

An Advanced Bioprocessing Concept for the Conversion of Waste Paper to Ethanol

Scientific Note

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INTRODUCTION

Solid waste material from residential sources and some industrial areas represents a heterogeneous mixture that is made up predominantly of paper products, glass, plastics, and metals. Although conservation programs have had an important impact, the volume of this material remains quite large, estimated to be in excess of 180,000,000 ts/yr in the United States (1). Similar industrial solids waste would be expected to be appreciable, but somewhat less. Over 87% of this material was sent to landfill or incineration (the former was by far the largest portion), and it was estimated that only 13% of such material was recycled or further used (1). Because of new environmental restrictions and lack of suitable new sites, disposal by landfill is becoming prohibitively expensive, or even impossible, in some areas of the country.

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Waste Paper as a Feed Material

Typically, municipal solid waste is made up of approx 48% paper, 25% glass, 13% plastics, 6% steel, 2% aluminum, and 5% other materials (2). Newsprint alone makes up 14% of the waste. Large fractions of various types of the solid-waste materials could be effectively recycled if fractionation and segregation of the components were carried out. There seems to be a current trend toward segregation by the generator.

Segregated waste paper products could be an ideal feed material for biological conversion to sugars (conversion of cellulose and hemicellulose) with the possibility of subsequent conversion to ethanol. If half of the newsprint was recycled and only half of the remaining paper products were available as a feedstock, this would still constitute over 30 million ts/yr of prime chemical feedstock based on 1986 estimates (1). Assuming that 80% of the included cellulose was converted to ethanol, this would represent over 4,000,000,000 gal/yr of ethanol or over 0.3 quad of potential new chemical energy (3). Feed material costs should be significantly less than that of wood chips (approaching zero), with the result that use of such material could represent the first industrial entry of a ligno-cellulosic-based commodity chemical.

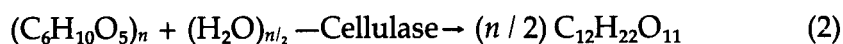
Biochemistry of Waste Paper Conversion

Waste paper is made up of three primary constituents: cellulose, hemicellulose, and lignin, on average constituting 61, 16, and 21%, respectively (4). The first two of these constituents are complex carbohydrates that can be hydrolyzed to the monomer sugars glucose and xylose by use of the appropriate enzyme systems. In this proposed processing concept, only cellulose will be utilized for subsequent bioconversion with the other constituents being used as a boiler fuel. Cellulase enzymes will be used as the biocatalysis for hydrolysis of cellulose to the intermediate product, glucose, after which it will be converted to ethanol. The process chemistry of interest is shown in the following equations (5-7):

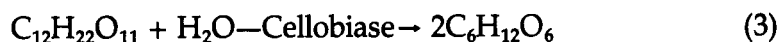
Overall cellulose hydrolysis to glucose (glucose inhibits the reaction)



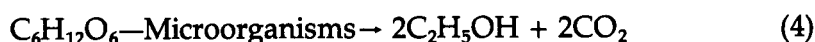
Cellobiose formation (an inhibiting intermediate product)



Hydrolysis of cellobiose to glucose



Bioconversion of glucose to ethanol



Unless extensive purification is carried out, the crude cellulase enzyme preparation is a mixture that has several isoenzymes, including those that interact with the internal structure and those that only interact with the end groups of the cellulose polymer. Cellobiose, an intermediate disaccharide that is also formed (Eq. [2]), and glucose inhibit the hydrolysis reaction with the former being more severely inhibitory. Fortunately, this chemical can be converted to glucose if a sufficient quantity of the enzyme cellobiase is present (Eq. [3]). This enzyme is also a constituent of the crude mixture of the cellulase enzymes, but usually is present at a relatively low concentration. Thus, in order to enhance the overall hydrolysis process, exposure to additional cellobiase could be necessary. Microbial fermentation of glucose to ethanol (Eq. [4]) would result in the final product.

Processing Steps

There have been previous estimates of a viable bioconversion processing system for waste-paper feed materials that appeared to be marginally economically feasible (4). However, the results of recent bioprocessing research and possible other advanced concepts could also be incorporated into such a processing scheme to make it even more economical. The primary processing steps must include:

1. A means of handling and size-reducing the waste paper;
2. The enzymatic hydrolysis system, in which the cellulase enzymes contact an aqueous slurry of the paper;
3. A means of producing the enzymes from the appropriate microorganisms;
4. A fermentation system for conversion of the hydrolyzed sugars to a crude aqueous ethanol; and
5. A means of concentrating and purifying the end product, probably by distillation.

Other necessary processing components will include filters, pumps, control system, and so forth, and necessary environmental control technology.

With conventional technology, the enzyme hydrolysis step (a stirred tank with cellulase enzymes at a concentration of 1–20 filter paper U/g of solid waste) requires too much time (> 40 h), enzyme costs are too high, the ethanol fermentation step (a batch-operated stirred tank with suspended yeast) is too long (> 24 h) and not well controlled, the separation and purification step (generally distillation) is too expensive, and waste treatment has not been well addressed. Advanced technologic concepts that address many of these processing problems should have a significant impact.

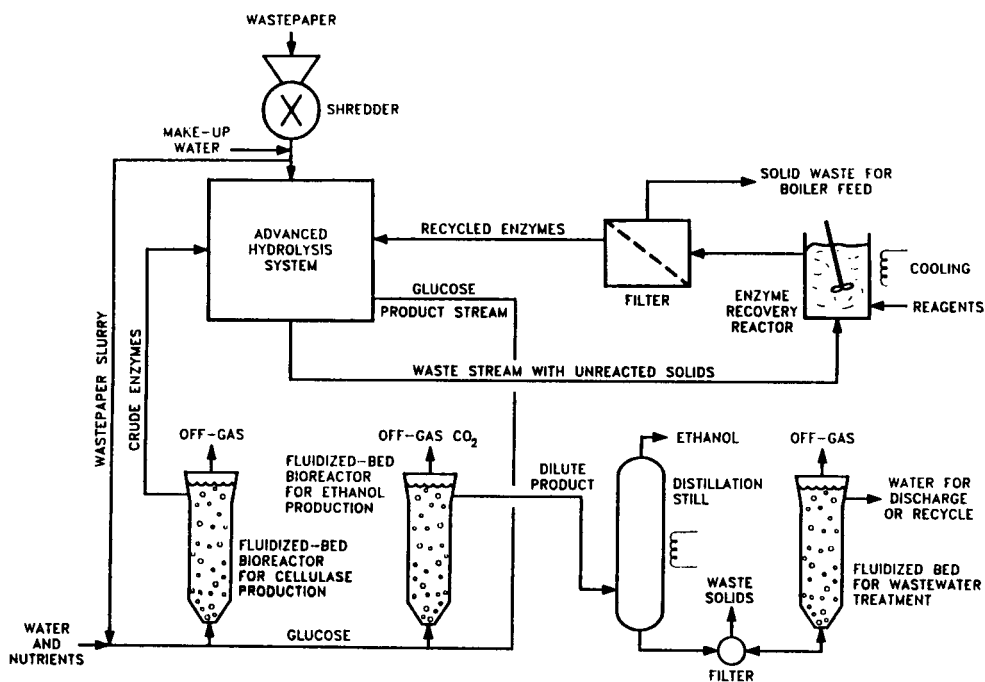


Fig. 1. Advanced process flow sheet for the biological production of ethanol from waste paper.

ADVANCED BIOPROCESSING SYSTEM

The most efficient bioprocessing flowsheet for processing waste paper should include all of the above processing steps while taking advantage of advanced concepts that have recently been developed or conceived (Fig. 1). The enhancements include a new concept for an enzyme hydrolysis bioreactor system that utilizes continuous attrition and membrane separations, a new approach to enzyme production and recycle, and advanced systems for ethanol production and waste-water treatment.

Feed Preparation

Size reduction of the waste paper and the formation of a thick aqueous slurry (pulping) will be required for further processing. Several adequate shredding systems, covering a wide range of sizes, are available on the market.

Cellulose Hydrolysis

The hydrolysis bioreactor represents the heart of the process. The paper slurry must be contacted with the cellulase enzymes for an adequate amount of time for saccharification to occur. Recent research at the

National Renewable Energy Laboratory has included the simultaneous saccharification of cellulose and fermentation of the resulting glucose to ethanol (SSF) (8). Not only does this combine two processing steps in one bioreactor, but it also maintains a low-glucose concentration, thus reducing product inhibition. Such systems are typically designed as simple, batch-fed, stirred tanks requiring many hours or even days for an acceptable yield. The resulting operating conditions for an SSF are a compromise that is not optimum for either of the combined steps. Research is continuing in this area with the prospects for future enhancements.

There are two basic problems that must be addressed in order to enhance this processing step. First, it appears that the enzyme process is accelerated when there is a large amount of substrate surface area available (smaller particle size) (9-11), and the rate of hydrolysis is further enhanced when fresh surface is continuously generated in an attrition bioreactor (12-14). There is also some indication that higher enzyme concentrations (> 100 FPU/g substrate) will also increase reaction rates, especially if there is sufficient surface area (15). In addition, as described above, we also know that the product and intermediate products of cellulose hydrolysis, glucose and cellobiose, inhibit the rate of reaction, so they must be maintained at low levels throughout the hydrolysis.

The concept of an attrition bioreactor has been further developed with continuous attrition being carried out in a high-speed centrifugal pump operating on a side stream (patent application submitted) (Fig. 2). Recent tests with an 800-mL bioreactor at 50°C and a pH of 5.0 have shown that there is a significant enhancement in enzyme hydrolysis of typical waste paper when continuous attrition is used in conjunction with reasonably high concentrations of cellulase of 80-160 FPU/g of substrate (Fig. 3). In this case, the substrate was pulped in a Waring Commercial Blender (Model 34BL97, Dynamics Corporation of America, New Hartford, CT) for 30 s prior to introduction to the bioreactor (*see* Fig. 3); the attriter was a high-speed, laboratory centrifugal pump (Model MDX-3, March Mfg., Inc., Glenview, IL). The cellulase product was Celluclast from Novo Nordisk Industries, Danbury, CT, and cellulase activity was measured by the method of Mandels et al. (16). Extra β -glucosidase activity was added by inclusion of 1 mL of Novozyme (Lot #513, Novo Nordisk Bioindustrials, Inc., Danbury, CT) per gram of waste paper. Glucose concentration was determined with a YSI Model 27 Analyzer (Yellow Springs Instrument, Inc., Yellow Springs, OH) after high-temperature enzyme deactivation.

Essentially complete hydrolysis could be achieved in 25 h, whereas $> 75\%$ conversion could be achieved in and about 6 h with a solids content of 2-4%. Enzyme deactivation in such a system was apparently quite low with over 95% of the initial activity still present after 24 h. These are preliminary results only, and additional confirmation will be required before the concept is shown to be technically and economically feasible.

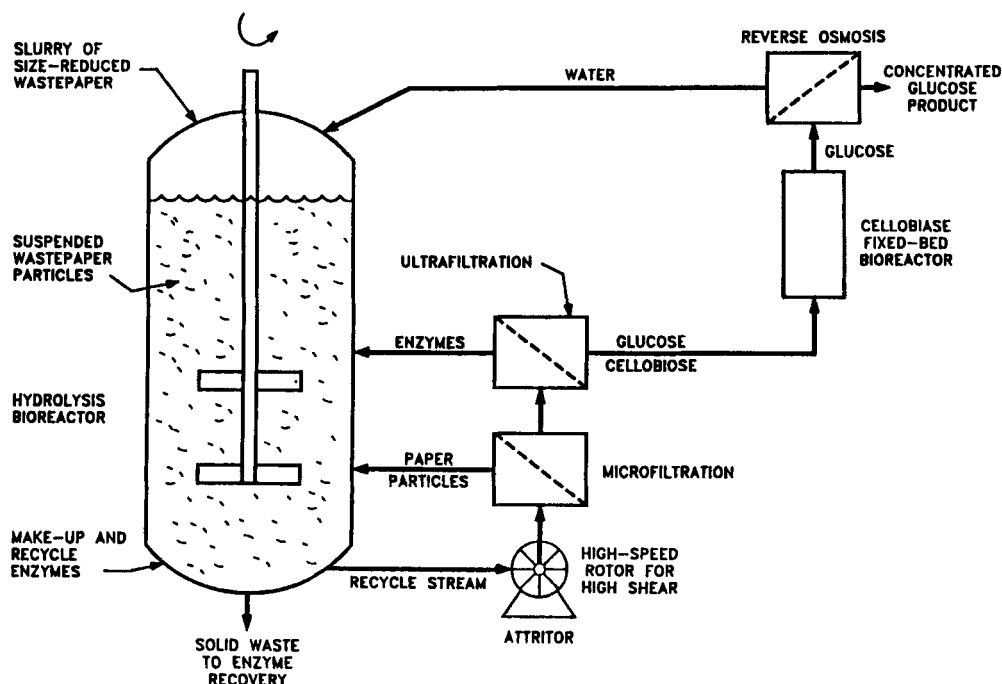


Fig. 2. Attrition bioreactor system composed of a stirred-tank hydrolyzer coupled to a recycle stream for high-shear attrition and glucose/cellobiose removal.

Membrane Processes for Cellobiose Conversion and Glucose Recovery

After attrition, a portion of the hydrolyzer side stream will be further processed to convert cellobiose to glucose and to remove glucose continuously. This should result in the maintenance of cellobiose and glucose levels in the hydrolyzer at low levels ($< 1\%$) while providing for a glucose product of higher concentration.

A microfiltration unit operating in a crossflow mode will be used to remove particulates and return them to the bioreactor. A typical separation medium membrane for such an application is a CMF membrane from the FilmTech Corporation (Minneapolis, MN). The technical staff from that company and the parent company, Dow Chemical Co., have indicated that similar systems have been operated without shutdown for periods up to a year (17). They believed that microfiltration would be required, in addition to the ultrafiltration unit, because of the high-solids loading. The primary operating cost of a such a system is the electricity used in the pump, which is estimated to require a 5-hp motor.

The filtrate from the microfiltration unit will then be processed through an ultrafiltration unit for recovery and recycle of the enzymes

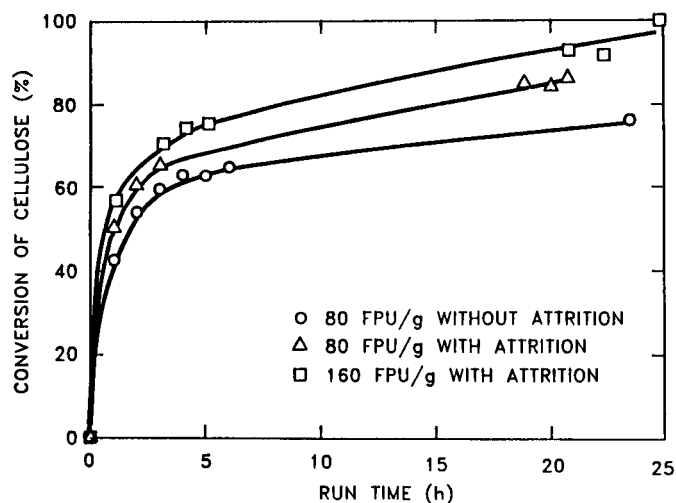


Fig. 3. The effects of attrition by a centrifugal pump in a recycle stream and cellulase concentration on the conversion of cellulose to glucose in waste paper at 50°C and a phosphate buffer at pH 5.0.

back to the hydrolyzer. A typical membrane for such a unit could be UF-38 from the FilmTech Corporation, again operating in the crossflow mode. Such systems have been used efficiently at large scale to recover various proteins in the food and dairy industries (17). The cost for such a system would be approx \$100,000, and the major operating cost is the electricity to operate a pump with a 5-hp motor.

The filtrate from the ultrafiltration unit would then pass through a fixed-bed bioreactor for conversion of cellobiose to glucose with the effluent progressing to a reverse-osmosis unit. The permeate from that unit would have a reduced glucose level, and it would be recycled back to the hydrolyzer, whereas the product stream would be enriched in glucose.

Cellobiose Conversion

It has been shown that the effects of cellobiose inhibition can be reduced by coupling a cellobiase reactor to the hydrolysis system (18). Such an approach can be most effectively done by using a fixed bed of immobilized cellobiase through which a portion of the hydrolyzer side stream can be forced to flow after filtration to remove particulates and enzymes (Fig. 2). The cellobiase can be immobilized by entrapment in stabilized gel beads (18). A fixed bed of the β -glucosidase in a columnar bioreactor operating at 40°C and a pH of 5.0 can be continuously convert >90% of cellobiose to glucose with a residence time of <90 min. Cellobiose concentrations of up to 100 mM have been effectively reduced. The immobilized biocatalyst was effective during several months of operation without apparent loss of activity (18).

Enzyme Recycle

Enzymes in the side stream are returned to the hydrolyzer by ultrafiltration. However, these biocatalysts can also be lost by adsorption to the solid residues. There is an indication that an alteration in the chemical and physical environment of the residue will cause a significant portion of the adsorbed enzymes to be released. Although it is somewhat dependent on the specific isoenzyme, the adsorption capacity of such residues apparently decreases by up to a factor of 3 when the pH is increased from 5.0 to 7.0 (19-21), and one researcher has reported that the adsorption capacity can be further reduced by decreasing the temperature to 5°C (19). Preliminary results at this laboratory indicate that a significant amount of the enzyme is released when the fermentation broth is removed and a fresh solution at a pH of 7.0 is used. We have not seen the effect of temperature decrease, but our work and that of others suggest that more than 80% of the adsorbed cellulase can be recovered and recycled with a change in pH. Scouting tests were made in a 150-mL, temperature-controlled bioreactor with 25 g/L waste-paper residue and cellulase enzymes at a concentration of 25 FPU/g of waste paper. The protein in the bulk solution was determined by the method of Bradford (22). Greater than 95% of the cellulose activity was maintained during this test.

Enzyme Production

The economics of purchasing the crude enzyme extract from a commercial enzyme manufacturer will be continuously monitored. However, early indications are that it will be less expensive to produce this bioreagent on-site. Rather than using a conventional batch-fed stirred tank for the bioreactor, our plans are to produce the crude enzyme extract from a continuous, columnar, fluidized-bed bioreactor utilizing immobilized microorganisms, either the fungus *Trichoderma reesei* or an appropriate bacterium (5,23).

Tests with *T. reesei* indicate that it can be effectively immobilized in the spore form into carrageenan beads followed by microbial growth, and these immobilized microorganisms can then be induced to produce cellulytic activity by sophorose and various carbohydrate oligomers. These latter are relatively inexpensive. Several different types of bacteria have been effectively immobilized and used in continuous fluidized-bed bioreactors (24,25). Various cellulase-producing bacteria, for example, *Pseudomonas fluorescens*, have been investigated for cellulase production at this laboratory and others (23). Such bacteria can also be immobilized and induced to produce the necessary enzymes. In this laboratory, we have found bacterial systems to be somewhat easier to manipulate, immobilize, and control, so they are the prime candidates for the continuous production of cellulases in a fluidized-bed bioreactor.

Bioconversion of Glucose to Ethanol

Fermentation of the sugars to ethanol or other oxychemicals will be carried out in an advanced fluidized-bed bioreactor utilizing immobilized microorganisms at high concentrations. This approach has been shown to be at least ten times more efficient than conventional technology when *Zymomonas mobilis* is used with industrial feed materials (24,26). In such systems, ethanol concentrations of >75% (wt/vol) can be maintained at an overall volumetric productivity >60 g/L·h. The immobilization material will be either crosslinked, industrial-grade carrageenan or modified bone gel that has been crosslinked. The resulting CO₂ from this fermentation (over 800,000 ts/yr for a 25M-gal/yr plant) could be recovered as a useful byproduct.

Product Concentration and Purification

Concentration and purification of the end product will initially be by relatively conventional distillation, although incorporation of a concentration step based on adsorption could be considered as a future upgrade. In the latter case, if a compatible solid sorbent can be found that also has high affinity for the product, a concept that was recently developed at this laboratory will be used. This concept utilizes a bioparticle fluidized-bed system that allows combination of both fermentation and product recovery by adsorbent particles moving countercurrent through a fluidized bed of biocatalyst particles (27). Such an innovative approach to product recovery could further enhance the economics of the process, but it needs to be more completely developed.

Environmental Control Technology

Although this bioprocessing system is expected to have minimal impact on the environment, there will be a waste-water stream containing carbonaceous material that will have to be treated before release or recycle. Fluidized-bed bioreactors with immobilized biodegrading biocatalysts in an aerobic environment will probably be the best approach. With such systems, it has been shown that carbon levels of several hundred ppm can be reduced to <10 ppm with a residence time of <1 h in the fluidized-bed bioreactor (28). Most of the solid residues will be used as an energy source, although the potential excess energy that could be produced was not claimed as a credit in the following cost estimates.

PROCESS ECONOMICS

As previously mentioned, a much earlier cost estimate for the conversion of waste paper to ethanol had been made by Wilke et al. (4) that

Table 1
Estimated Capital Costs of 25,000,000-Gal/Yr Ethanol Plants^a

Processing area	Woody biomass and SSF, (1986 technology), \$M	Waste paper and advanced tech., \$M
Feed handling	7.9	7.9
Acid pretreatment	11.6	—
Enzyme production	15.1	5.0
Saccharification and fermentation	40.8	27.2
Distillation	11.4	11.4
Off-site tankage	4.6	4.6
Environmental control	9.5	6.3
Utilities	33.8	30.9
Total	134.7	93.3

^aFrom ref. 29 with modifications for advanced processing of waste paper and costs adjusted to early 1993 from cost indexes in ref. 31.

showed marginal economic viability for the technology as of 1976. More recent (1988) comprehensive process economic analyses were made on a somewhat similar processing system utilizing the conversion of woody biomass to alcohol with enzymatic hydrolysis of the feedstock and fermentation of the resulting sugar (29,30). This economic study was made with conventional technology except for the hydrolysis, which was simultaneous saccharification and fermentation. The resulting cost estimates were made to establish a baseline case from which the effects on overall costs could be evaluated for various advanced processing concepts. Woody biomass requires a processing system that is rather similar to that required for waste paper, except that the wood requires extensive preprocessing, and it is a much more expensive feed material than waste paper.

The results of the cost estimates indicate that with conventional technology, the capital cost for a plant that will produce 25 million gal/yr of fuel-grade ethanol from woody biomass would be \$134.7 million adjusted to early 1993 dollars (Table 1) (31). Estimated production cost for such a facility was \$1.92/gal (Table 2), a cost that would be most uneconomic. The authors of that study speculated that if advanced processing concepts could be integrated into such a system, then production costs could be <\$1.00/gal (31).

This process analysis was adjusted by the ORNL staff to accommodate waste paper as the feed material with the previously mentioned advanced processing steps. Further, it was assumed that the feed material had zero cost, 80% of the cellulose was converted to ethanol, lignin and hemicellulose were used as boiler fuel, 80% of the enzymes were recovered and recycled, and the ethanol yield was 98% of theoretical.

Table 2
Estimated Production Costs of 25,000,000-Gal/Yr Ethanol Plants^a

	Woody biomass and SSF, (1986 technology), \$/gal ethanol	Waste paper and advanced tech., \$/gal ethanol
Raw materials		
Wood or paper	.684	.000
chemicals	.057	.019
Utilities	.007	.007
Labor	.083	.076
Overhead and maintenance	.395	.322
Operating costs	1.226	.424
Capital charges ^b	.695	.482
Ethanol selling price	1.921	.906

^aFrom ref. 29 with modifications for advanced processing of waste paper and costs adjusted to early 1993 from cost indexes in ref. 31.

^bCapital recovery factor = 0.13, 15% internal rate of return, 20-yr straight-line depreciation.

All of these assumptions have been verified on a small bench scale, and they would result in a requirement of about 1100 dry ts/d of waste paper for the 25,000,000-gal/yr plant.

The specific adjustments to the cost estimate include:

1. Elimination of the pretreatment step, because the paper product has already had considerable preprocessing;
2. The use of enzyme recycle, which should reduce enzyme production requirements to only 1/3 that of the base case;
3. Use of an advanced hydrolysis attrition reactor and a fluidized-bed fermenter will require only 2/3 of the capital of the base case for these two processing steps, since the resident time of this reactor size will be reduced by at least a factor of 2; and
4. The introduction of advanced environmental control technology, which will cut the capital cost for that item by 1/3.

Based on the projected reactor sizes, the capital costs for the hydrolysis and fermentation steps should be even less than the proposed reduction by 1/3, but, there will be some additional cost because of the incorporation of side-stream processing that will include the attriter (modified centrifuger pump) and the membrane systems. Estimated capital costs for the entire system would be \$93.3 million.

These assumed processing advances also resulted in a much lower production cost (Table 2). The major change was the elimination of feed materials cost and a decrease in the chemicals cost, since dilute-acid treatment was not required. There was also a small decline in the labor, overhead, and maintenance charges because fewer and more efficient unit

operations were being used. Capital charges were, of course, significantly less. The resulting estimated production cost was \$0.91/gal ethanol. This would be very competitive when compared to the open market cost of >\$1.15/gal that is currently quoted (32). Such a cost differential would allow there to be a modest increase in the price of mixed waste paper to about 1/3 that of wood chips. Because most waste paper is still sent to a landfill with tipping costs in excess of \$30/t (33), the cost of this waste material is not expected to be prohibitive, especially until many such plants are built.

CONCLUSIONS

Waste paper is a plentiful and low-cost lignocellulosic feed material that may represent the most direct way to penetrate the market with an advanced bioprocessing system. Innovative bioprocessing concepts integrated into such a system for the production of ethanol should be economically viable. Several of the proposed processing advances for such a system have only been studied on a laboratory scale, so a more thorough process development and scale-up effort will be required.

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