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STEAM EXPLOSION OF SUGARCANE BAGASSE AS A PRETREATMENT FOR CONVERSION TO ETHANOL

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Abstract—The State of Hawaii is interested in converting the large volume of agricultural residues, principally sugarcane bagasse, that is generated in the state into transportation fuels. One of the technologies that is currently being evaluated is steam explosion as a pretreatment for conversion of the bagasse into ethanol. In order to identify the optimum conditions of the steam explosion cycle, a range of operating temperatures (188–243°C) and residence times (0.5–44 min) were used to pretreat bagasse in a 101 batch reactor. The analytical results were also used to evaluate the "reaction ordinate" concept. The exploded bagasse samples were examined as to total mass recovery, weight loss by water extraction, composition of water extracts, total sugar recovery, and conversion of the polysaccharide of the exploded biomass to monosaccharide by a cellulosic enzyme mixture. Steam explosion followed by enzymatic saccharification was found to be an effective pretreatment for converting biomass into monosaccharides. However, the total sugar recovery, and thus the ethanol "potential", of the process was relatively insensitive to changes in reaction conditions due to the trade-off between xylose recovery and glucose recovery. Furthermore, it was found that the reaction ordinate concept was not universally valid for the variety of sample characteristics examined in this study. © 1998 Elsevier Science Ltd. All rights reserved

Keywords—Steam explosion; bagasse; ethanol; saccharification; enzymatic; reaction ordinate

1. INTRODUCTION

Various agencies within the State of Hawaii have long recognized the synergistic benefits that could result between Hawaii's established agriculture industry and an industry utilizing the lignocellulosic biomass byproduct. Of the many products capable of being produced from biomass, there are several factors unique to Hawaii which favor fuel grade ethanol as the product with the best chance of economic success. For example, the small state population generates little or no local demand for other products that can be produced from biomass. On the other hand, the cost of shipping products to the U.S. mainland or abroad, coupled with the high cost of land and labor resources in Hawaii, would make most products produced from biomass in Hawaii uncompetitive with other sources. Finally, all transportation fuels used in Hawaii must be imported at high cost. Thus, the break-even price for ethanol from a biomass-based refinery in Hawaii would be higher than on the mainland.

The present study was designed to evaluate steam explosion, followed by enzymatic saccharification, as a pretreatment for converting biomass into ethanol. Steam explosion was selected as the processing technology because of recent reports that steam explosion renders biomass more readily digestible by enzymes. ¹⁻³ Furthermore, steam explosion requires little or no chemical input and thus is environmentally benign relative to other technologies, such as acid hydrolysis; environmental concerns are of paramount importance when considering any new industry for Hawaii. The enzymatic hydrolysis was also selected for environmental reasons.

Other researchers have reported success with enzymatic treatment of water-washed steam exploded biomass, ¹⁻¹¹ the water extraction removes the majority of the hemicellulose fraction, as well as the degradation products, from the cellulose fiber. Until recently, this separation was desirable because an organism did not exist that could ferment both the pentose sugars from the hemicellulose and the glu-

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can from the cellulose. However, new developments in genetically engineered bacteria have produced such an organism. 12 Thus, it has become advantageous to retain the partially hydrolyzed hemicellulose with the cellulose. The aim of this study was to identify the optimum steam explosion condition such that the total fermentable sugar content after enzymatic hydrolysis was maximized. At the core of the study was a 95-sample test matrix. The large number of samples also provided an opportunity to evaluate the "reaction ordinate" concept as proposed by Overend and Chornet. 13 Parameters examined in light of the reaction ordinate included: Mass recovery; total water extractables; pH of the water extract; furfural content of the water extract; recovery of xylose; recovery of glucose; and total "ethanol potential". The ethanol potential was defined as the amount of ethanol that could be produced from the post-enzymatic hydrolysis broth, assuming 100% fermentation efficiency, as a percentage of the amount of ethanol that could theoretically be produced from the feedstock.

2. MATERIALS AND METHODS

Sugarcane bagasse was obtained from Oahu Sugar Company (Waipahu, HI); the material for the study was collected at one time and kept frozen until just before processing. The bagasse had a moisture content of 50.4%. Each of the steam explosion runs utilized 1 kg (wet) of fiber and was conducted in a 10-l jacketed steam exploder (Stake Technology) that incorporated a 160-l discharge chamber. The discharge chamber was vented through a cooling coil thereby allowing for complete material recovery.

A total of 95 different steam explosion conditions were examined. The conditions incorporated reaction temperatures of 188–243°C and run times of 0.5–44 min; the test matrix is presented in Table 1. At the conclusion of each run, the exploded fiber was quantitatively removed from the discharge chamber, the collected condensate was added back to the sample, and the exploded sample was sealed in a polyethylene container. All samples were kept frozen until analysis.

The first analytical procedure performed on the steam exploded samples was water extraction. The water extraction was conducted in duplicate using 500-ml glass columns fitted with water jackets. For each sample representative aliquots were quantitatively packed into the columns and distilled water was pumped up through the fiber using a peristaltic pump. Typically, approx. 100 g (dry) of fiber fit into a column. A total of 3000 ml of distilled water was used for each extraction. The distilled water reservoir and the water circulated through the column jackets were kept at 50°C.

The enzymatic assay used in the study was provided by the National Renewable Energy Laboratory. The basic procedure involved hydrolysis of 1.7 g dry weight of neverdried unextracted steam exploded fiber with commercial enzyme (Environmental Biotechnologies, Inc.). The hydrolysis was conducted in 100 ml of a yeast extract-peptone broth for 48 h at a pH of 5.0 and a temperature of 38°C. The approximate enzyme loading was 25 IFPU per gram of cellulose in the samples; the same amount of enzyme was used for each sample despite anticipated differences in cellulose content between different samples. At the conclusion of hydrolysis the samples were filtered, boiled for 5 min and an analytical quantity was retained for monosaccharides determination.

All analyses for saccharides and saccharide degradation products were performed according to procedures previously reported.¹⁴

3. RESULTS AND DISCUSSION

The "reaction ordinate" concept as proposed by Overend and Chornet has been reported to be a useful measure of reaction conditions during steam explosion processing. Because of the large number of samples in the study, it was convenient to convert the individual steam explosion conditions into their respective "log R_0 ", defined as:

Table 1. Steam explosion conditions

Temp (°C)	Residence time (min)
188	13, 15, 17,19, 21, 23, 26, 29, 32, 35, 38, 41, 44
193	9, 11, 13, 15, 17, 19, 21, 23, 25, 27, 29, 31, 33
199	7, 8, 9, 10, 12, 14, 16, 18, 20, 22
204	4.5, 5.5, 6.5, 7.5, 8.5, 9.5, 10.5, 11.5, 12.5, 13.5
210	3, 4, 5, 6, 7, 8, 9, 10, 11, 12
216	2.5, 3, 3.5, 4, 4.5, 5, 5.5, 6, 7
221	1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5
227	1, 1.5, 2, 2.5, 3, 3.5
232	0.75, 1, 1.25, 1.5, 2, 2.5
238	0.5, 0.75, 1, 1.25, 1.5, 1.75
243	0.5, 0.75, 1, 1.25

 $\log R_0 = \log\{t^* \exp[(T - 100)/14.75]\}\$ t = time, minutes; $T = \text{temperature, }^{\circ} \text{C}$

All of the conditions used in the study fell into the $log R_0$ range of 3.7–4.3.

In addition to identifying the optimum steam explosion pretreatment condition for preparing ethanol from sugarcane bagasse, the results from the various analyses were used to evaluate the efficacy of the reaction ordinate concept. Interpretation of the results was facilitated by preparing the data as 3-dimensional plots. All of the plots are in terms of temperature, reaction ordinate, and the variable of interest. Parameters examined were: mass recovery; weight loss (from water extraction); pH of water extracts; furfural content of water extracts; glucan to glucose conversion; xylan to xylose conversion; and ethanol potential.

3.1. Mass recovery

The mass recovery data are presented in Fig. 1(a) and (b). The recoveries varied from 78 to 99% of feedstock mass and favored low severity and/or low temperature. The mass recovery was fairly constant for R_0 across temperature.

3.2. Weight loss (from water extraction)

Weight loss due to water extraction varied from 9 to 28%; the data are presented in Fig. 2(a) and (b). The weight loss was not constant for constant reaction ordinate.

3.3. pH of water extracts

The pH of a water extract was lower for high severity and/or low temperature samples, although the trend is very light; values ranged from 3.2 to 4.7. The data are presented in Fig. 3(a) and (b).

3.4. Furfural content of water extracts

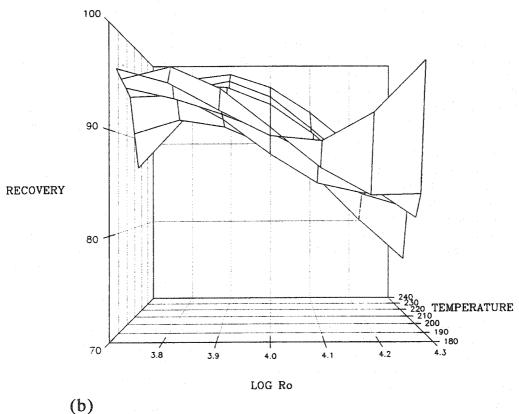
The furfural content of the water extracts, reported as a percentage of original xylose, was higher for higher severity and/or low temperature samples. The observed relationship with respect to temperature at constant severity could be due to the thermal decomposition/polymerization of the furfural in the reactor during the runs. The data are presented in Fig. 4(a) and (b).

3.5. Glucose conversion, xylose conversion, and ethanol potential

The glucose and xylose conversion values incorporate both the steam explosion pretreatment and the subsequent enzyme digestion. The values were calculated as the percentage of the original feedstock sugar present as monosaccharide in the enzyme liquor; the value was also dependent on the mass recovery. Only glucose and xylose were determined because they are the predominant sugars present in sugarcane bagasse. The ethanol potential was calculated by assuming 100% conversion to ethanol of the feedstock sugars and the monosaccharide present in the enzyme liquor; again the ethanol potential was plotted as a percentage of the theoretical (feedstock) value. For glucose conversion, the values ranged from 41 to 67% and were greater for higher severities; 216°C was an apparent temperature optimum. The xylose conversion ranged from 17 to 85% and was greater for lower severities; the xylose conversion was fairly constant for constant $\log R_0$. The ethanol potential ranged from 36 to 64% and was relatively insensitive to severity due to the inherent trade-off between the glucose and xylose yields. It is not known whether any of the conversion potentials could be enhanced by modification of the enzyme procedure. The glucose conversion, xylose conversion and ethanol potential data are presented in Figs 5-7, respectively. For comparison purposes, the glucose conversion, xylose conversion and ethanol potential of the unexploded bagasse (enzyme treatment only) were 13.8, 15.5 and 14%, respectively.

4. CONCLUSIONS

Steam explosion followed by enzymatic saccharification is an effective pretreatment for converting biomass into monosaccharides. It is possible that the conversion results from this study could be improved with modifications in the enzymatic digestion protocol. It is not known whether enzyme liquors would be suitable for fermentation because of the presence of known inhibitors, such as furfural. The explosion results indicate that steam explosion processing optimums are highly feedstock dependent, since different carbohydrates compositions dictate different conditions. Feedstocks high in xylose content require (a)
MASS RECOVERY FROM STEAM EXPLOSION TRIALS



MASS RECOVERY FROM STEAM EXPLOSION TRIALS

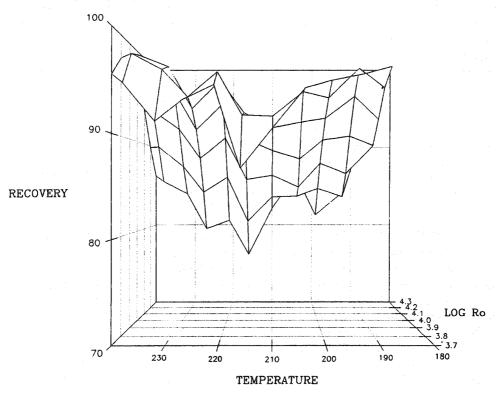
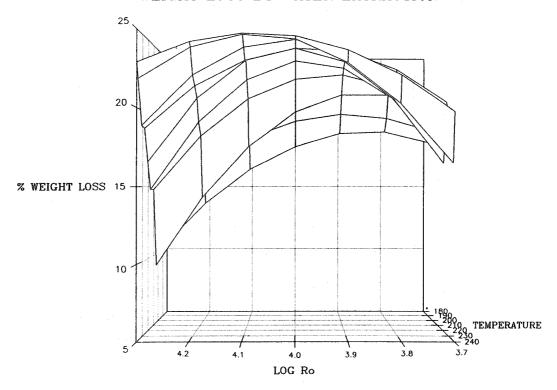


Fig. 1. (a) Mass recovery from steam explosion trials. (b) Mass recovery from steam explosion trials.

(a)

WEIGHT LOSS BY WATER EXTRACTION



(b)

WEIGHT LOSS BY WATER EXTRACTION

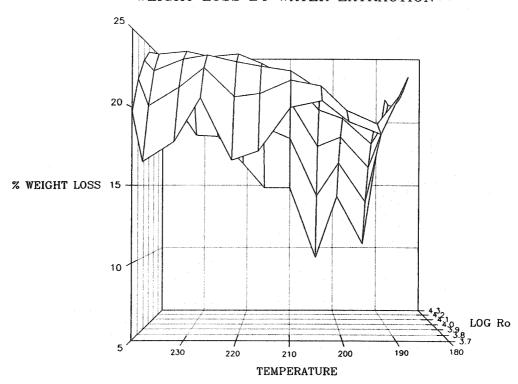
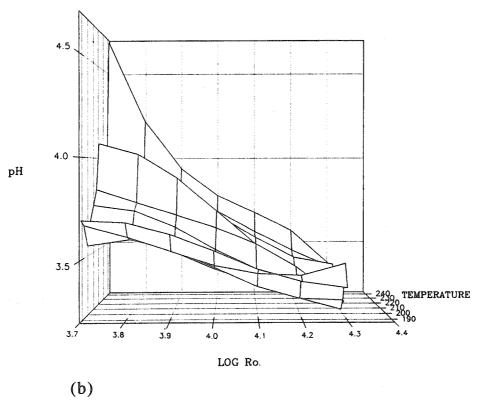


Fig. 2. (a) Weight loss by water extraction. (b) Weight loss by water extraction.

(a) $_{\mathrm{PH}}$ OF WATER EXTRACTS FROM EXPLODED BAGASSE



pH OF WATER EXTRACTS FROM EXPLODED BAGASSE

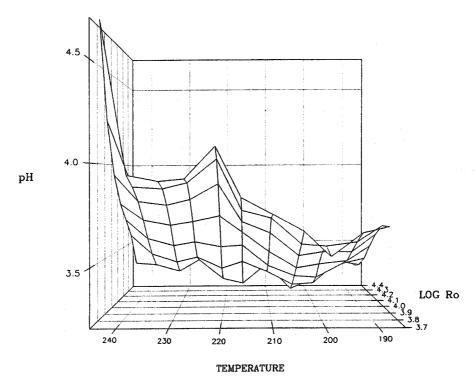
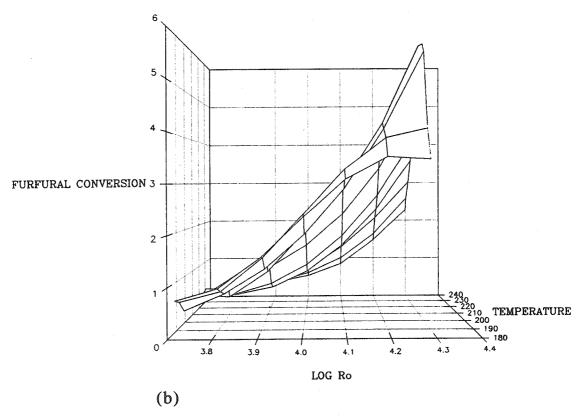


Fig. 3. (a) pH of water extracts from exploded bagasse. (b) pH of water extracts from exploded bagasse.

(a)
FURFURAL CONVERSION OF EXPLODED BAGASSE



FURFURAL CONVERSION OF EXPLODED BAGASSE

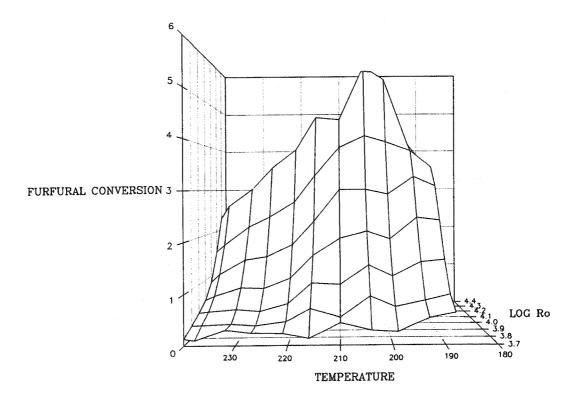
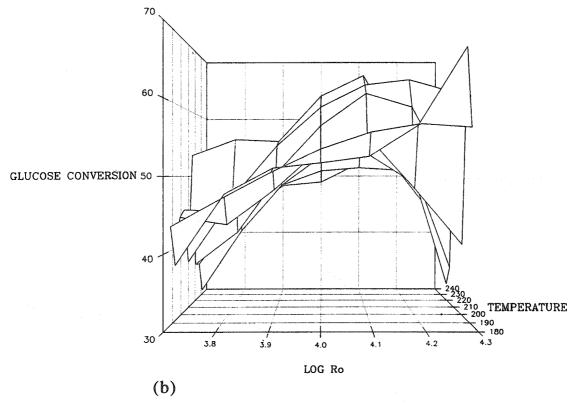


Fig. 4. (a) Furfural conversion of exploded bagasse. (b) Furfural conversion of exploded bagasse.

(a)
GLUCOSE CONVERSION OF EXPLODED BAGASSE



GLUCOSE CONVERSION OF EXPLODED BAGASSE

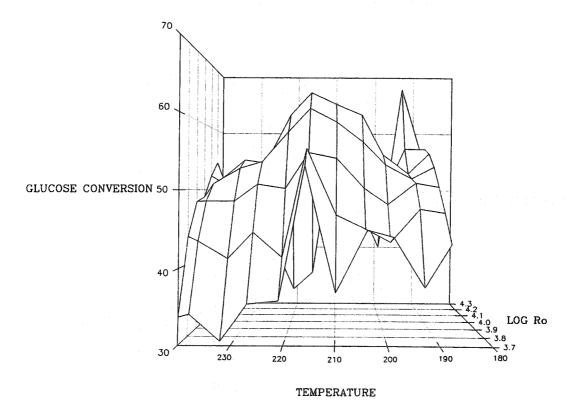
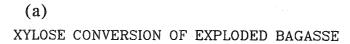
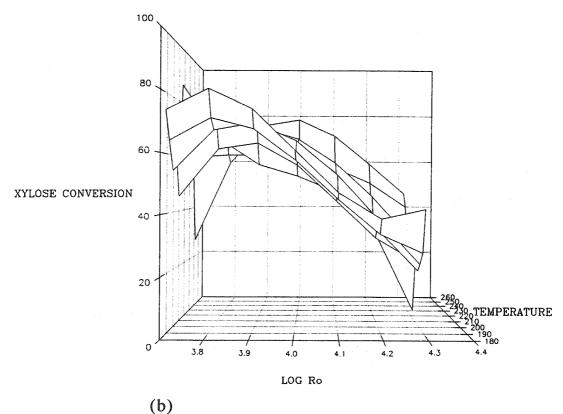


Fig. 5. (a) Glucose conversion of exploded bagasse. (b) Glucose conversion of exploded bagasse.





XYLOSE CONVERSION OF EXPLODED BAGASSE

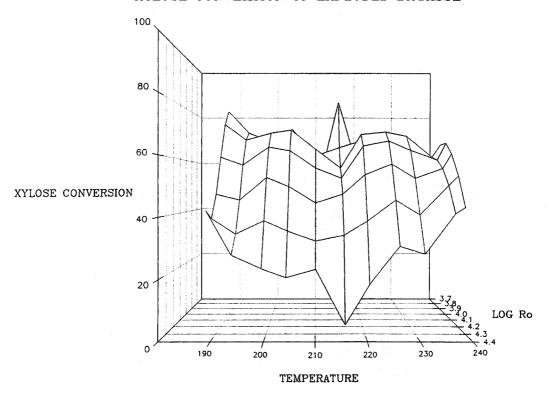
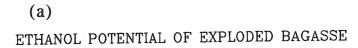
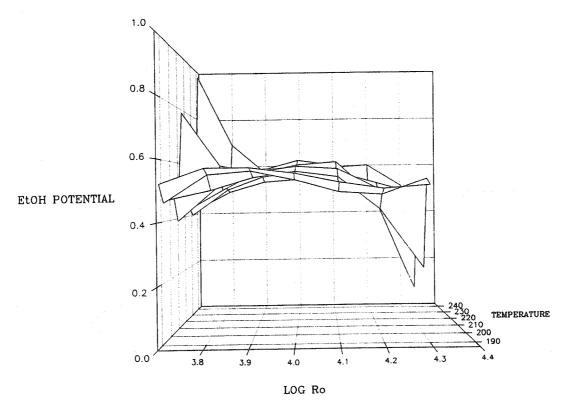


Fig. 6. (a) Xylose conversion of exploded bagasse. (b) Xylose conversion of exploded bagasse.





(b) ETHANOL POTENTIAL OF EXPLODED BAGASSE

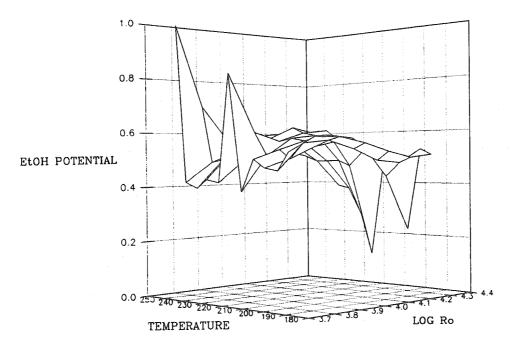


Fig. 7. (a) Ethanol potential of exploded bagasse. (b) Ethanol potential of exploded bagasse.

milder conditions, with shorter run times, than feedstocks lower in xylose (higher in glucose). For the variety of sample characteristics examined in this study, the reaction ordinate concept does not appear to be universally valid. Nonetheless, the reaction ordinate remains a useful bookkeeping method of reporting steam explosion conditions.

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