after testing, specimens were cleaned and reweighed to calculate weight loss. The reported corrosion rate is an average value from two specimens. The specimens were immersed, using Teflon™ holders, in 1.2 L of solution. The test duration was 7 d at 25°C and 24 h for all other temperatures.

The results are presented in Table 1. No weld or galvanic corrosion was noted for any of the specimens, and when corrosion occurred, it was uniform. At 25°C, little corrosion occurred in any of the test specimens, indicating that any of the listed materials could be used at ambient temperatures. At 104°C, significant differences in corrosion rates were apparent

Table 1
Corrosion Rates for Various Metals

Test material[a]	Test temperature, °C	Corrosion rate µm/yr	Corrosion rate mils/yr[b]
Type 304L	25	8	0.3
Type 316L	25	5	0.2
Stellite 6	25	<3	<.01
Stellite 6 welded to 304L	25	5	0.2
Stellite 6 welded to 316L	25	5	0.2
Type 316L[c]	104	2840	112
Carpenter 20Cb-3	104	13	0.5
Carpenter 20Mo-6	104	13	0.5
Monel 400	104	686	27
Hastelloy B-2	104	381	15
Carpenter 20Mo-6	180	5920	233
Carpenter 20Mo-6	200	23,400	923
Monel 400	180	3230	127
Monel 400	200	20,100	791
Hastelloy B-2	180	508	20
Hastelloy B-2	200	813	32
Zirconium 705	180	10	0.4
Zirconium 705	200	13	0.5
Zirconium 702	180	8	0.3
Zirconium 702	200	10	0.4

[a] All materials tested in 2.0 wt% sulfuric acid.
[b] 1 mil = 0.001 in.
[c] Test solutions for 104, 180, 200°C data include 0.1% formic acid.

between the different metals. Type 316L has a severe corrosion rate, and even Monel 400 and Hastelloy B-2 have rather high rates. However, Carpenter 20 would be acceptable for this service. At 180–200°C, only zirconium would be acceptable for this service. The corrosion rates for all other metals were extremely high, and significant differences in rate were apparent between 180 and 200°C.

SUMMARY

This work was undertaken to reduce some of the uncertainties associated with building a pilot plant for the conversion of lignocellulosic biomass to ethanol. The objective was to test equipment with real material at conditions that are expected to be encountered during operation of the plant. Successful operation of pumps was observed at high solids concentrations (27–30%) on pretreated biomass slurries. Pretreated biomass slurries

can also be agitated at rather high solids concentrations (18%), but mixing requires large power inputs. Alternative equipment and designs may be required to reduce this power input. Pretreated and fermented biomass slurries were tested in pilot-scale decanting centrifuges. These tests were also successful and will be used to design a centrifuge for the NREL pilot plant. Corrosion testing showed that sulfuric acid can cause significant corrosion problems. Initially, the NREL plant will use materials with low corrosion rates to avoid problems of rapid equipment turnover and of significant amounts of corrosion products in the hydrolyzate that could inhibit the fermentation. The NREL pretreatment reactor was constructed from zirconium for this reason. The materials proposed for the pilot plant are not being considered for a commerical facility because of their high cost. Alternative materials or linings, as well as different technologies, will have a significant cost impact on the pretreatment system.

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