Appl Biochem Biotechnol (2011) 163:90–101
DOI 10.1007/s12010-010-9019-8

Evaluation of Target Efficiencies for Solid–Liquid Separation Steps in Biofuels Production

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Received: 8 May 2009 / Accepted: 22 June 2010 /
Published online: 4 July 2010

Abstract Development of liquid biofuels has entered a new phase of large scale pilot demonstration. A number of plants that are in operation or under construction face the task of addressing the engineering challenges of creating a viable plant design, scaling up and optimizing various unit operations. It is well-known that separation technologies account for 50–70% of both capital and operating cost. Additionally, reduction of environmental impact creates technological challenges that increase project cost without adding to the bottom line. Different technologies vary in terms of selection of unit operations; however, solid–liquid separations are likely to be a major contributor to the overall project cost. Despite the differences in pretreatment approaches, similar challenges arise for solid–liquid separation unit operations. A typical process for ethanol production from biomass includes several solid–liquid separation steps, depending on which particular stream is targeted for downstream processing. The nature of biomass-derived materials makes it either difficult or uneconomical to accomplish complete separation in a single step. Therefore, setting realistic efficiency targets for solid–liquid separations is an important task that influences overall process recovery and economics. Experimental data will be presented showing typical characteristics for pretreated cane bagasse at various stages of processing into cellulosic ethanol. Results of generic material balance calculations will be presented to illustrate the influence of separation target efficiencies on overall process recoveries and characteristics of waste streams.

Keywords Solid–liquid separations · Ethanol production · Lignocellulosic biomass

Introduction

The development of biomass conversion technologies to fuels and chemicals has entered the phase of pilot and commercial demonstrations. Efforts have focused on depolymerization of biomass matrices and hydrolysis of available sugar polymers into fermentable

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sugars. Pretreatments have been developed that deconvolute lignocellulose. Steam explosion [1–3], ammonia fiber explosion [4–6], lime [7], dilute acid [8, 9], or concentrated acid [10] treatments are among the options that have been tested on a pilot scale on cellulosic feedstocks. In most cases, pretreatment is focused on a specific component of the biomass—hemicellulose, cellulose, or lignin. Dilute acid pretreatment methods typically lead to partial hydrolysis of hemicellulose, while alkaline-based methods target cellulose. Concentrated acid pretreatment hydrolyzes most of the biomass components. In all cases, attempts are made to minimize production of inhibitors of fermentation.

The advent of the demonstration phase in lignocellulosic utilization for biofuels brings engineering challenges in applying process design, scale-up, and selection of equipment to new process technologies. Few systematic studies have been presented with respect to solid–liquid separations in cellulosic ethanol production [11, 12]. Several features of agricultural biomass-derived feedstocks complicate the development of reliable and consistent operation of solid–liquid separations. For example, variability in biomass properties throughout the processing season, due to differences in growing areas and storage conditions, may drastically change filterability of pretreated biomass. Particle size reduction and increasingly available surface area, which facilitates chemical reactions and mass transfer, complicate the task of separation or fractionation of biomass components. Since complete separation of target components from hydrolyzed mixtures is virtually impossible, realistic separation targets have to be established for each process step. The separation targets must be determined experimentally, based on available technologies and scale-up considerations.

In the present paper, the influence of target separation efficiencies on ethanol yields and other process parameters is demonstrated for aqueous ammonia biomass pretreatment [13]. Because of the similarities in processing technologies for various agricultural feedstocks, the methodology used in this study may be helpful in analyzing other solid–liquid separations used in biomass conversions.

Process Design and Target Efficiencies

A simplified flow diagram illustrating the main steps of the Audubon Sugar Institute aqueous ammonia pretreatment process is presented in Fig. 1. The process was developed to convert grassy feedstocks, such as cane bagasse, to ethanol. After pretreatment in a tumbling reactor, solid biomass is separated from the liquid in a screening device (Separation 1). A portion of fine solid particles is lost with the liquid stream. After Separation 1, biomass is washed in the same screening device to remove fermentation inhibitors remaining in the solid phase (Separation 2). To decrease solids moisture content for following saccharification and fermentation (SSF) step, washed solids are milled in a three-roller sample mill (Separation 3). After SSF is complete, the residual solids, mainly xylans, are separated from the reaction mixture (Separation 4). Ethanol is then distilled from the liquid phase, and the solid phase is used for further processing.

Experimental procedures must be established to characterize the process streams, select appropriate separation techniques, and provide data for material and energy balances. Depending on the process configuration, separations may target either the solid or liquid phase for downstream processing. A simple diagram in Fig. 2 illustrates that solid–liquid separations always produce two streams, where each stream contains traces of the other one. Thus, solid phase contains liquid, and some solid particles are present in the liquid phase.

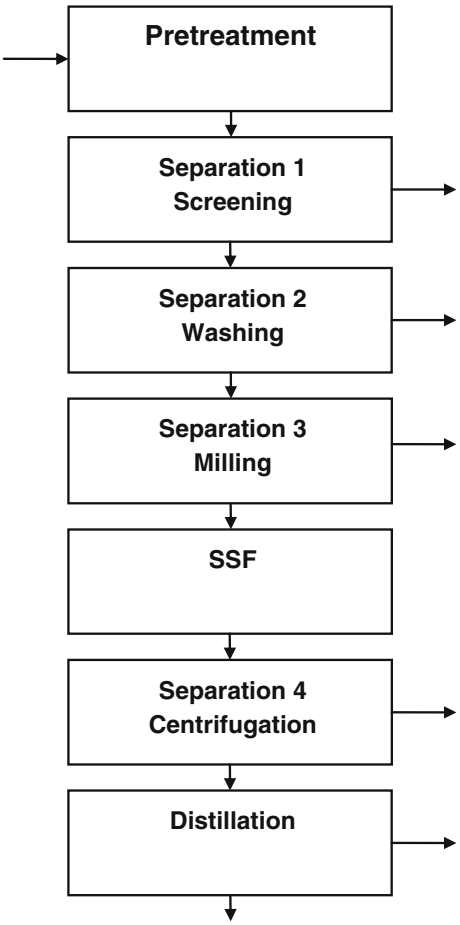

Fig. 1 Simplified flow diagram of Audubon Sugar Institute (ASI) bagasse-to-ethanol process

Efficiency targets must be defined for each solid–liquid separation step. For example, the goal of pretreatment in the ASI process is to partially solubilize lignin and facilitate enzymatic hydrolysis and saccharification at high solids loading. Therefore, removal of inhibitors and subsequent cake moisture reduction must be achieved through solid–liquid

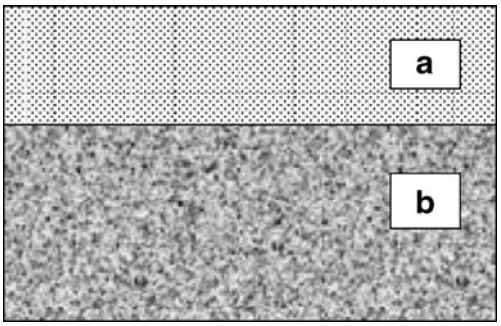

Fig. 2 Solid–liquid separations. **a** Liquid phase with entrained solids. **b** Solid phase with entrained liquid)

separations. It is the objective of the current study to evaluate the sensitivity of the process parameters to separation efficiencies for each step.

Material Balance Constraints

Selection of input parameters is an important task for establishing a reliable mass balance. The methodology applied in the present work requires input parameters to be either measured directly or calculated from the results of pilot experiments. Table 1 contains input data that were used for calculating solid and liquid balances in each step of the ASI process. The developed mass balance accounts for the presence of suspended solids in liquid streams.

Separation efficiency was defined as the fraction of suspended solids recovered from the liquid phase. Washing efficiency was calculated as the percentage of dissolved solids removed from the solid phase. Results of mass balance calculations were used to model the influence of separation parameters throughout the process on ethanol yield and the volume of effluent streams.

Experimental Procedures

Samples were taken from various stages of a pilot plant that processed sugarcane bagasse into ethanol using the ASI aqueous ammonia procedure [13]. Settling and decanter centrifugation unit operations were selected to illustrate the impact of solid–liquid separations on overall process parameters. Based on results of particle size analysis, it

Table 1 Set of input parameters for mass balance.

Unit Operation	Measured Parameters			
	Dissolved Solids (%)	Moisture Content (%)	Suspended Solids (%)	Separation Efficiency (%)
Pretreatment				
Solid Biomass		x		
Pretreated Slurry	x		x	
Separation 1—Screening				X
Liquid Phase	x		x	
Solid Phase		x		
Separation 2—Washing				
Liquid Phase	x			
Solid Phase		x		
Separation 3—Milling				
Liquid Phase	x			
Solid Phase		x		
Enzymatic Hydrolysis and Fermentation				
Fermentation Slurry	x		x	
Separation 4—Centrifugation				X
Liquid Phase	x		x	
Solid Phase		x		

was expected that the two unit operations would demonstrate significant differences in separation performance.

Settling

Settling tests were performed in a 100-ml clear graduated cylinder. Samples were heated to a temperature of 95 °C, stirred well, poured into the cylinder, and allowed to settle for 10 min. Settling time was selected based on empirical scale-up factors for commercial clarifiers used in the sugar industry. Formation of a solid–liquid interface was observed, and the interface level was recorded every 30 s. The process temperature was monitored. After 10 min of settling, the supernatant was carefully decanted and analyzed for suspended and dissolved solids. Solids concentration and moisture content of the settled mud (underflow) were also measured.

Centrifugation

A Hermle Z320 centrifuge fitted with a swinging bucket rotor was used for centrifugation experiments. Samples were preheated to 95 °C, placed into 50-ml Corning centrifuge tubes, and spun at 3,200 rpm for 4 min. The overflow was then carefully decanted. Solid phase moisture content was measured. Concentrations of dissolved and suspended solids in the liquid phase were analyzed. The centrifugal force was selected at about 1,000×g which is typical of commercially available decanter centrifuges. It is understood that the laboratory results would not match the separation efficiencies of commercial centrifuges, mainly due to the difference in residence time. Scale-up of centrifugation was not possible with the available quantities of feed material.

Analytical Procedures

Concentration of dissolved solids was measured using a Reichert AR200 digital refractometer for streams with relatively low concentrations of suspended solids. The refractometer wave length is 589 nm and it was calibrated for sugar solutions. The instrument reading may not be exact due to the presence of soluble lignins that have refractive indices different than that of solubilized sugars. The effect of lignin measurement error on overall concentration was estimated by adding lignin solution to the prepared sugar solution. Because of poor lignin solubility, at 3–4% DS the effect of lignin was negligible within 5%. Solubility of 20,000 MW lignin at room temperature was measured at 1.4 g/l. When suspended solids concentrations exceed 2–5% (depending on the type of suspensions), the accuracy of dissolved solids measurements tends to decrease. For thicker suspensions, the samples should be filtered. Corrections should be made for the weight of removed suspended solids. If this is neglected, analytical errors can lead to significant discrepancies in material balance calculations [14].

Moisture content was determined using a Sartorius Mark 3 moisture analyzer. Samples weighing about 2 g were homogenized and placed on the moisture analyzer's balance and dried at 100 °C until a constant weight was obtained. Sartorius Mark 3 Moisture Analyzer was also used to determine suspended solids concentrations in the slurries using the following procedure.

A Whatman No. 4 filter paper (with a nominal particle retention size of 20–25 μm) was dried and weighed on the analyzer's scale. Samples of slurries (5 ml) were filtered, and the filters were placed in the moisture analyzer for drying at 110 °C until constant weight was

reached. The total weight of the filter, suspended solids, and water are thus calculated. The moisture analyzer is programmed to report the concentration of the suspended solids in milligrams per liter. It is understood that a certain amount of fine particles may be carried through the filter paper during the initial phase of filtration. The volume of "breakthrough" particles is negligible compared to the total volume retained on the filter paper. Using paper with smaller pore size is possible but the total volume of filtered sample is reduced due to fouling. This in turn reduces the accuracy of measurement.

Particle size distribution of the streams was characterized using CILAS 1180L laser diffraction analyzer. The samples were initially prescreened through an 18-mesh sieve to avoid plugging of the analyzer cell. Compositional analysis of samples was performed using standard procedures recommended by NREL [15].

Results and Discussion

Separation 1—Screening

Pretreated biomass from the tumbling reactor was discharged into a stainless steel cylindrical screen with opening size about 0.5 mm. The screen surface area was oversized to avoid pressure buildup during reactor discharge. The 80 psi discharge pressure was released instantaneously. The screen held the pretreated bagasse while the liquid fraction drained by gravity for 10 min. This allowed for reduction of moisture content to approximately 80%. A portion of fine suspended solids was lost in the liquid phase flowing through the screen during pressure release. Measurements of volume and suspended solids concentration of collected liquid fraction showed that the loss of biomass at this step may reach as high as 3–4% of the total feed load. The screening process step described above is designated as Separation 1 in Fig. 1.

A variety of separation methods may be considered for suspended solids recovery from the liquid fraction from Separation 1 and reduction of BOD and COD of the waste stream. Results of settling and centrifugal separation tests are discussed below.

Based on particle size analysis data, settling without the addition of flocculants was not expected to perform at high efficiency due to the presence of very fine particles (about 35% of particles were smaller than 100 μm). Settling tests were carried out according to the description in the "Experimental Procedures" section above. Because of the high turbidity of the solution, no noticeable interface was detected for the first few minutes of settling. Temperature drop was observed during the settling period, which would not be typical for operation of commercial clarifiers. Maintaining constant temperature is expected to improve settling characteristics. The average separation efficiency of the settling process was calculated at 43%. The overflow was not clear and contained a significant amount of suspended solids (concentration of 22,600 mg/l). Despite the low separation efficiency, a settling process may still be applied in combination with another more efficient method to recover suspended solids lost during Separation 1.

The experimental results of settling and centrifugation are presented in Table 2. Values of ethanol yield were calculated using the developed material balance program. Maximum theoretical yield was calculated based on the measured value of 37% glucans in raw bagasse prior to pretreatment. These data were corroborated by various researchers [16, 17].

Under the assumptions that 5% solids are carried with the liquid phase after Separation 1 and solids in the liquid phase are of similar composition to pretreated biomass, recovery at 43.4% efficiency could increase the ethanol recovery from 94.6% to 96.7% of theoretical

Table 2 Effect of separation efficiencies on ethanol yield loss.

	Feed Suspended Solids (mg/l)	Overflow Suspended Solids (mg/l)	Underflow Suspended Solids (mg/l)	Separation Efficiency (%)	Increase in Ethanol Yield (points)
Settling	35,826	22,643	57,081	43.4%	2.1
Centrifugation	25,397	8,105	97,075	72.6%	3.6

yield. At 72.6% recovery measured in centrifugation tests, the yield increases from 94.6% to 98.2%.

Apparently, recovery of suspended solids from the liquid fraction of Separation 1, even at relatively low efficiencies, results in a reduction of overall ethanol loss.

Separation 2—Washing and Inhibitor Removal

Selection of proper equipment and techniques for the separation steps outlined in Fig. 1 is critical for optimization of final product yield. To simplify analysis in the model, it was assumed that 100% of glucans were converted to glucose, and all available glucose was then converted to ethanol at a ratio of 0.51 g ethanol/g glucose in the absence of inhibitors. Our pilot experiments showed that fermentation of pretreated bagasse in the presence of inhibiting components, such as lignin, furfurals, and organic acids, without washing, can reduce the ethanol yield to 0.36 g ethanol/g glucose [18]. As wash water usage increased, ethanol yield improved in a linear fashion. Calculated ethanol yield as a function of inhibitor removal is presented in Fig. 3. The theoretical yields were calculated assuming 5.0%, 7.5%, and 10.0% solids loss to the liquid streams (separation efficiencies of 90%, 92.5%, and 95%, respectively).

Significant ethanol losses can be incurred if washing stage is not efficient. Decrease of both cake moisture content and dissolved solids content should lead to improved yields. Consequently, a combination of washing and milling may be required to achieve removal of inhibitors as well as reduce moisture contents of cakes to acceptable levels for an SSF step. The volume of wash water is an important parameter that affects process economics. High wash water use can improve process yield by reducing concentration of inhibitors, but this will increase the volume of effluent with its concomitant cost of disposal.

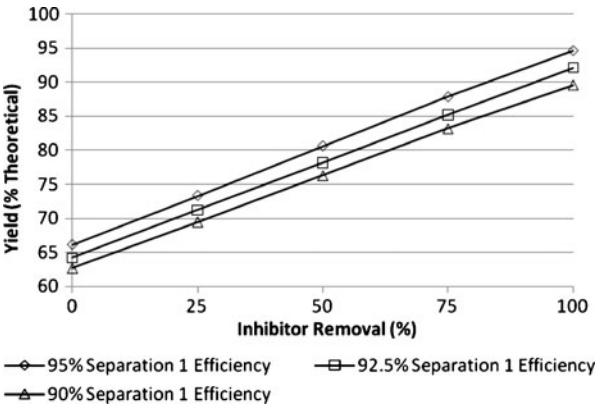

Fig. 3 Effect of inhibitors on ethanol yield

Single stage washing experiments were performed to obtain additional information on dissolved solids concentration of wash effluent and determine the relationship between wash water use and inhibitor removal. A 200 g sample of ammonia pretreated bagasse was collected and filtered under vacuum using a commercial screen with 0.44×5 mm slots. Cake moisture content and the concentration of dissolved solids in the filtrate were analyzed before washing. Wash water was then added within the range of 0.5–6.0 g water per gram of pretreated bagasse in 0.5 g increments. Effluent from each incremental washing step was analyzed for dissolved solids. Composite effluent samples were also collected and analyzed. Washing efficiencies were calculated for each washing ratio, and results from these experiments are presented in Fig. 4.

Results illustrate that complete washing in a single stage required large volume of water (6.2:1 ratio). A multistage washing procedure will be required in a commercial process to minimize effluent volume and cost of waste handling.

Cake Moisture Content

The ratio of wash water required to remove inhibitors is a function of both moisture content of solids from Separation 1 and washing efficiency. Cake properties also affect the washing efficiency. For illustration purposes, cake characteristics were considered to be constant. Data obtained in single stage washing experiments were used to calculate water requirements as a function of cake moisture content. Results plotted in Fig. 5 are calculated values of wash water requirements at various degrees of inhibitor removal (washing efficiencies).

As a general trend, wash water is reduced at lower moisture content of solid phase. For example, at 100% washing efficiency, reduction of cake moisture content from 80% to 50% results in about 65% water savings. This is especially important considering that the wash water stream must be treated as an effluent. The cost of an additional milling stage to reduce the solid phase moisture content before washing should be considered.

Separation 3—Milling

A milling stage (Separation 3) was introduced to increase solids loading for saccharification and fermentation experiments. A three-roll sugar mill was used to reduce moisture content

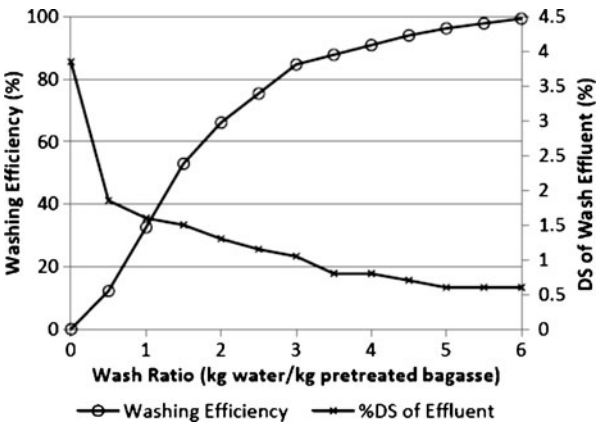

Fig. 4 Evaluation of efficiency of a single stage washing step

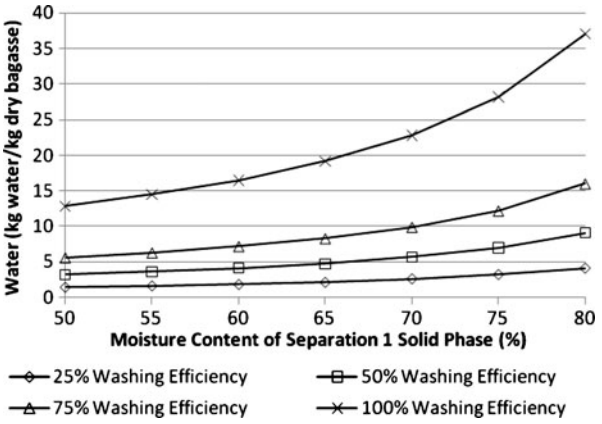

Fig. 5 Effect of moisture content of Separation 1 solid phase on wash water requirements

of pretreated bagasse. The major goal of milling equipment, e.g., in sugar production, is to remove liquid from the solid phase in a multistage process. It is usually accompanied by washing. Milling results in certain particle size reduction when fibers go through the mill rollers.

Because the mill does not have a means to capture fine particles and prevent them from being lost with the liquid stream, a portion of solids is always entrained in the liquid phase. Our earlier studies indicate that this loss can be as high as 3–4% of feed solids, which results in a corresponding reduction in ethanol yield. Results from the model material balance are presented in Fig. 6 showing the effect of moisture content of milled cake on calculated yield. The washing efficiency at the preceding stage (Separation 2) varied in the calculations between 50% and 100%.

Results demonstrate that reduction of cake moisture is beneficial for waste reduction and improved solids content for the saccharification step. Monavari et al. came to similar conclusions while investigating the effects of washing, pressing, and filtering on the inhibitor content of pretreated biomass. They found that unwashed biomass produced higher sugar yields if pressing was incorporated before enzymatic hydrolysis rather than filtration. This is due to the fact that pressing resulted in lower solid phase moisture content.

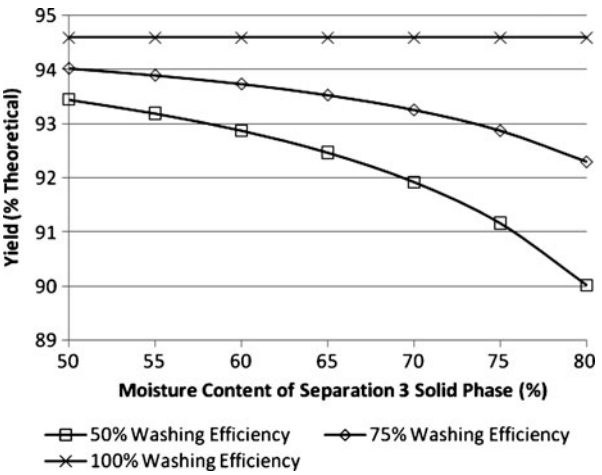

Fig. 6 Effect of moisture content of milled solids on ethanol yield at various washing efficiencies at the preceding stage

Thus, the concentration of inhibiting components in the filtered solids was found to be higher than in pressed biomass [12].

Separation 4—Centrifugation

Fermentation broth was found to contain suspended solids at a level making it unacceptable to be fed directly to a distillation column. Additionally, solid phase containing xylans can be recovered for further processing. A sample of slurry after fermentation was subjected to settling and centrifugation using methods described in the "Experimental Procedures" section.

Virtually no separation of suspended solids was observed during settling of the fermented slurry. An interface was formed within 2 min, but the overflow level was measured at only 9% of the total cylinder height. The suspended solids concentration of the underflow was 35,200 mg/l, which was only slightly higher than initial concentration of 32,854 mg/l. Centrifugation tests demonstrated relatively high separation efficiency (95.6%). Results representing mean values of four parallel tests are shown in Table 3. The underflow was well packed and represented 27.5% of the total original volume; moisture content of the underflow was 80%.

The final concentration of ethanol in the solution and the moisture content of the cake define ethanol loss with the solids fraction. Yield was calculated assuming 2.75% ethanol concentration in the liquid phase. The graph in Fig. 7 illustrates that ethanol yield is more sensitive to changes in cake moisture than to separation efficiency (within the separation range expected of centrifugal operation).

The separation targets for step 4 will depend on the limitations of suspended solids content in the feed to distillation columns. An additional washing step may be considered to recover ethanol from solid precipitate.

To illustrate the sensitivity of overall process efficiencies on the parameters of solid–liquid separations, two processing scenarios were compared (Table 4). Process parameters used in Scenario 2 are more typical for larger scale biomass separations [19].

A 9.7% increase in ethanol yield can be achieved by utilizing more efficient separation techniques leading to lower moisture reduction of solid cake. An additional 40% reduction in wash water requirements is expected.

Conclusions

Biomass derived ethanol production requires several solid–liquid separation steps. The separation efficiencies influence ethanol yield and the volume and the compositions of waste streams.

Table 3 Separation of suspended solids from fermentation slurry.

	Feed	Overflow	Underflow	Separation 4—Efficiency (%)
Volume (ml)	40	29	11	
SS Concentration (mg/l)	32,854	1,995	139,146	
Mass of Solids (mg)	1,314	58	1,256	95.6

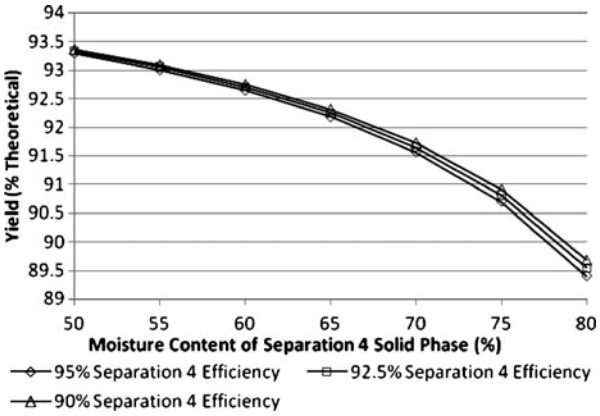

Fig. 7 Effect of moisture content of Separation 4 solid phase on yield

Particle size distribution of pretreated biomass determines what portion of the feedstock is lost when a liquid phase is separated. The losses can reach up to 10% of the total solid biomass, with equivalent yield reduction. Additional separation steps may be required to recover these lost solids.

A mass balance for an aqueous ammonia pretreatment process for bagasse was established. Input parameters were measured at both bench and pilot scale. The sensitivity of process parameters to reduction of moisture content of solid phase was determined. Separation targets were established to maximize overall process efficiency.

Reduction of both water and dissolved solids content in the solid phase affects the level of inhibitor removal and liquid effluent volume. Up to 30% yield reduction may be expected if inhibitors are not removed completely. Capital and operating cost of separation equipment will be evaluated in future studies.

Wash water requirements based on a single stage washing are quite high. More efficient countercurrent washing procedures must be considered for process scale-up.

Table 4 Comparison of two separation scenarios.

	Scenario 1	Scenario 2
Separation 1		
Separation Efficiency (%)	90	95
Moisture Content (%)	80	65
Separation 2 (Washing)		
Washing Efficiency (%)	75	75
Separation 3 (Milling)		
Moisture Content (%)	70	50
Separation 4 (Centrifugation)		
Separation Efficiency (%)	90	95
Moisture Content (%)	80	65
Calculated Values		
Yield (% Theoretical)	83.7	92.7
Wash Water Use (kg water/kg dry bagasse)	13.15	7.89

Acknowledgments This research was supported by DOE award (DE-FG36-08GO88151). This support does not constitute an endorsement by DOE of the views expressed in this article.

We would like to thank our colleagues from Audubon Sugar Institute, Dr. Don Day and Dr. Giovanna DeQueiroz, for their useful technical discussions and Iryna Tishechkina for helping in the experimental work.

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