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BIORESOURCE TECHNOLOGY, OXFORD, v. 111, pp. 447-452, MAY, 2012
http://www.producao.usp.br/handle/BDPI/42687

Bioresource Technology 111 (2012) 447–452

Contents lists available at SciVerse ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Mass balance of pilot-scale pretreatment of sugarcane bagasse by steam explosion followed by alkaline delignification

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ARTICLE INFO

Article history:
Received 19 September 2011
Received in revised form 31 January 2012
Accepted 1 February 2012
Available online 16 February 2012

Keywords:
Sugarcane bagasse
Steam explosion
Delignification
Pilot scale

ABSTRACT

Five pilot-scale steam explosion pretreatments of sugarcane bagasse followed by alkaline delignification were explored. The solubilised lignin was precipitated with 98% sulphuric acid. Most of the pentosan (82.6%), and the acetyl group fractions were solubilised during pretreatment, while 90.2% of cellulose and 87.0% lignin were recovered in the solid fraction. Approximately 91% of the lignin and 72.5% of the pentosans contained in the steam-exploded solids were solubilised by delignification, resulting in a pulp with almost 90% of cellulose. The acidification of the black liquors allowed recovery of 48.3% of the lignin contained in the raw material. Around 14% of lignin, 22% of cellulose and 26% of pentosans were lost during the process. In order to increase material recovery, major changes, such as introduction of efficient condensers and the reduction in the number of washing steps, should be done in the process setup.

1. Introduction

In order to satisfy the growing demand for renewable energy without affecting the food sector, raw materials, such as lignocellulosic bioresources, should be utilised (Hahn-Hägerdal et al., 2006; Wyman, 2008). The conversion of lignocellulose into different kinds of chemicals and materials according to a biorefinery concept is also a relevant issue for the sustainable development of the chemical industry in a future *post-petroleum* scenario (Zhang, 2008).

In Brazil, the generation of sugarcane bagasse has increased during the last years as a result of the expansion of ethanol production. Per ton of harvested cane, 270–280 kg of bagasse (50% moisture) is produced. The annual production of sugarcane bagasse in Brazil is estimated at 186 million tons (Soccol et al., 2010). Currently, most of the bagasse is used as solid fuel in sugar mills and ethanol distilleries; however, it has been demonstrated that modernisation of the boilers and the rationalisation optimisation of steam usage could satisfy the energy requirements of the plants with around 50% of the produced bagasse. The excess bagasse could be used in different applications, including ethanol production (Cardona et al., 2010; Soccol et al., 2010).

A key step in the production of ethanol from lignocellulosic materials is the hydrolysis of cellulose, which can be catalysed either with acids or enzymes. Because of its higher potential sugar yield and its lower environmental impact, enzymatic hydrolysis is more attractive than acid hydrolysis (Taherzadeh and Karimi, 2007); however, enzymatic hydrolysis is restricted by several factors, mainly by the crystalline structure of native cellulose fibres, and by the presence of hemicelluloses and lignin on the cellulose surface, which impedes access of cellulases to the substrate (Himmel et al., 2007; Laureano-Pérez et al., 2005). Therefore, a pretreatment must be implemented prior to enzymatic hydrolysis (Hendriks and Zeeman, 2009). This pretreatment step strongly influences downstream costs by determining fermentation toxicity, enzymatic hydrolysis rates, enzyme loadings, mixing power, waste treatment demands, and other process variables (Wyman et al., 2005).

Among several pretreatment methods investigated for lignocellulosic bioresources (Hendriks and Zeeman, 2009; Mosier et al., 2005), steam explosion has been proven to be effective for different materials (Saddler et al., 1993), including sugarcane bagasse (Martín et al., 2002, 2008; Rocha et al., 2011a,b), and its effectiveness can further be increased if lignin is removed from the pretreated material (Palonen, 2004).

In an effective pretreatment, all the biomass components should be recovered in a way that they could be upgrade to valuable products, but important material losses often occur which affect the economy of the process. Since many pretreatment studies report only the compositional analysis of the streams, it is often difficult to verify the integrity of the biomass components. Applying mass

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doi:10.1016/j.biortech.2012.02.005

balance calculations allows assessing the recovery of each component and the material losses occurring in each operation (Hatzis et al., 1996). In a recent report, Garlock et al. (2011) used material balances for comparing different pretreatment technologies for switchgrass, but steam explosion was not included.

Therefore, this work was aimed to gather mass balance data on pilot-scale steam explosion pretreatment and alkaline delignification processes in order to estimate the cost of ethanol production from sugarcane bagasse cellulose.

2. Methods

2.1. Raw material

Sugarcane bagasse batches, each weighing approximately 200 kg and having 50% moisture content, were obtained from several sugarcane mills and ethanol distilleries (Vale do Rosário, Santa Elisa, São Martinho, Nova América, Usina Açucareira Ester S.A-Cosmópolis) in São Paulo state, Brazil. The material was dried at ambient temperature until the dry matter content was above 90%, and stored at 4 °C. A portion of each type of bagasse was milled in a Willey type mill to a particle size of 16/60 mesh and used for raw material analysis.

2.2. Pretreatment

Pretreatment and delignification processes were carried out according to the scheme shown in Fig. 1. In each pretreatment experiment, 10 kg of dry bagasse was loaded into a 200-L stainless steel reactor (Confab Industrial S/A, São Caetano do Sul, S.P., Brazil). The reactor was hermetically closed, and steam was injected until a pressure of approximately 1.3 MPa (equivalent to 190 °C) was achieved. After 15 min of pressurisation, the reactor was suddenly depressurised by an operator standing at a safe distance, and the steam-exploded slurry was discharged into a 500-L cyclone, and collected in a cylindrical container. By centrifugation of the slurry at 542.1*g* for 10 min in a 100-L semi-industrial centrifuge (GRISANTI Máquinas industriais Ltda, Ribeirão Pires, S.P., Brazil), the solid fraction, hereafter referred to as cellulignin, was separated from the liquid fraction, hereafter referred to as hemicellulosic hydrolysate. The cellulignin was thoroughly washed by resuspending it in a previously determined volume of water and centrifugation at 542.1*g* for 10 min until the yellow colour of the effluent was totally removed. A total of five washing cycles was performed. In each cycle, the exact amount of used water and of the removed effluent was measured.

2.3. Posthydrolysis

In order to achieve complete hydrolysis of the hemicelluloses-derived oligomers contained in the liquid streams, the hemicellulosic hydrolysates and the effluents from the washing steps were posthydrolysed by adjusting the pH to 0.9–1.0 with 98% H_2SO_4 and incubation at 121 °C for 30 min in autoclave (Phoenix AV plus 100 L, Araraquara, S.P., Brazil). The composition of the effluents obtained in each washing cycle was analysed by HPLC. The recovery of polysaccharides was calculated from the amount of detected monosaccharides.

2.4. Alkaline delignification

The cellulignin obtained in each of the five pretreatment experiments was submitted to alkaline delignification in a 350-L stainless steel-coated cast iron reactor, equipped with a stirrer, and heated through a jacket with thermal oil. The reactor was loaded with water and heated to 95 °C. After that, 6.6 kg of dry cellulignin and NaOH solution were added. The final concentration of NaOH was 1% (w/v), and the liquid-to-solid ratio was 20:1. The reaction mixture was heated to 100 ± 2 °C and held under stirring (80 rpm) for 1 h.

When the reaction time had elapsed, the reactor was discharged, and the raw cellulose (hereafter referred to as cellulosic pulp) was separated from the lignin-rich soluble fraction (hereafter referred to as black liquor) by centrifugation at 542.1*g* for 10 min. The cellulosic pulp was thoroughly washed (seven washing cycles) until lignin was completely removed and no more yellow colour was observed. Then, it was submitted to chemical characterisation and stored at 4 °C for further enzymatic hydrolysis studies. The black liquor was collected and stored for further lignin recovery.

2.5. Lignin precipitation

Lignin was isolated from the black liquors by precipitation after acidification. The liquors were transferred to a 500-L tank, and approximately 1.5 L of 98% sulphuric acid was added until a pH of approximately 2.0 was reached. The precipitated lignin was separated by centrifugation (542.1*g*, 10 min), and washed six times with water (100 L each time) until a final pH of approximately 6.0 was achieved and negative test for sulphates using the barium chloride method (Welcher and Hahn, 1964) was obtained. The washed lignin was dried at 70 °C and weighed. The concentration of soluble lignin in the supernatant was determined for each of the six washings.

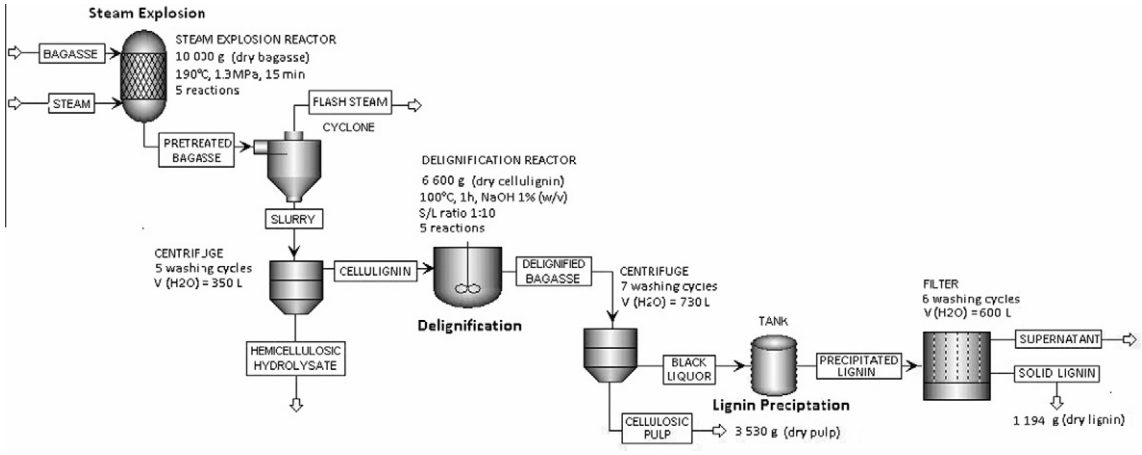

Fig. 1. Flow chart of the separation processes for the main components of sugarcane bagasse.

2.6. Analysis of the chemical composition of the solid materials

The chemical composition of the solid materials (raw bagasse, cellulignin and cellulosic pulp) was analysed by analytical acid hydrolysis followed by chromatographic analysis of the hydrolysate, and by gravimetric determination of acid-insoluble lignin following a methodology adapted by Rocha et al. (1997). Raw bagasse was extracted with a 2:1 (v/v) ethanol/cyclohexane mixture for 8 h before hydrolysis in order to remove organic extractives. The extractive-free bagasse was washed with water at 70 °C, dried, and submitted to hydrolysis. In the analysis of cellulignin and cellulosic pulp, no prior extraction was performed. All the analyses were performed in triplicates.

Aliquots (2 g) of the material were treated with 10 mL of 72% H_2SO_4 in a 100-mL beaker maintained in a thermostated bath at 45 °C for 7 min with vigorous shaking. The reaction was stopped by addition of 50 mL of distiled water, and the resulting mixtures were quantitatively transferred to 500-mL Erlenmeyer flasks, where the acid was diluted with water to a final volume of 275 mL. For completing the hydrolysis of the unhydrolysed oligosaccharides, the flasks were covered with aluminium foil and autoclaved for 30 min at 1.05 atm. After depressurisation, the flasks were cooled to room temperature, and the hydrolysis mixtures were filtered using previously dried and weighed analytical filter papers. Each hydrolysate was collected in a 500-mL volumetric flask, and the filtration residue was washed with 50-mL portions of distiled water. The water washes were added to the flask with the hydrolysate until completing the flask volume, and the mixture was immediately submitted to HPLC analysis.

The acid-insoluble lignin retained on the filter was thoroughly washed until the sulphate anions were removed (approximately 1500 mL), and dried at 105 °C until constant weight. For determination of the ash content in acid-insoluble lignin, the dry residue was quantitatively transferred to a weighed crucible, incinerated at 300 °C for 40 min and at 800 °C for 2 h. The mass of the ashes was determined in an analytical balance after cooling the crucible with the incinerated sample in a desiccator (ASTM, 1966).

2.7. Analysis of the chemical composition of the liquid fractions

Sugars, acetic acid and furan aldehydes in the liquid streams generated in the process (hemicellulosic hydrolysates, black liquors and washing waters), as well as in the analytical acid hydrolysates were analysed by HPLC. Triplicate samples were filtered through a Sep Pak C18 filter, and the filtrate was injected into the HPLC system (Shimadzu C-R7A, Kyoto, Japan). Cellobiose, glucose, xylose, arabinose and acetic acid were separated with an Aminex HPX 87H (300 × 7.8 mm, BIO-RAD, Hercules, CA) at 45 °C using 5 mM H_2SO_4 as mobile phase at a flow rate of 0.6 mL min^{-1}, and detected with an RI detector (Shimadzu RID-6A). Furfural and hydroxymethilfurfural (HMF) were separated on an RP-18 (C-18) 125 × 4 mm column (Hawlett-Packard), and detected with a UV–visible detector (Shimadzu SPD-10A) at 25 °C using a mobile phase composed of 1% acetic acid-containing 1:8 acetonitrile–water solution pumped at 0.8 mL min^{-1}. The concentrations of glucose, cellobiose and HMF were used for calculating the cellulose content, whereas the content of hemicelluloses was calculated based on the concentrations of xylose, arabinose, acetic acid and furfural. Their masses were divided by the dry weight of the initial material and multiplied by the hydrolysis factors, which were 0.9, 0.95 and 1.29, respectively, for glucose, cellobiose and HMF, and 0.88 for both xylose and arabinose, 0.72 for acetic acid and 1.37 for furfural.

For the quantification of the lignin contained in all the liquid streams, 5 mL aliquots were diluted with distiled water and 2 mL of 6.5 M NaOH giving a final volume of 100 mL and a pH of approximately 12.0. The absorbance of the resulting mixture was read at 280 nm in a UV-spectrophotometer (Shimadzu UV-150-02). After that, soluble lignin was calculated according to the following expression, using previously determined absorptivity values (Rocha et al., 1993), and considering the dilution ratios:

$$C_{lig} = 4.187 * 10^{-2} \ (A_{lig280} - A_{pd280}) - 3.279 * 10^{-4} \quad (1)$$

where:

C_{lig}: concentration of soluble lignin (g L^{-1});
A_{lig280}: absorbance of the solution at 280 nm;
A_{pd280}: absorbance of furfural and HMF

$$A_{pd280} = C_1 \cdot \varepsilon_1 + C_2 \cdot \varepsilon_2 \quad (2)$$

where:

C_1: furfural concentration (g L^{-1});
C_2: HMF concentration (g L^{-1});
ε_1: furfural UV-absortivity at 280 nm: ε_1 = 146.85 cm^{-1} g^{-1} L (experimental value);
ε_2: HMF UV-absortivity at 280 nm: ε_2 = 114.00 cm^{-1} g^{-1} L (experimental value);

3. Results and discussion

The compositional analysis of the raw materials revealed that, independent of the origin, the chemical composition of the bagasse was very similar, with rather narrow standard deviations between the mean and the values of the individual samples (Table 1). The obtained composition is in agreement with previous published (Sanjuán et al., 2001; Martín et al., 2006; Martín and Thomsen, 2007; Rocha et al., 2010; Rocha et al., 2011a).

3.1. Pretreatment

The solid material recovered after pretreatment was 6625 g, which indicates that around one third of the raw bagasse was solubilised. Most of the pentosan fraction (82.6%) and the whole acetyl group fraction were solubilised, whereas, cellulose (90.2%) and lignin (87.0%) were mainly recovered in the pretreated solids. The mineral components were rather evenly distributed in both solid and liquid fractions. The recoveries of the individual components were close to previously reported yields (Rocha et al., 2010). Canilha et al. (2011) realised a pretreatment of sugarcane bagasse with H_2SO_4 2.5% (w/v) at 150 °C for 30 min and observed a bagasse solubilisation of 41.7% w/w with less solubilisation of cellulose and lignin. According to the chemical composition of the cellulignin (Table 1), the contents of cellulose and lignin in the solid material increased by approximately one third compared to their content in the raw bagasse, whereas the pentosan content decreased by around 74%.

The obtained cellulignin was submitted to five washing steps and the average concentrations of the different components in the liquid fractions are given in Table 2, which also shows the volumes of fresh water used in the washing and obtained effluents, as well as the pH of the effluents. The amounts of extracted components decreased with the number of washes. Although in the fourth wash the concentration of extracted components was very low, another washing step was performed to ensure maximal removal of chromophoric compounds not bound to the biomass. Since the concentration of the compounds of interest in the effluent of the fifth wash was negligible, it was discarded. A total of 225 L of liquid fractions was collected, and the total volume of fresh water used was 350 L. For future works, it would be of interest to investigate how much water would be required if only one washing step would be performed.

The concentrations of pentoses in the liquid fractions were considerably higher than those of glucose (Table 2), which is in accor-

Table 1
Mean chemical composition of the raw bagasse, cellulignin and cellulosic pulp. Standard deviations are shown in parentheses.

Component	Content, % (w/w)			Mass in a 10-kg batch of raw bagasse, g		
	In raw bagasse	In cellulignin	In cellulosic pulp	In raw bagasse	In cellulignin	In cellulosic pulp
Cellulose	42.3 (0.5)	57.5 (0.3)	89.6 (0.9)	4225.0 (50.0)	3809.0 (28.5)	3162.0 (31.8)
Pentosans	25.1 (0.3)	6.6 (0.0)	3.4 (0.1)	2506.0 (30.0)	437.0 (1.4)	120.0 (1.8)
Acetyl groups	3.7 (0.0)	0.0 (0.0)	0.0 (0.0)	365.0 (2.0)	0.0 (0.0)	0.0 (0.0)
Total lignin	24.7 (0.1)	32.5 (0.2)	5.4 (0.0)	2474.0 (12.0)	2153.0 (14.6)	190.6 (0.7)
Ash	3.5 (0.0)	2.8 (0.0)	1.6 (0.3)	350.0 (3.0)	186.0 (2.0)	56.5 (10.6)
Total	100.0 (1.0)	99.4 (0.7)	100.0 (0.0)	10,000.0 (98.0)	6625.0 (46.5)	3530.0 (117.7)

Table 2
Volumes of fresh water (FW) and effluents, and average pH and composition of the liquid fractions obtained after washing the cellulignin.

	$Volume_{FW}$, L	$Volume_{Filtrate}$, L	pH	Concentration, g/L			
				Glucose	Pentoses	Acetyl groups	Lignin[a]
Hemicellulosic hydrolysates	–	8.9 (0.9)	3.5 (0.1)	4.9 (0.10)	55.6 (2.2)	11.2 (1.2)	6.7 (0.5)
First wash	100.0	68.5 (3.5)	3.75 (0.1)	1.2 (0.3)	13.4 (0.9)	2.6 (0.7)	1.7 (1.3)
Second wash	50.0	32.5 (2.5)	4.1 (0.2)	0.7 (0.1)	6.5 (0.5)	1.4 (0.1)	1.1 (0.1)
Third wash	100.0	86.0 (1.5)	5.7 (0.2)	0.1 (0.0)	1.1 (0.1)	0.2 (0.1)	0.3 (0.0)
Fourth wash	50.0	30.0 (2.0)	5.9 (0.2)	0.1 (0.0)	0.9 (0.0)	0.0 (0.0)	0.02 (0.0)
Fifth wash	50.0	D	ND	ND	ND	ND	ND

D, discarded; ND, not detected.
[a] Measured as aromatics.

dance with the solubilisation patterns for pentosans and cellulose (Table 1). However, the detected sugar concentrations did not exactly match the amount of solubilised polysaccharides. The recovery of polysaccharides, calculated using the sugar concentrations in the liquid fractions, revealed the occurrence of considerable carbohydrate losses due to degradation reactions (Fig. 2). The amount of pentoses detected in the liquors accounted for a recovery of 84.1% of the solubilised pentosans, which more than doubled the recovery achieved for the solubilised cellulose (33.6%). The lower recovery of glucans indicates that degradation reactions during pretreatment occurred to a higher extend for pentoses than for glucose. This result is in accordance with those of other reports, since it is known that the susceptibility of pentoses to degradation under acidic conditions is higher than that of hexoses (Canilha et al., 2011).

The recovery graph (Fig. 2) also reveals some losses of lignin and acetyl groups. The analysis method counts as lignin other aromatic compounds, such as furfural, HMF and some extractives absorbing the UV 280-nm wavelength. Therefore, the losses of that component were probably lower than the measured losses.

Taking into account the volumes of the liquid streams and the concentrations of their main components (Table 2), the masses of the components recovered in the liquid phase were calculated. The sum of those masses accounted for only around 2500 g, while the material solubilised during pretreatment amounted almost 3400 g. In other words, 26.5% of the solubilised material was lost. The losses might have occurred during the sudden opening of the reactor valve. In order to avoid losses during steam explosion, a condensation system for recovering the volatile compounds would be required.

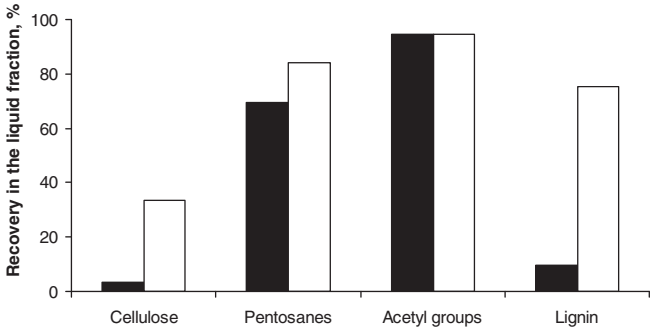

Fig. 2. Recovery of the main components in liquid fractions of the pretreatment based on the content in the raw material (black bars), and on the amount of solubilised components (white bars).

3.2. Delignification

In order to obtain a cellulose-rich pulp, the cellulignin was subjected to alkaline delignification. Significant solubilisation of lignin occurred, but thorough washing was required for recovering most of the solubilised lignin since an important part of it remained in the pulp. The average concentrations and mass of lignin in the liquors, as well as the volumes of fresh water used and the pH of the filtrates are given in Table 3. The amount of extracted lignin decreased from 1342 g in the black liquor to around 7 g in the sixth washing step. A total of 513 L of liquor containing 2766.6 g of lignin was collected in the washing steps. After that, an additional wash with 100 L of water was performed for the complete removal of lignin and total discoloration of the filtrate, which had a final pH of 7.6. Thus, the total volume of fresh water used was 730 L.

The mass of cellulosic pulp obtained after delignification was 3530 g, which is equivalent to 53.3% of the cellulignin entering delignification and to 35% of the raw bagasse (Fig. 3). Since the content of cellulose in the pulp was 89.6% (Table 1), 83% of the cellulose contained in cellulignin was preserved in the pulp, whereas the recovery based on raw bagasse was 74.8%, *i.e.*, a quarter of the initial cellulose was solubilised during the pretreatment and delignification process.

Around 91% of the lignin contained in the cellulignin was solubilised during delignification as deduced from the recovery chart (Fig. 3). Since part of the lignin was solubilised during pretreatment, the solubilisation occurring during delignification corresponds to 79% of the lignin contained in the raw bagasse. In addition to lignin, most of the pentosans (72.5%) and ash (69.6%) remaining in cellulignin were also solubilised during delignification.

Table 3
Volumes of fresh water (FW) and obtained liquors, and average pH and lignin content in the black liquor and the effluents obtained by washing the cellulosic pulp.

	$Volume_{FW}$, L	$Volume_{Liquor}$, L	pH	Lignin	
				Concentration, g/L	Mass, g
Black liquor	30	110	12.2 (0.3)	12.2 (0.3)	1342.0 (13.4)
First wash	100	69	11.9 (0.4)	8.0 (0.6)	552.0 (41.4)
Second wash	100	65	10.5 (0.3)	6.1 (0.5)	393.3 (32.5)
Third wash	100	70	9.8 (0.2)	4.0 (0.2)	280.0 (14.0)
Fourth wash	100	68	8.9 (0.2)	2.1 (0.1)	139.0 (6.8)
Fifth wash	100	65	8.3 (0.2)	0.8 (0.1)	53.3 (5.0)
Sixth wash	100	66	7.6 (0.1)	0.1 (0.0)	7.26 (1.0)

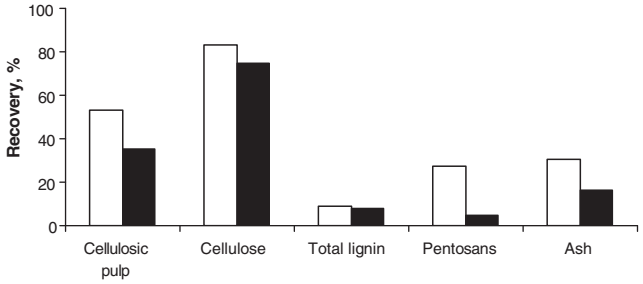

Fig. 3. Recovered material after delignification. White columns, based on the mass of cellulignin; black columns, based on raw bagasse.

Table 4
Volumes of fresh water (FW) and effluents, pH and lignin content in the supernatant of the lignin precipitation step and of the fresh water used in lignin washing.

	$Volume_{FW}$, L	$Volume_{Filtrate}$, L	pH	Lignin content	
				Concentration, g/L	Mass, g
Supernatant	–	480	2.1	0.74 (0.06)	345.6 (30.2)
First wash	100	100	3.9	0.88 (0.05)	88.4 (5.3)
Second wash	100	100	4.5	0.53 (0.04)	52.8 (3.8)
Third wash	100	100	4.9	0.16 (0.01)	15.6 (1.1)
Fourth wash	100	100	5.8	0.04 (0.00)	3.6 (0.3)

3.3. Lignin precipitation

Lignin contained in the black liquors and washes was precipitated, and washed with fresh water. The parameters of the supernatant obtained after lignin precipitation, and of the liquid streams obtained by lignin washing are displayed in Table 4. The water used in the washings (100 L per step) was recovered. This is different to the washing of the cellulignin after pretreatment and of the cellulosic pulp after delignification, where part of the used water was always retained in the washed solids. The amount of the precipitated lignin was 1194 g, which is equivalent to 48.3% of the lignin contained in the raw material (Table 5), and to 55.5% of the lignin contained in cellulignin. A considerable fraction of lignin either remained in the supernatant or was removed with the washes. The sum of the masses of lignin detected in the liquid streams amounted to 506 g, which is equivalent to 20.4% of the lignin contained in the raw bagasse.

Material balances revealed that 13.8% of the lignin contained in the raw bagasse was lost (Table 5). Most of the losses (10.6%) occurred in the delignification and precipitation stages, whereas in the pretreatment only 3.2% of the lignin was lost.

The material balance of cellulose showed that only three fourths of the initial content was recovered in the pulp, while the other fourth was lost with different streams (Table 5). Most of the losses (15.3%) occurred during delignification, and they might be due to peeling reactions, which are often responsible for the degradation of polysaccharides in alkaline pulping (Fengel and Wegener, 1989). Almost 10% of cellulose was hydrolysed during pretreatment, but one third of that amount (3.3% of the initial content) was detected as glucose in the hemicellulosic hydrolysate. An important part of the losses in the pretreatment was due to the formation of furfural (0.75%) and HMF (0.10%). Other degradation products (formic acid and levulinic acid) could be formed, but were volatilised during the blow-up operation of the pretreatment reactor, since they are not detected by HPLC. Physical factors during the filtration and washing operations might also have contributed to the losses both in the pretreatment and in the delignification.

The amount of pentosans identified in the final products amounted to 74.2% of their initial content, including 69.4% recovered as pentoses in the hemicellulosic hydrolysate and effluent of cellulignin washes, and 4.8% contained in the cellulosic pulp (Table 5). The balance revealed that the losses of pentosans, which are attributable to degradation and to physical reasons during filtration and washing, were comparable in pretreatment (13.1%) and delignification (12.6%).

The total mass of the acetyl group fraction recovered as acetic acid in the hemicellulosic hydrolysate was 94.4% of the initial content in raw bagasse (Table 5). The losses, accounting for 5.6%, are also attributable to steam stripping during venting of the reactor and to physical reasons during filtration and washing.

No calculation of the material balance of the ash was made, since some exogenous inorganic material, such as sand and soil, is retained in the filtering device, which makes it difficult to accurately determine the mineral compounds.

Taking into account the components recovered in a valuable form, *i.e.* cellulose in the pulp, precipitated lignin, and hemicelluloses in the hydrolysate, only 64.4% of the material was recovered. Taking into account all the components recovered in the solid and in the liquid streams, 76.4% of the material was recovered. The lack of closure of the mass balance is attributed to the degradation of sugars during pretreatment, the stripping of volatile sugar degra-

Table 5
Mass balance of cellulose, lignin, pentosans and acetyl groups in the global process.

	In the pulp, %	In the hydrolysate, %	Precipitated, %	Remaining in the washes, %	Losses, %	
					In pretreatment	In delignification
Cellulose	74.9 ± 0.8	3.3 ± 0.4	–	–	6.5 ± 0.7	15.3 ± 0,8
Lignin	7.7 ± 0.1	9.8 ± 1.1	48.3 ± 4.7	20.5 ± 1.6	3.2 ± 0.1	10.6 ± 0,4
Pentosans	4.8 ± 0.1	69.4 ± 9.7	–	–	13.1 ± 0.5	12.6 ± 0,7
Acetyl groups	0	94.4 ± 9.3	–	–	5.6 ± 0,1	–

dation products during flashing of the steam explosion reactor and also to physical losses occurring during washing of cellulignin, cellulosic pulp and solid lignin obtained during pretreatment, delignification and lignin precipitation, respectively.

4. Conclusions

Although steam explosion pretreatment combined with alkaline delignification is an effective process for the separation of the main components of sugarcane bagasse, the material balances show that at pilot scale considerable losses occur due to degradation of carbohydrates, stripping of volatile by-products during decompression of the steam explosion reactor, and washout during washing of the solid fractions obtained during different stages of the process. In order to increase material recovery, major changes, such as introduction of efficient condensers and the reduction in the number of washing steps, should be done in the process setup.

Acknowledgements

CM gratefully acknowledges the financial support given by the Alexander von Humboldt Foundation. GJMR gratefully acknowledges the Escola de Engenharia de Lorena/Universidade de São Paulo and Brazilian Bioethanol Science and Technology Laboratory (CTBE). VFNS gratefully acknowledges the financial support given by the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Brazil.

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