NATIONAL RENEWABLE ENERGY LABORATORY
GOLDEN, COLORADO

SUBCONTRACT ACO-9-29067-01
PROCESS DESIGN AND COST ESTIMATE OF CRITICAL EQUIPMENT IN THE BIOMASS TO ETHANOL PROCESS

REPORT 99-10600/14
LIQUID/SOLID SEPARATION

REVISION 1
MARCH 6, 2001
WEB

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Seattle, Washington 98109

REPORT 99-10600/14

LIQUID/SOLID SEPARATION

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LIQUID/SOLID SEPARATION

SUBCONTRACT ACO-9-29067-01 — **NATIONAL RENEWABLE ENERGY LABORATORY**
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1. EXECUTIVE SUMMARY

The National Renewable Energy Laboratory (NREL) under the direction of the Office of Fuels Development at the U.S. Department of Energy has, over the years, developed a process for converting cellulosic biomass to fuel ethanol based on NREL and subcontracted research and standard engineering practices. Three specific variations of the process were considered for the work in this subcontract: Pretreatment with Enzymatic Hydrolysis (P100), Two-Stage Countercurrent Acid Hydrolysis (P200), and Two-Stage Dilute Acid Hydrolysis (P300).

In Process 100, biomass feedstock in chip form is introduced to the plant and screened. The chips are passed to a scalper screen to remove very large materials and then onto a chip thickness sizing screen. We assume that approximately 20% of the incoming material will be oversized and will require processing through a singe disc refiner system. The disc refiner reduces oversized material to less than 19 mm, suitable for the pretreatment reactors. The biomass is pretreated to make it more susceptible to acid penetration. During pretreatment, much of the hemicellulosic portion of the biomass is hydrolyzed into soluble sugars in a continuous hydrolysis reactor. This reactor uses steam and dilute sulfuric acid to initiate hydrolysis. Afterwards, the liquid portion of the pretreated slurry must be separated from the solids to facilitate conditioning of the liquid portion to remove compounds, such as acetic acid, that may be toxic to downstream fermentative organisms. Once the liquid stream is conditioned properly (most likely via ion exchange and overliming), it is recombined with the solids and sent to fermentation.

This process uses simultaneous saccharification and co-fermentation (SSCF) to hydrolyze cellulose and ferment the resulting glucose and other sugars present to ethanol in the same vessel. As this design currently stands, a portion of the pretreated hydrolysate is drawn off and used to produce cellulase enzyme. The enzyme is then added to the fermentation vessels. A recombinant ethanologen is used to ferment multiple sugars to ethanol. The resulting beer is then sent to distillation and dehydration to purify and concentrate the ethanol. The lignin portion of the original biomass gets carried through the system and exits with the distillation bottoms. This lignin is used as fuel for the burner/boiler system in the plant. As a result, it must be dewatered sufficiently to achieve proper combustion.

Process 200 uses no acid in the first stage and a countercurrent reactor design in the second stage of hydrolysis to convert the hemicellulose and a large portion of the cellulose in the biomass to soluble sugars. The second-stage reactor separates the solids and liquor containing dissolved sugars. The solids are sent to the boiler and the liquid is sent to the oligomeric reactor and flash tank. The liquor is then neutralized and sent to fermentation. The back end of the process is the same as Process 100, but the process stream is liquid only.

Processes 200 and 300 differ from P100 in that no enzymes are used. All hydrolysis is accomplished thermochemically. In the first stage of hydrolysis, the hemicellulosic portion of the biomass is

hydrolyzed to soluble sugars. These sugars must then be washed from the slurry prior to the second stage of hydrolysis or else they will be degraded at the more severe conditions. The soluble sugar stream is neutralized (with stoichiometric amounts of lime) and sent to fermentation. The solids stream, primarily cellulose and lignin, is sent to the second stage of hydrolysis to further hydrolyze cellulose to glucose. After hydrolysis this stream is also sent to fermentation. The back end of the process is the same as Process 100.

Separation of solids from liquid streams and washing of the separated solids are required in the P100 and P300 processes. Liquid/solid separation is also required after distillation in the P100 process. NREL contracted with Harris Group Inc. (HGI) to do an interactive study with NREL engineers to identify liquid/solid separation equipment best suited to achieve the process goals, facilitate testing of that equipment, and develop associated costs for equipment alternatives that satisfy those goals.

The basis for the design and equipment sizing is a biomass feed rate of 2000 dry metric tons per day. Testing was done utilizing hardwood chip feedstock. Other feedstocks, including corn stover, sugar cane bagasse, softwood chips, and rice straw, will probably be commercialized before hardwood chips.

1.1 Post-Distillate Liquid/Solid Separation

The process objectives for liquid/solid separation equipment for P100 post-distillate slurry are to minimize moisture in the solids as well as minimize solids in the separated liquid. Centrifuges at Baker Hughes and Alfa Laval were tested. A Pneumapress pressure filter was also tested. The pressure filter produced cake solids of 88% by weight with total liquid solids of 2.97%, while the centrifuges produced cake between 20% and 26% solids with total liquid solids of 4.27% by weight. Based on budgetary equipment quotations, the estimated installed- cost for an Alfa Laval centrifuge system is $8,800,000. The estimated installed cost for the Pneumapress system is $8,100,000 with an estimated average power demand of 830 kW and an estimated annual power cost of approximately $332,000. The Pneumapress pressure filter is recommended as the best equipment for this application based on testing and equipment evaluation done to date.

1.2 Process P100 Ambient Pressure Liquid/Solid Separation

The process objective for liquid/solid separation for Process P100 is to minimize acetic acid carryover in the solids while limiting the wash water to 132,000 kg/hr (equals 0.58 lb water/lb feed based on 230,545 kg total feed to liquid/solid separation). An acetic acid level of 3.3 g/kg in solids was established as the required maximum with 1.65 g/kg as the desired level. Two horizontal belt filters, a pressure filter, and a filter press were tested.

The proposed Black Clawson horizontal belt filter would limit acetic acid carryover in solids to 1.7 g/kg with 300,000 kg/hr (1.29 lb water/lb feed) of wash water and an estimated installed cost of $27,000,000. The projected wash water flow significantly exceeds the 132,000 kg/hr limit. Black Clawson utilized test results to project the number of wash stages required to achieve the required acetic removal efficiency.

The proposed Baker Hughes horizontal belt filter used two stages of countercurrent washing to meet the targets for acetic acid in the final cake. Baker Hughes has not provided data defining the specific amount of acetic carryover in solids. The amount of wash water required is 1144 gpm, which is 15% above the maximum wash filtrate (989 gpm) allowed. The estimated installed cost of this option is $6,300,000.

The Pneumapress pressure filter produced a residual acetic acid level of 0.9 g/kg, which is below both the required and desired acetic acid levels. This acetic acid residual was achieved with 0.58 lb of wash water per lb of feed (or 133,000 kg/hr of wash water). The wash water flow is also very close to the wash water maximum required. The estimated installed cost of a Pneumapress pressure filter is $8,500,000 with estimated average power demand of 980 kW with an annual electrical power cost of $392,000.

The Pneumapress pressure filter is recommended as the best equipment for this application based on testing and equipment evaluation done to date.

1.3 Process P300 Ambient Pressure Liquid/Solid Separation

The process objective for liquid/solid separation for the P300 process is to maximize sugar recovery from the solids with 95% removal solids required and 98% removal desired. Two horizontal belt filters, a pressure filter, and a filter press were tested.

The proposed Black Clawson horizontal belt filter with six wash stages would remove 95% of the sugar in the solids. The estimated installed cost of the equipment is $49,700,000. Black Clawson utilized test results to project the number of wash stages required to achieve the required sugar removal efficiency.

The proposed Baker Hughes horizontal belt filter, with two stages of countercurrent washing, removed 95% of the sugar in the solids. This would require 1144 gpm of wash water, which is slightly above the maximum wash filtrate (1065 gpm) allowed. To increase the sugar recovery to 98% would require three stages of washing and 2204 gpm of wash filtrate. The estimated installed cost of this option is $6,300,000.

The proposed Pneumapress pressure filter would produce 97% sugar recovery. The estimated installed cost for a system with Pneumapress pressure filters is $8,500,000 with estimated annual electrical power cost of $392,000.

The Pneumapress pressure filter is recommended as the best equipment for this application based on testing and equipment evaluation done to date.

1.4 Process P100 Elevated Temperature Liquid/Solid Separation

Testing of a Pneumapress bench scale unit took place at NREL's facilities in Golden, Colorado. No quantitative analyses were done on resultant filtrate or cake solids from these tests. However, from qualitative observation, the results appeared to be promising. NREL has plans to perform some general liquid/solid separation on a pilot scale Pneumapress filter. If those tests are successful and if funds are available, a pilot scale unit would be purchased. This unit would be capable of operating at elevated temperature and pressure conditions.

2. OBJECTIVES

The key objective of this work is to improve the process design and accuracy of the estimate for segments of the process requiring liquid/solid separation. NREL engineers will incorporate the results of this study into the NREL model with the assistance of HGI. The design estimate is for the "*Nth*" plant to be built in order to eliminate costs associated with a "one-of-kind" or first system built.

3. DISCUSSION

Liquid/solid separation is required in the following process locations:

- After distillation in Process P100
- After first-stage hydrolysis reactor elevated temperature and pressure, Processes P100 and P300
- After first-stage hydrolysis reactor ambient pressures, Processes P100 and P300

All equipment considered for these process applications is current technology, commercially available, and in use in other industries in similar liquid/solid separation process functions. Report 10600/2, Liquid/Solid Separation Vendor Comparison, outlines the basis for equipment and vendor selection and can be found in Appendix F.

The following sections discuss process objectives; equipment options; test results; equipment-specific information including size, operating principles, and expected performance based on test data; as well as power requirements and the capital costs associated with each equipment option.

3.1 Liquid/Solid Separation Equipment

A range of liquid/solid separation equipment was bench scale tested for the various process applications. Following is a brief description of each equipment type.

3.1.1 Centrifuge

The decanter-type centrifuge use centrifugal force to accelerate the sedimentation of solid particles to be separated from a liquid. The suspension to be treated is fed into a rotor composed of a bowl and screw. These turn at high speed, the screw slightly faster than the bowl. The screw conveys and evacuates the decanted solids toward the conical end of the bowl while the clarified liquid is evacuated from the opposite end. Decanter centrifuges provide continuous (as opposed to batch) processing of slurry mixtures.

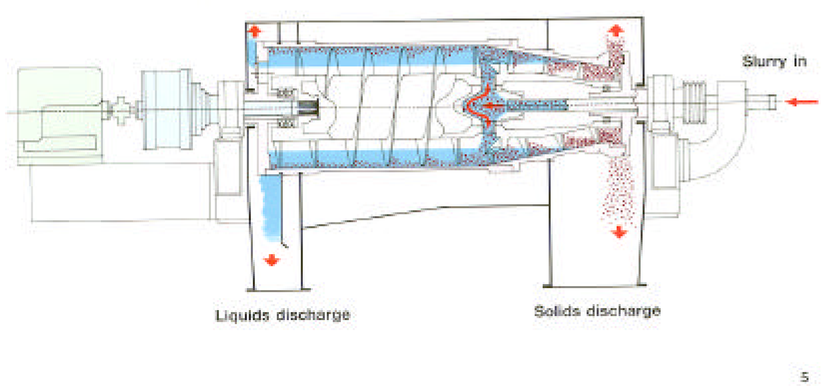

Figure 1.Centrifuge (Alfa Laval)

3.1.2 Filter Press

Filter presses (also called plate and frame presses) operate in a batch mode. First, a pneumatically controlled hydraulic pump applies high pressure to securely close the plates and seal them against internal bypass and/or excessive external leakage. As the illustration indicates, wet slurry is then pumped in through the center inlet and forced into chambers, which are formed by the vertically oriented matching recessed plates. The pumping action serves as the motive force to provide liquid/solid separation.

Vendors of this equipment offer various enhancements including diaphragm expression, heated water diaphragm expression, vacuum evaporation of liquid from the cake. Cake washing can be accomplished by introducing wash water to the cake after dewatering. Each plate has a filtrate drainage area that is covered with a cloth filter media that traps particles. As the solids build up, they act as filter medium, allowing only clear filtrate to pass through for discharge through the outlet ports. As the chambers fill with cake, the differential pressure increases to the maximum design limit and the stream of filtrate reduces. The plates are opened at the end of a filtration cycle and the cake is discharged.

Filter presses are used in a wide range of applications including minerals, pharmaceuticals, municipal sludge, and gypsum. Liquid/solid separation performances of filter presses vary significantly with the characteristics of the solids. In general, inorganic materials such as gypsum can be dewatered to higher degree than organic material. Size ranges of machines can vary from 2 ft^2 of filter area to over 10,000 ft^2 of filter area. Filter presses can often obtain dryer cake solids than other types of liquid/solid separation equipment. However, they are batch operation, can have large space requirements, and tend to be somewhat more expensive than alternative technologies. Because there is no positive mechanism for removing cake from filter presses, cake release characteristics should be verified for use of this equipment. It is possible to wash cake solids in a filter press by adding a wash cycle to the operation. Because the cake is oriented vertically, "short circuiting" of wash liquid can occur.

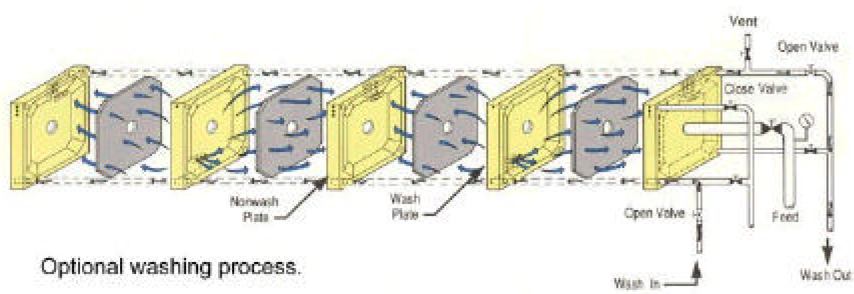

Figure 2. Filter Press (generic)

3.1.3 Belt Filter Press

A belt filter press is a dewatering device that applies mechanical pressure to slurry. The slurry is sandwiched between two tensioned belts by passing those belts through a serpentine of decreasing diameter rolls. The machine can actually be divided into three zones: gravity zone, where free draining water is drained by gravity through a porous belt; wedge zone, where the solids are prepared for pressure application; and pressure zone, where medium, then high, pressure is applied to the conditioned solids.

Typically, a belt filter press receives a slurry ranging from 1% to 4% feed solids and produces a final product of 12% to 50% cake solids. Performance depends on the nature of the solids being processed. Belt presses are commercially available in widths up to 3 meters. They are used in a wide range of industries including pulp and paper, municipal sewage sludge, and minerals processing. Advantages include continuous operation as opposed to batch and relatively low initial cost. Filter belts can be a significant operating cost, particularly where abrasive solids are being dewatered. Belt presses tend to obtain lower cake solids than other dewatering methods including filter presses, pressure filters, and in some cases centrifuges. Because of their “open” design, hooding and ventilation systems are required to contain fumes, odor, and moisture.

Figure 3. Belt Press (generic)

3.1.4 <u>Horizontal Belt Filter</u>

Horizontal belt filters operate in a continuous mode. These machines are best applied where washing of solids is required. Dewatering is accomplished at each wash stage with vacuum applied to the cake through the filter media from below. Both Thermo Black Clawson and Baker Hughes make horizontal belt filters. While construction details of these machines differ significantly, both use similar principles of operation. Feed is introduced to a filter belt (“wire” – see note below). Liquid is extracted through the filter belt, while solids are retained on the belt. Shower water is applied to solids to remove sugar and/or acetic acid utilizing multiple-stage countercurrent washing. Solids are discharged onto a conveyor. These machines can provide high washing efficiency with a relatively low wash-water-to-solids ratio. Liquid-to-solids wash ratios vary significantly, depending on the characteristics of the solids.

Note: Wire is a term used in the pulp and paper industry for the fabric media used to support and dewater incoming slurries. Fabric composition is typically synthetic fiber such as polypropylene that is suitable for the chemistry and temperature of the application.

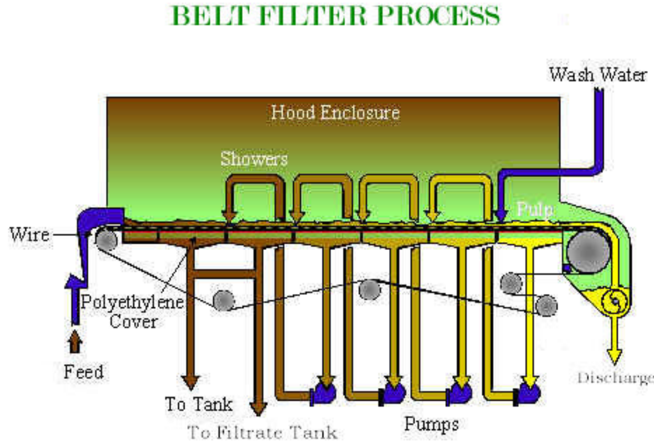

Figure 4. Horizontal Belt Filter (Thermo Black Clawson)

3.1.5 <u>Pneumapress Pressure Filter</u>

A Pneumapress pressure filter operates in a batch mode. Slurry is pumped into cavities formed by multiple horizontally oriented plates. Air pressure applied to solids captured on filter media drives liquid from the solids cake. The pressure filter provides batch liquid/solid separation as follows:

(1) Slurry is pumped into filter chambers formed by lowering the upper plate onto the filter media. Filtrate is collected at the lower plate and flows out through the filter outlet.

(2) Compressed air or gas forces the liquid from the solids retained on the filter media and dries the solid "cake." The cake may be washed after initial liquid/solid separation.

(3) The upper plate raises and the filter media indexes forward, discharging the cake cycle to the operation.

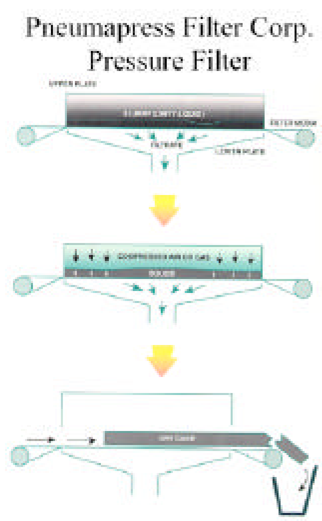

Figure 5. Pressure Filter (Pneumapress Press Filter Corp.)

Because the cake is oriented horizontally, efficient displacement washing is possible. Pressure filters are utilized in a wide range of applications and industries including gypsum, food, pharmaceuticals, and power. The Pneumapress pressure filter is capable of obtaining very high cake solids, depending on solids characteristics. It can also be utilized for high (greater than atmospheric) temperatures and pressures.

3.1.6 Extractor

Crown Iron Works makes an "extractor" that utilizes countercurrent wash flow to extract soluble components of feedstock. A sample of the hydrolyzate slurry was sent to Crown Iron Works for evaluation. The feed slurry has a solids concentration of about 28%. Crown stated the maximum discharge solids concentration that could be expected from its machine would be 15% to 18%. Hence, without additional liquid/solid separation equipment, this equipment would not function in this application. Based on this information, the extractor was not considered to be viable technology for this application.

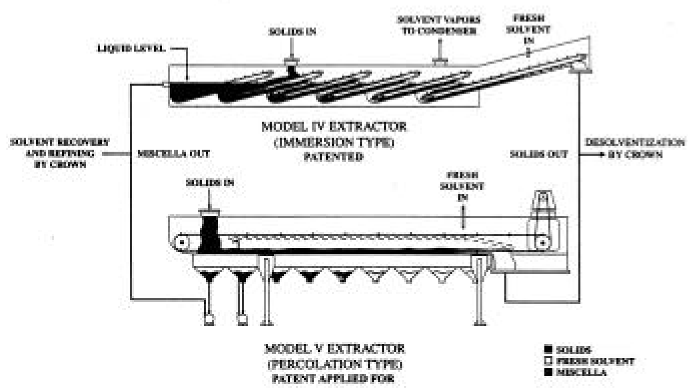

Figure 6. Crown Iron Works Extractor

3.2 Liquid/Solid Separation After Distillation

Liquid/solid separation of the bottoms after the first-effect evaporator is required in the P100 process. The solid fraction generally consists of lignin and unreacted cellulose, while the liquid fraction is primarily water. Process objectives include the following:

- Production of solids with minimum moisture content – The solids are used as a fuel in a burner. Waste stack heat will be utilized to evaporate the remaining water in the solids. One of the process objectives is to minimize the amount of boiler heat used in drying solids after liquid/solid separation.
- Production of filtrate with minimum solids – This water is fed back to fermentation. A minimal amount of solids can be accommodated in the fermentation process.

3.2.1 Materials of Construction

This is a moderate- to low-corrosion application. Sulfuric acid concentration in the feed slurry is approximately 0.1%. Standard industrial corrosion-resistant metallurgy such as Type 316 stainless steel is appropriate for wetted components of metal process equipment where temperatures are maintained below approximately 92ºC at this acid concentration. The feed stream temperature is 86ºC for this process step.

3.2.2 Test Material

Test material for post-distillate liquid/solid testing was prepared as described in the NREL report entitled *Experimental Plan EPD0002 – 100L SSF of Pretreated Yellow Poplar* and can be found in Appendix F.

3.2.3 Equipment/Vendor Options

A range of equipment is available for this application. Centrifuges are currently shown on NREL's process flow diagram. The options that were tested include centrifuges, filter presses, belt presses, and pneumatic pressure filters. Three vendors were selected to test equipment to determine the optimal technology to achieve the process goal. Report No. 10600/2, Liquid-Solid Separation (Appendix F) outlines the vendor selection basis. The following vendors and equipment options were tested for this application:

Manufacturer	Equipment
Alfa Laval Separation	Centrifuge
Baker Hughes	Centrifuge, filter press, belt press
Pneumapress Filter Corporation	Pressure filter

3.2.4 Test Results

A summary of test results for each of the vendor tests is provided below. Test reports, data, and an NREL sample analysis for each of the tests may be found in the applicable vendor appendix.

3.2.4.1 Centrifuge

Alfa Laval and Baker Hughes performed a bench scale "spin tests" of the post-distillate slurry. Spin tests generally provide an indication of feasibility of liquid/solid separation of slurry. The solids concentration obtained in a full-size machine is generally better than can be achieved in a laboratory test. (See Table 1.)

3.2.4.2 Filter Press

Baker Hughes performed laboratory bench scale liquid/solid separation tests with post-distillate slurry for its filter press. The results are summarized in Table 2. NREL analysis of samples from these tests may be found in Appendix B.

Table 1
Centrifuge Spin Tests

Item	Unit	Value	
		Alfa Laval	Baker Hughes
Feed slurry			
Total solids	% by weight	7.26	no data
Total suspended solids	% by volume	23	no data
Viscosity	cP	13.5	no data
Test conditions			
Spin time	minutes	1.5	no data
Centrifugal force	G's	1,500	no data
Slurry temperature	°C	86	no data
Separated streams			
Cake total solids	% by weight	19.9 [1]	22.9 [2]
Cake description		Firm	Heavy mud consistency
Liquid total solids	% by weight	4.26 [1]	3.07 [2]
Liquid dissolved solids	% by weight	3.54 [1]	2.83 [3]

[1] Alfa Laval test data
[2] NREL analysis of Baker Hughes samples, average value of three tests
[3] Baker Hughes test data

Table 2
Baker Hughes Filter Press

Item	Unit	Value
Feed slurry		
Total solids	% by weight	no data
Total suspended solids	% by volume	no data
Viscosity	cP	no data
Test conditions		no data
Separated streams		
Cake total solids (Option A)	% by weight	range 34.9 – 39.7 [1]
Cake total solids (Option B)	% by weight	range 39.7 – 44.4 [1]
Liquid total solids	% by weight	2.67 [2]
Liquid dissolved solids	% by weight	no data

[1] Baker Hughes test data
[2] Results of a single test

3.2.4.3 Belt Filter Press

Baker Hughes did bench scale liquid/solid separation testing of a belt press. The Baker Hughes report excludes any data or discussion of results. The belt press data in Table 3 is from an NREL analysis of samples from belt press testing of post-distillate slurry that Baker Hughes did. It is notable that polymer was utilized to dewater these solids on the belt press. See Baker Hughes' report dated 2/5/01 (Appendix B) for additional test results.

3.2.4.4 Pressure Filter

Pneumapress Filter Corporation did bench scale liquid/solid testing of the pressure filter at its facility in Richmond, California (Table 4). Cake solids and filtering time are both significantly affected by cake thickness. Cake thickness is controlled by the amount of slurry introduced per unit area of filter. Hence, equipment sizing is directly affected by this cake thickness.

3.2.4.5 Test Comparison Summary

Table 5 provides a summary of average percent solids by weight for cake and filtrate/centrate products from testing.

3.2.5 Equipment Costs

Below are capital costs for the Pneumapress and centrifuge options. Capital cost information will be provided for belt presses and filter presses when it becomes available from Baker Hughes. Pneumapresses are the recommended technology. Hence, a more detailed capital cost estimate has been developed for this equipment. All estimates exclude the costs of buildings. Electrical energy costs are provided for a Pneumapress installation.

3.2.5.1 Centrifuge

Alfa Laval states that the laboratory scale tests for the centrifuge do not provide sufficient information on definitive sizing of equipment for the process flows. However, it does provide a good indication of the viability of centrifuges for the application, the type of centrifuge to apply to the process, and a general idea of the level of liquid/solid separation that can be accomplished with centrifuge technology. Based on the results of the tests, Alfa Laval estimates that between five and eight P7600 centrifuges would be required. Alfa Laval suggested that using seven machines as a basis for a capital cost estimate would provide an appropriately conservative estimating approach. The price does not include auxiliary equipment such as pumps, tanks, and conveyors. HGI estimates an installed cost for this system to be $8,800,000. A preliminary equipment list is included in Appendix A.

Table 3
Baker Hughes Belt Press Tests

Test	Item	Unit	Value
D-1	Cake solids	% by weight	27.32
D-2	Cake solids	% by weight	26.90
D-2F	Cake solids	% by weight	34.16
D-3	Cake solids	% by weight	24.74
D-4	Cake solids	% by weight	25.33
D-4E	Cake solids	% by weight	28.39
	Cake solids	avg % by weight	27.81
D-1	Gravity drain liquor	% by weight	2.48
D-2	Gravity drain liquor	% by weight	2.35
D-4	Gravity drain liquor	% by weight	2.40
	Gravity drain liquor	avg % by weight	2.41

Table 4
Pneumapress Pressure Filter

Item	Unit		
Test run		D1	D2
Slurry introduced to pressure filter	ml	240	100
Air pressure	psig	100	100
Slurry temperature	°C	80 to 86	80 to 86
Time for filtrate to clear	seconds	120	20
Blowdown time	seconds	60	30
Cake thickness	in.	½	5/32
Filtrate quality	--	Clear	Clear
Cake total solids	% by weight	41.96	88.04
Cake description		firm w/wet surface	very firm w/dry surface
Liquid total solids	% by weight	2.87	2.95
Filter area	in^2	3.14	

Table 5
Percent Total Solids by Weight

	Alfa Laval		Baker Hughes		Pneumapress	
	Cake	Liquid	Cake	Liquid	Cake	Liquid
Centrifuge	19.9%	4.26%	22.09%	3.07%	---	---
Belt press			27.81%	2.41%		
Filter press			34.9%–39.7% [2] 39.7%–44.4% [3]	2.67%		
Pressure filter					88.04% [1]	2.95%
Feed slurry	% by wt	7.26%	Measured by Alfa Laval (See Appendix A)			

[1] Based on 5/32-in. cake
[2] Option A
[3] Option B

3.2.5.2 Pressure Filter

Pneumapress provided a budgetary quotation for this application. Four Pneumapress pressure filters would be required for the configuration that provided 88% cake solids during testing. An equipment list and cost estimate may be found in Appendix D. The estimated installed cost for a liquid/ solid separation system utilizing Pneumapress pressure filters is $8,100,000. Cycle times were considered in sizing equipment.

For an application with 50% of the current design capacity (flow rate to the pressure filter in kg/hr), two of the four pressure filters would be eliminated. Hence, the total estimated cost of a liquid/solid separation for 50% of current design capacity is $4,400,000.

For an application with 150% of the current design capacity, six pressure filters would be required. It is estimated that the total installed cost for liquid/solid separation for a plant with 150% of the original design capacity cost would be $11,613,000.

The estimated installed horsepower for a Pneumapress installation is 1490 hp with an estimated average power demand of 830 kW. Assuming $0.05/kW-hr and 8000 hours of operation per year, the annual electrical energy operating cost is estimated to be $332,000. Horsepower for this equipment can be scaled linearly with feed rate to the equipment.

3.2.5.3 Filter Press

Baker Hughes provided budgetary quotations for two equipment options. The first (Option A) was for five Model 2000FBM-103-PP-RP-HS-225-32 mm filter presses complete with accessories and controls. This option is a non-membrane filter press that is expected to achieve cake solids of between 37.3% and 39.7%. Estimated installed equipment cost for this system is $17,300,000.

Option B is for five Model 2000FBM-106-PP-mem/RP-HS-225-32 mm with accessories and controls. This option is a membrane-type filter press that is expected to achieve cake solids of between 39.7% and 44.4%. Estimated installed equipment cost for this system is $24,500,000.

3.3 Liquid/Solid Separation First-Stage Hydrolysis, Ambient Pressure/Temperature, Processes P100 and P300

Liquid/solid separation is required after the first-stage hydrolysis reactor in the P100 and P300 processes. The solid fraction consists of unreacted solids. The process objectives of this liquid/solid separation are as follows:

- **Process P100** – Remove the maximum amount of toxins from the solids as practical. Toxins consist of acetic acid and other soluble fermentation toxins. The amount of wash water required to accomplish this should be limited to a maximum of 132,000 kg/hr

(0.58 lb water/lb feed) because this is the total amount of water added to fermentation. The required level of residual acetic acid in 3.3 g/kg in solids with a desired level of 1.65 g/kg.

- **Process P300** – Remove soluble sugar from the solids. Sugar that carries forward to the second-stage hydrolysis will be destroyed. The desired level of sugar recovery from the feed slurry is 95% with a desired level of 98%.

3.3.1 Test Material

First-stage hydrolyzate slurry utilized for these tests was generated from pulp-size aspen hardwood chips at the TVA facility in Muscle Shoals, Alabama. The test material was made in a zirconium-lined digester with a liquid/solids ratio of 4:1 and an acid concentration of 0.55% in the liquid phase. The test was conducted at a temperature of 173°C and a pressure of 112 psig with a retention time of 15 minutes.

TVA collected slurries from first-stage hydrolysis and analyzed them for sugar content, acetic acid, HMF, furfural, and moisture content. The average moisture content was 59.7% and 59.3% for each of two samples tested. The results of the analysis for the remainder of the parameters are given in Table 6.

Table 6
Composition of First-Stage Hydrolyzate Slurry from Pilot Plant in mg/ml

	Drum 11	Drum 13	Drum 14	Drum 16
Glucose	9.90	11.60	11.60	10.70
Xylose	76.20	62.90	80.00	82.00
Galact.	<1.25	<1.25	---	<1.25
Arab.	2.25	1.90	1.90	2.25
Mann.	11.70	8.40	9.33	11.70
Acetic acid	21.10	18.00	19.80	21.20
HMF	0.29	0.36	0.31	0.31
Furfural	1.60	1.78	1.74	1.60

Note: The description of how material was produced and these results were extracted from TVA's April 14, 1999 report, Acid Hydrolysis Support, First And Second Stage Hydrolysis Testing, MPO No. DCO-8-18081-0 (Appendix F).

3.3.2 Materials of Construction

The P100 and P300 liquid/solid separation functions are moderate corrosion applications. Sulfuric acid concentration in the feed slurry is approximately 0.4%. Standard industrial corrosion-resistant metallurgy such as Type 316 stainless steel is appropriate for wetted components of metal process equipment where temperatures are maintained below approximately 82ºC at this acid concentration. Because both the P100 and P300 filtrate streams are processed below this temperature downstream of liquid/solid separation, the process stream temperature can be dropped prior to liquid/solid separation to allow use of 316 stainless steel.

3.3.3 Equipment/Vendor Options

Because the primary goal for both the P100 and P300 processes is solids washing with a limited amount of water, displacement countercurrent washing is a desirable approach to get the best washing results with limited wash water. Equipment types were limited to those that could do countercurrent washing. The equipment options tested for this application include horizontal belt filter, pressure filter, and filter presses. Screw presses, belt presses, and decanter centrifuges are not good candidates for this application, because it is not possible to do displacement washing with these machines. The following vendors tested equipment for this application:

Manufacturer	Equipment
Thermo Black Clawson Inc.	Horizontal belt filter
Baker Hughes	Horizontal belt filter, filter press
Pneumapress Filter Corporation	Pressure filter
Crown Iron Works	Extractor

3.3.4 Test Results

3.3.4.1 Horizontal Belt Filter, Processes P100 and P300

Black Clawson performed bench scale laboratory tests to simulate its Chemi-Washer for both P100 and P300 conditions. Tests were run three times at each condition. Table 7 shows averages of the results for the three test conditions. Black Clawson's complete test report may be found in Appendix C.

Baker Hughes also did laboratory testing to simulate its horizontal belt filter. However, the Baker Hughes report is not presented in sufficient detail to allow any analysis of its data for this equipment.

3.3.4.2 Pressure Filter

Table 8 presents a summary of the Pneumapress hydrolyzate test results.

3.3.5 Equipment Costs

3.3.5.1 Horizontal Belt Filter, P100 Process

Black Clawson provided a budgetary quotation for Chemi-Washers for the P100 application. Three 8-meter-wide x 20-meter-long machines were proposed. Equipment sizing, number of wash stages, and the volume of wash water required for the applicable level of washing efficiency are determined from extrapolation of test results.

HGI estimates the installed cost for this equipment and associated auxiliary equipment to be $27,000,000. A preliminary equipment list with order-of-magnitude prices for auxiliary items is included in Appendix C.

Baker Hughes provided a budgetary quotation for a horizontal belt washer for the P100 application. Based on test results, Baker Hughes estimates that 1144 gpm (1.1 to 1.2 lb/lb feed slurry) would be required to achieve the target acetic acid residual level. Baker Hughes proposed three Model 4.2 127 EIMCO-Extractor horizontal belt filters with accessories and controls. The estimated installed cost for this equipment and associated auxiliary equipment is $6,300,000.

Table 7
Summary of Black Clawson Hydrolyzate Test Results

Test	No. of Wash Stages	Wash Water (lb water/lb feed)	Feed Slurry Temp (°C)	Wash Water Temp (°C)	Cake Solids (% Dry Weight)	Cake-Washing Effectiveness			
						Glucose Removal Efficiency (%)	Xylose Removal Efficiency (%)	Acetic Acid Removal Efficiency (%)	Residual Acetic Acid (g/kg OD solids)
1	no wash		85		31.58				52.36
2 [1]	1	0.59	85	46	32.00	69.3	69.7	65.5	18.07
3 [1]	1	1.11	85	46	31.58	75.3	76.0	72.4	14.44
4 [2]	1	1.11	85	63	38.76	82.4	82.4	92.0	9.89
5 [1]	4	1.09	85	46	29.84	92.0	92.5	91.7	4.35
6 [2]	4	0.86	85	63	29.75	92.2	92.8	92.5	3.94
7 [2]	4	1.11	85	63	30.42	93.8	94.5	94.5	2.90
8 [2]	4	0.59	85	63	30.73	88.7	89.1	88.4	6.10

[1] Process 100
[2] Process 300

Black Clawson
Summary Test Results for Chemi-Washer

Process P100 level required	Acetic acid 3.3 g/kg in solids*
Process P100 level desired	Acetic acid 1.65 g/kg in solids*
Process P300 level required	95% removal of sugar (X and G)**
Process P300 level desired	98% removal of sugar (X and G)**

* Analysis of test results showed none of the P100 tests produced acetic acid levels at or below 3.3 g/kg. Test 5 produced 4.35 g/kg. More wash stages and/or more wash water are required for greater acetic acid removal.

** Analysis of test results showed none of the P300 tests produced 95% or greater washing efficiency of sugars. Test 7 produced 94.5%and 93.8% washing efficiency for both X and G sugars, respectively. More wash stages and/or more wash water are required for greater sugar removal.

Table 8
Summary of Pneumapress Hydrolyzate Test Results

Test	No. of Washes	Wash Water (lb water/lb feed)	Feed Slurry Temp (°C)	Wash Water Temp (°C)	Cake Solids (% Dry Weight)	Cake-Washing Effectiveness: Glucose Removal Efficiency (%)	Xylose Removal Efficiency (%)	Acetic Acid Removal Efficiency (%)	Residual Acetic Acid[3] (mg/ml)
1	0	0	85	--	54.55	45%	45%	45%	12.6
2 [1]	1	0.67	85	47	49.68	57%	57%	54%	10.6
3 [2]	1	0.73	85	63	52.58	71%	71%	70%	7.5
4 [2]	1	0.91	85	63	51.08	64%	65%	63%	8.7
7	2	1.09	80	no data	55.72	97%	98%	97%	0.5
7A	2	1.09	no data	"	57.09	96%	97%	96%	0.8
8	2	0.87	85	"	54.70	96%	97%	97%	0.8
9	2	0.58	85	"	54.76	95%	96%	95%	0.9

[1] Process 100
[2] Process 300
[3] Value reported from NREL laboratory analysis of samples

Pneumapress
Summary of Test Results

Process P100 level required	Acetic acid 3.3 g/kg in solids*
Process P100 level desired	Acetic acid 1.65 g/kg in solids*
Process P300 level required	95% removal of sugar (X and G)**
Process P300 level desired	98% removal of sugar (X and G)**

* Analysis of test results showed that with two wash stages and 0.58 lb wash water per lb of feed, the residual level of 0.9 g/kg acetic acid was achieved, below both required and desired levels.

** Analysis of Test 8 results showed that 96% glucose and 97% xylose removal was achieved with two wash stages and 0.87 lb of wash water/lb of feed. More wash stages and/or more wash water would be required to obtain 98% sugar removal.

3.3.5.2 Horizontal Belt Filter, P300 Process

Black Clawson provided a budgetary quotation for Chemi-Washers for the P300 application. Four 10-meter-wide x 22-meter-long machines were proposed. Based on the test results and Black Clawson's projections of washing efficiency, to achieve a sugar removal of 95% from the washed solids six wash stages each for each machine are required. The washers would be run with 1105 gallons of wash water per bdst (bone dry standard ton) of feed. Equipment sizing, number of wash stages, and the volume of wash water required for the applicable level of washing efficiency are determined from extrapolation of test results. HGI estimates the installed cost for this equipment and associated auxiliary equipment to be $49,700,000.

Baker Hughes provided a budgetary quotation for a horizontal belt washer for the P300 application. Based on test results, Baker Hughes estimates that 1144 gpm (1.1 to 1.2 lb/lb feed slurry) would be required to achieve 95% sugar removal. Baker Hughes proposed three Model 4.2 127 EIMCO-Extractor horizontal belt filters with accessories and controls. The estimated installed cost for this equipment and associated auxiliary equipment is $6,300,000.

3.3.5.3 Pressure Filter, P100 and P300 Processes

Pneumapress provided a budgetary quotation for these applications. The proposed equipment for the P100 and P300 processes is identical. Three Pneumapress pressure filters would be required with a total of 1080 sq ft of filter area. A simplified block flow diagram, equipment list, and order-of-magnitude capital cost estimate may be found in Appendix D. The estimated installed cost for the Pneumapress pressure filter system is $8,500,000. Cycle times were considered in sizing equipment.

For an application with 50% of the current design capacity (flow rate to the pressure filter in kg/hr), one of the three pressure filters would be eliminated. Each of the remaining two pressure filters would have a throughput capacity of 75% of each of the machines for the 100% capacity case. Hence, the total estimated cost of a liquid solid separation for 50% of current design capacity is $4,151,000.

For an application with 150% of the current design capacity, five pressure filters would be required, four having the same capacity as each of the machines for the 100% case and one having 50% capacity of that capacity. Hence, it is estimated that the total installed cost for liquid solid separation for a plant with 150% of the original design capacity cost would be $10,880,000.

The estimated installed horsepower for a Pneumapress installation for either process P100 or P300 is 1755 hp with an estimated average power demand of 980 kW. Assuming $0.05/kW-hr and 8000 hours of operation per year, the annual electrical energy operating cost is estimated to be $392,000. Energy costs are expected to vary linearly with the equipment throughput capacity.

3.4 Liquid/Solid Separation First-Stage Hydrolysis, Elevated Pressure/Temperature, Process P100

As an alternative to ambient pressure washing, liquid/solid separation could be provided after the first-stage hydrolysis reactor at an elevated temperature to keep lignin in solution. The expected temperature is 135ºC. Washing is expected to be required.

The process objective is to remove as much of the toxins from the solids as practical. The amount of wash water required to accomplish this should be limited to a maximum of 132,000 kg/hr (.58 lb wash water/lb feed) , because this is the total amount of water added to fermentation. If washing proves to be too difficult, cooking could be done at a slightly lower temperature. Potentially, liquid/solid separation could be done without washing.

3.4.1 Test Material

See description of test material in paragraph 3.3.1.

3.4.2 Materials of Construction

Type 316 stainless steel, as recommended for the ambient pressure liquid/solid separation application, is not suitable for 0.4% H_2SO_4 at a temperature of 135ºC. While no condition-specific testing has been done, testing was done by InterCorr International for a 0.6% H_2SO_4 at 190ºC. Alloy 825 exhibited a corrosion rate of 8 mpy in these tests. Normally, 5 mpy is considered an acceptable corrosion rate. Because H_2SO_4 corrosion is strongly affected by temperature, it is likely that Alloy 825 will function satisfactorily at 135ºC. Testing at the actual process conditions of Alloy 825 and testing with other less costly materials are recommended to verify acceptability.

3.4.3 Equipment/Vendor Options

Pneumapress Filter Corporation's pneumatic pressure filter has been identified as equipment that is capable of liquid/solid separation and countercurrent batch washing at pressures and temperatures above the boiling point of the slurry. The Pneumapress filter is capable of high cake solids and good washing with clear filtrate. Countercurrent washing would be done with multiple washes in a batch operation. See paragraph 3.1.3.5 for a schematic of the pressure filter.

3.4.4 Test Results

Testing of a Pneumapress bench scale unit took place at NREL's facilities in Golden, Colorado. No quantitative analyses were done on resultant filtrate or cake solids from these tests. However, from qualitative observation, the results appeared to be promising. NREL has plans to perform some general liquid/solid separation on a pilot scale Pneumapress filter. If those tests are successful and if funds are available, a pilot scale unit would be purchased. This unit would be capable of operating at elevated temperature and pressure conditions.

4. CONCLUSIONS AND RECOMMENDATIONS

4.1 Liquid/Solid Separation After Distillation

The objective of this process function is to dewater post-distillate solids to produce minimum moisture for a fuel burner with maximum heat value. At this writing, Baker Hughes has not yet satisfactorily completed testing that would allow evaluation of its belt filter press and filter press. Table 9 provides a comparison of representative test results with associated estimated installed equipment costs.

Table 9
Post-Distillate Comparison of Test Results and Estimated Direct Capital Equipment Costs

Equipment	Manufacturer	Cake Solids	Estimated Installed Cost
Centrifuge	Alfa Laval	19.9%	$8,800,000
Centrifuge	Baker Hughes	22.9%	No data
Filter press (Option A)	Baker Hughes	34.9%–39.7%	$17,300,000
Filter press (Option B)	Baker Hughes	39.7%–44.4%	$24,500,000
Pressure filter	Pneumapress	88.0%	$8,100,000
Belt Filter press	Baker Hughes	no data	No data

Based on the data available from testing done to date, the Pneumapress pressure filter produced dramatically drier cake compared to either Alfa Laval or Baker Hughes centrifuges and at essentially the same installed capital cost of the centrifuges. While full-size centrifuges generally develop somewhat drier cake than laboratory scale units, it is unlikely that they would come close to the cake solids exhibited by the Pneumapress pressure filter testing. The Pneumapress pressure filter is recommended as the best equipment for this application based on testing and equipment evaluation done to date. It is recommended that testing be repeated on a larger scale Pneumapress to confirm the repeatability of these results and optimize the process approach.

4.2 Liquid/Solid Separation First-Stage Hydrolysis, Ambient Pressure/Temperature, Process P100

The objective of this process function is to minimize carryover of acetic acid in hydrolyzate solids. At this writing Baker Hughes has not yet satisfactorily completed testing that would allow evaluation of its horizontal belt filter. Baker Hughes provided a capital cost estimate for its equipment; however, without reliable test results, there is no reliable basis for equipment sizing. Table 10 provides a comparison of representative test results with associated estimated installed equipment costs.

Table 10
P100 Comparison of Test Results and
Estimated Direct Capital Equipment Costs

Equipment	Manufacturer	*Wash Water (lb/lb feed)	Acetic Acid Residual (mg/ml)	Estimated Installed Cost
Horizontal belt filter	Baker Hughes	1.1 to 1.2	No data	$ 6,300,000
Horizontal belt filter	Black Clawson	1.1	1.7	$27,000,000
Pressure filter	Pneumapress	0.58	0.9	$ 8,500,000

*Feed as received from hydrolysis

Based on the test results and Black Clawson's projections of washing efficiency, to achieve the threshold acetic acid level of 1.7 g/l in the washed solids, five wash stages each for each machine are required. Per Black Clawson, the washers would be run with 940 gallons of wash water per bdst (bone dry standard ton) of feed. That is the equivalent of 1.1 kg of wash water per kg feed or approximately 300,000 kg/hr. That is far in excess of the limit of 132,000 kg of water per hour. Similarly, Baker Hughes' belt filter uses even more wash water.

The Pneumapress pressure filter produced significantly lower acetic acid residual with 0.58 lb wash water/lb of feed. That is approximately equal to 133,000 kg/hr of wash water. In addition, a Pneumapress installation has substantially lower capital cost than the Black Clawson horizontal belt filter. It is recommended that the Pneumapress pressure filter be selected for use in future work in this application based on superior performance and capital cost for equipment tested to date.

4.3 Liquid/Solid Separation First-Stage Hydrolysis, Ambient Pressure/Temperature, Process P300

The objective of this process function is to remove sugar from hydrolyzate solids. At this writing Baker Hughes has not yet satisfactorily completed testing that would allow evaluation of its horizontal belt filter. Baker Hughes provided a capital cost estimate for its equipment; however, without reliable test results, there is no reliable basis for equipment sizing. Table 11 provides a comparison of representative test results with associated estimated installed equipment costs.

Table 11
P300 Comparison of Test Results and
Estimated Installed Equipment Costs

Equipment	Manufacturer	*Wash Water (lb/lb feed)	Sugar Removal Efficiency (% Glucose/% Xylose)	Estimated Installed Cost
Horizontal belt filter	Baker Hughes	1.1 to 1.2	95%	$ 6,300,000
Horizontal belt filter	Black Clawson	1.3	95%/95%	$49,700,000
Pressure filter	Pneumapress	0.87	96%/97%	$ 8,500,000

*Feed as received from hydrolysis

The Pneumapress pressure filter produced significantly higher sugar removal with less wash water and at substantially lower capital cost than the Black Clawson horizontal belt filter. The Baker Hughes horizontal belt filter did not have adequate test data to justify a recommendation by HGI. If Baker Hughes could produce test data to back up its claims, this horizontal belt filter may be worth consideration. However, it does appear that both horizontal belt filters will require high wash water to obtain 98% sugar removal. It is recommended that the Pneumapress pressure filter be selected for use in future work in this application based on superior performance and capital cost for equipment tested to date.

4.4 Liquid/Solid Separation First-Stage Hydrolysis, Elevated Pressure/Temperature, Process P100

All testing for liquid/solid separation at elevated temperature took place at NREL facilities utilizing a bench scale Pneumapress pressure filter and equipment that simulates the Pneumapress pressure filter. NREL plans to evaluate a pilot scale Pneumapress for this process application as well as for other liquid/solid separation process applications including P300, P100 ambient temperature, and post-distillate. If evaluations are successful and funding can obtained, a pilot scale Pneumapress will be considered for purchase so that further liquid/solid separation testing can be done.