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Pretreatment of Lignocellulosic Municipal Solid Waste by Ammonia Fiber Explosion (AFEX)

MARK T. HOLTZAPPLE,* JOSEPH E. LUNDEEN, RUSSELL STURGIS, JOHN E. LEWIS, AND BRUCE E. DALE

Department of Chemical Engineering, Texas A&M University, College Station, TX 77843

ABSTRACT

The Ammonia Fiber Explosion (AFEX) process treats lignocellulose with high-pressure liquid ammonia and then explosively releases the pressure. The combined chemical effect (cellulose decrystallization) and physical effect (increased accessible surface area) dramatically increase lignocellulose susceptibility to enzymatic attack. For example, bagasse digestibility is increased 5.5 times and that of kenaf core is increased 11 times using extracellular cellulases from *Trichoderma reesei*. In this study, we applied the AFEX process to mixed municipal solid waste (MSW) and individual components (e.g., softwood newspaper, kenaf newspaper, copy paper, paper towels, cereal boxes, paper bags, corrugated boxes, magazines, and waxed paper). Softwood newspaper proved to be the most difficult component to digest because of its high lignin content. A combination of oxidative lignin cleavage and AFEX was required to increase softwood newspaper digestibility substantially, whereas AFEX alone was able to make kenaf newspaper digestible. Because most MSW components have been substantially delignified in the paper-making process, AFEX only marginally increased their digestibility.

Index Entries: Municipal solid waste; Ammonia Fiber Explosion; pretreatment; cellulase; ethanol.

*Author to whom all correspondence and reprint requests should be addressed.

INTRODUCTION

Currently, the US annually produces about 165 million t of municipal solid waste (MSW), or about 1.8 kg/person/d (*1*). About 73% is disposed of in landfills, 13% is incinerated, and 14% if recycled. Recycling (*2*) is often considered the most environmentally sound approach; however, it is not a panacea. Paper can only be recycled two to three times before the fibers become unacceptably short. Also, paper must be sorted, and the deinking procedures can actually make recycled pulp more expensive than virgin pulp. Incineration suffers from the NIMBY (not-in-my-backyard) syndrome because of public concern over air emissions and ash disposal. Other approaches to MSW disposal/utilization include composting (*3*), pyrolyzing/gasifying (*4–6*), methane production in landfills (*7*) or fermentors (*8,9*), and chemical/fuel production (e.g., organic acids or ethanol) (*10,11*).

Chemical/fuel production is the focus of this article. Table 1 shows that about 60% of the dry matter in "typical" MSW is lignocellulose (*1,12*). It could potentially be converted to 228 L ethanol/wet t MSW. If all the lignocellulose in MSW were converted to ethanol, it would annually produce about 38 billion L, or 7.3% of the 510 billion L of liquid transportation fuels annually consumed in the US (*13*).

Lignocellulose is generally resistant to enzymatic hydrolysis and requires pretreatment. In the AFEX pretreatment (*14–17*), lignocellulose is soaked with high-pressure (ca. 27 atm) liquid ammonia at moderate temperatures (ca. 65°C) for about 15 min causing cellulose to decrystallize. Then the pressure is instantaneously released, causing the ammonia to flash violently and disrupt the fibrous structure. The combined chemical effects (cellulose decrystallization, hemicellulose prehydrolysis, lignin alterations) and physical effect (increase in accessible surface area) markedly increase the susceptibility of lignocellulose to enzymatic hydrolysis. Agricultural wastes respond well to AFEX treatment; bagasse digestibility is increased 5.5 times, and kenaf core is increased 11 times (*17*). The cost of AFEX is estimated to be $25–30/t, depending on the electricity price (*16*).

MATERIALS AND METHODS

Sample Preparation

Softwood and kenaf newspaper were shredded and then ground in a Wiley mill with a 2-mm screen prior to AFEX treatment. After AFEX treatment, the sample was ground in a Wiley mill with a 1-mm screen, so it could pass through a 30-mesh sieve. The MSW components (e.g., copy paper, corrugated boxes, cereal boxes, and so on) were ground in a Wiley mill to pass a 30-mesh screen. The mixed MSW was ground in a Wiley mill with a 2-mm screen.

Table 1
Composition and Ethanol Potential of 1 T Raw Wet MSW (*1,12*)

	Wet wt, kg	Dry wt, kg	Assumed carb content, %	Estimated potential ethanol,* L
Biodegradable				
Paper				
Corrugated boxes	111	100	90	55
Newspaper	74	66	80	32
Packaging	57	51	70	22
Office and printer paper	53	48	100	29
Miscellaneous nonpackaging	33	30	70	13
Books and magazines	27	24	50	12
Tissue paper	16	15	100	9
Subtotal	371	333		172
Yard waste				
Grass	60	21	70	9
Brush	22	13	70	6
Greens	22	9	70	4
Leaves	75	37	70	16
Subtotal	179	80		34
Food waste				
Garbage	68	19	70	8
Fats	13	13	0	0
Subtotal	81	32		8
Wood	38	30	70	13
Subtotal	669	476		228
Refractory				
Glass	97	95	0	0
Metals	96	93	0	0
Plastic	72	71	0	0
Rubber and leather	25	24	0	0
Textiles	21	19	0	0
Miscellaneous	19	17	0	0
Subtotal	330	319		0
Grand total	1000	795		228

*Assume ethanol yield is 85% of theoretical.

AFEX Treatment

Prewetted MSW was charged to a 4-L reactor (*16,17*). Ammonia was added, and the cold reactor was electrically heated. It required a heat-up time of about 25 min to reach the desired temperature (ca. 65°C). After the desired soak time, the pressure was explosively released into a blowdown tank to a final pressure of 1.5 atm absolute. The MSW was then removed and air-dried to remove residual ammonia.

Hydrogen Peroxide Treatment

Some softwood newspaper was treated with hydrogen peroxide to cleave lignin prior to AFEX treatment (*18*). Sodium hydroxide was slowly added to a 12% H_2O_2 solution to adjust the pH to 11.5. This procedure was performed in an ice bath to remove heat and prevent a runaway reaction. The concentrated H_2O_2/NaOH solution was diluted to various concentrations (1, 2.5, 5, and 10%). Softwood newspaper was added to the dilute solutions to make a 5.9 wt% slurry. The newspaper soaked at room temperature (23°C) for 6 h, and then was washed three times with distilled water, dried, and reground in the Wiley mill to pass a 30-mesh screen.

Peracetic Acid Treatment

Some softwood newspaper was treated with peracetic acid to cleave lignin prior to AFEX treatment (*18*). Peracetic acid was prepared by slowly adding acetic anhydride to 35% hydrogen peroxide (3:2, by volume). (**IMPORTANT:** Reversing the order of chemical addition may result in an explosion.) The stirred reaction vessel was placed in an ice bath that maintained the reaction temperature at 60–70°C. The solution stood for 24 h at 23–25°C prior to use. It was used immediately, since decomposition occurs at a rate of 0.2–1.0%/d. The resulting peracetic acid concentration was measured by the potassium permanganate–sodium thiosulfate method. The concentrated peracetic acid was diluted to 0.3, 1.0, and 2.0% with distilled water. Softwood newspaper was added to the dilute solutions to make a 9.1% slurry and treated for 1 d at room temperature. The paper was then filtered, washed five times with distilled water, dried, and reground in a Wiley mill to pass a 30-mesh screen.

Saccharification

AFEX-treated biomass (7.5 g) was added to 150 mL of 0.05M, pH 4.8, citrate buffer, which was prewarmed to the reaction temperature of 50°C in a 100-rpm shaking water bath. Genencor 300P cellulase/hemicellulase with a filter paper activity (*19*) of 132 IU/g dry powder was added, so that the cellulase loading was 5 IU/g dry biomass. Since *Trichoderma reesei* cellulase/hemicellulase has a specific activity of about 0.7 IU/mg protein, this enzyme loading corresponds to 7 kg cellulase protein/t dry biomass. Novo 188 cellobiase with an activity (*20*) of 250 CBU/mL was added so that the cellobiase loading was 28.4 CBU/g dry biomass. Liquid samples (1 mL) were taken using a mechanical pipet with an enlarged tip. The samples were boiled in a sealed test tube for 30 min to denature the enzyme. The boiled samples were filtered through 0.22-μm nylon membrane filters. The reducing sugar concentration was measured using the DNS assay (*21*). (Note: All reducing sugar yields are expressed as mg Equivalent glucose/g dry substrate, since glucose was used as the calibration standard.) The hydrolysis was performed for 3 d with samples taken

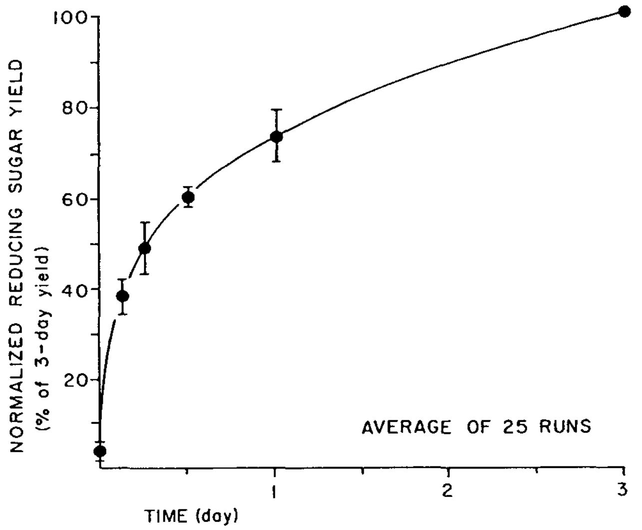

Fig. 1. Normalized hydrolysis profile for softwood newspaper. (Three-day yield = 100, error bars are ±1 SD.)

periodically during the run. A correction (about 50 mg Eq glucose/g dry biomass) was applied to the reducing sugar measurement to account for the sugar in the enzyme solution.

RESULTS AND DISCUSSION

Softwood Newspaper

Most of the newspaper in the US is derived from softwood trees because of their long fibers and abundance. Newspaper consists mainly of mechanical pulp (i.e., mechanically ground raw wood) with small amounts of kraft pulp to add strength and improve color. Newspaper has a very high lignin content (18–30%) (*22*), so it not very digestible.

Including controls, a total of 25 softwood newspaper samples were hydrolyzed. In all cases, the curves that describe sugar production vs time had a similar shape; only the scale was different. Figure 1 shows sugar production vs time with the data normalized to the 3-d yield. The 3-d sugar yield is a convenient measure of enzymatic susceptibility, since essentially no further hydrolysis is observed with increasing incubation time. The normalized curve allows determination of sugar yields at times other than 3 d. For example, about 50% of the 3-d sugars were released in 6 h, whereas 75% were released in 24 h.

The softwood newspaper samples were treated according to the conditions described in Table 2. Figure 2 shows the effect of ammonia loading on reactivity as indicated by the 3-d yield. A sharp peak occurred at 2.8 kg NH_3/kg dry softwood newspaper, so this was used in subsequent studies.

Table 2
Summary of AFEX Treatment Conditions

Experiment	NH_3 loading,* kg/kg	H_2O loading,* kg/kg	Reactor temperature, °C	Time at temperature,† min
Softwood newspaper				
1	0.8–5.8	0.25	75	10
2	2.8	0.25	75	0–85
3	2.8	0–0.5	75	10
4	2.8	0.07	50–100	10
5 (H_2O_2)	2.0	0.07	65	10
6 (peracetic acid)	2.0	0	65	10
Kenaf newspaper				
1	0.8–4.8	0.25	75	10
2	2.8	0.25	75	0–60
3	2.8	0–0.5	75	10
4	2.3	0.08	50–100	10
MSW components				
1	3.0	0.06	65	10
Mixed MSW				
1	3.0	0.06	65	10

*Note: Loadings are expressed on a dry-wt basis.
†Note: Time-to-temperature is ca. 25 min.

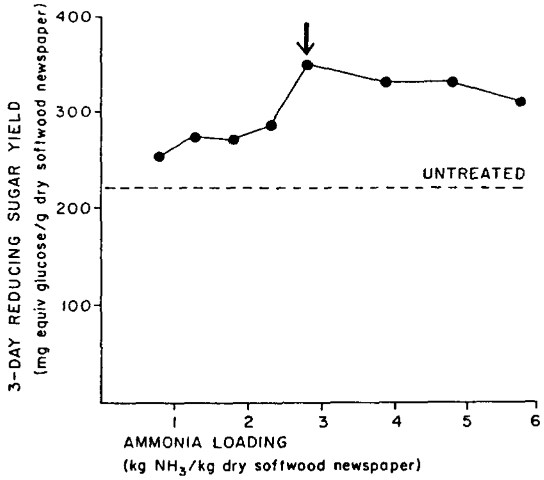

Fig. 2. Softwood newspaper experiment 1: Effect of ammonia loading on sugar yields.

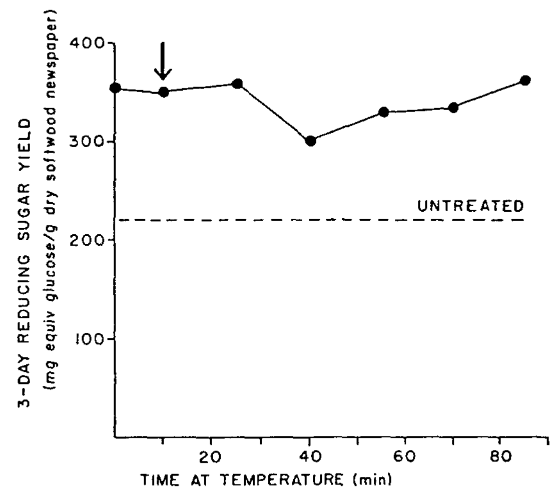

Fig. 3. Softwood newspaper experiment 2: Effect of treatment time on sugar yields. (Note: Time-to-temperature is ca. 25 min.)

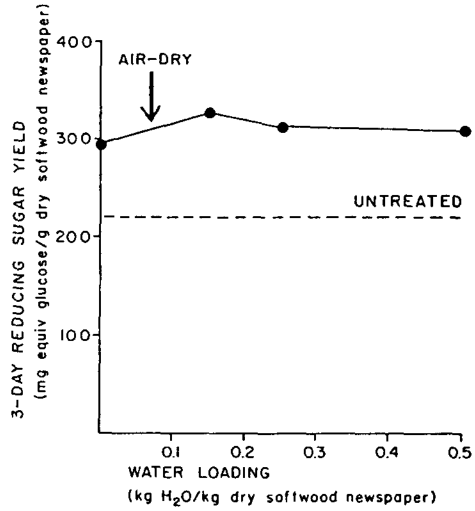

Fig. 4. Softwood newspaper experiment 3: Effect of water loading on sugar yields.

Figure 3 shows that samples with zero time at temperature were fairly reactive since the heat-up time was so long (about 30 min). Ten minutes were selected as the treatment time to guarantee the softwood newspaper had sufficient ammonia contact.

Figure 4 shows that the effect of water loading is fairly flat, so air-dry newspaper was used in subsequent studies. Figure 5 shows that there was very little effect of temperature, although there may be a slight decrease above 65°C. To be consistent with kenaf newspaper, 65°C was selected as the best temperature.

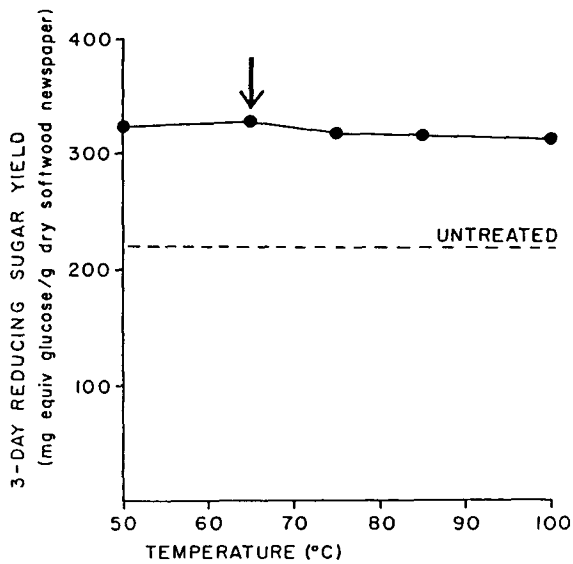

Fig. 5. Softwood newspaper experiment 4: Effect of temperature on sugar yields.

The sugar yield from raw newspaper was about 220 mg Eq glucose/g dry softwood newspaper. After AFEX treatment, the sugar yield was typically about 330 mg Eq glucose/g dry softwood newspaper, a 50% increase in digestibility.

Hydrogen peroxide is a well-known delignifying agent for paper pulp bleaching (*23*). Since lignin hinders enzyme access to carbohydrates, hydrogen peroxide pretreatment has been studied by Gould (*24,25*) and others (*26,27*) to increase biomass digestibility. The pH must be about 11.5 to favor the hydroperoxy anion (HOO^-), which is believed to be the reactive species that oxidizes lignin. Since ammonia decomposes and calcium hydroxide is not a strong enough base to raise the pH, sodium hydroxide was used as recommended by Gould. Sodium hydroxide has the added benefit of swelling the biomass to increase digestibility. Figure 6 shows that the highest level of H_2O_2/NaOH pretreatment increased the digestibility to a level near that obtained with AFEX treatment. When H_2O_2/NaOH-treated softwood newspaper was subsequently AFEX treated, the digestibility substantially increased in an additive, rather than synergistic, manner. Using the highest hydrogen peroxide loading, the sugar yield was raised from 220 to 502 mg Eq glucose/g dry softwood newspaper, a 128% digestibility increase. Unfortunately, the H_2O_2/NaOH pretreatment appears to be uneconomical because of high chemical costs. At an H_2O_2 loading of 0.4 kg H_2O_2/kg dry softwood newspaper, the corresponding NaOH loading is 0.18 kg NaOH/kg dry softwood newspaper. Using the costs cited in Table 3, the chemical cost is \$616/t biomass for H_2O_2 and \$119/t biomass for NaOH. Most of the NaOH could probably be recycled, but it would be difficult to do since solubilized organics would have to be purged from the recycle stream.

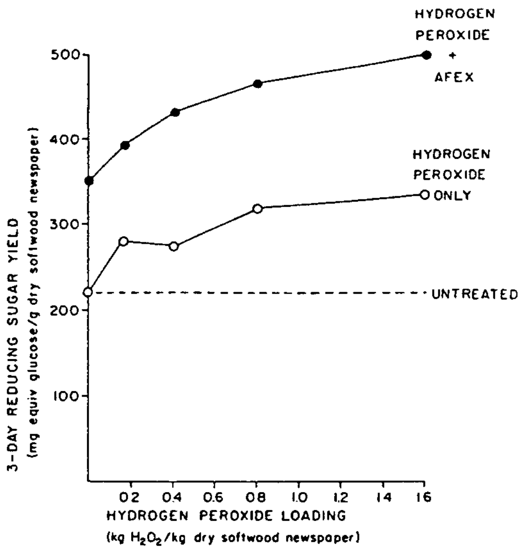

Fig. 6. Softwood newspaper experiment 5: Effect of hydrogen peroxide and AFEX treatment on sugar yields.

Table 3
Relevant Chemical Costs

Chemical	Cost	Reference
Hydrogen peroxide	\$1.54/kg H_2O_2	*31*
Sodium hydroxide	\$0.66/kg NaOH	*31*
Acetic anhydride	\$1.05/kg acetic anhydride	*31*
Ammonia (Gulf Coast)	\$0.09/kg NH_3	*31*
Ammonia (Midwest)	\$0.16/kg NH_3	*31*
Food-grade *T. reesei* cellulase	\$33.00/kg cellulase protein	*32*
Fuel-grade *T. reesei* cellulase	\$6.60/kg cellulase protein*	*33*

*Estimate.

Peracetic acid is another well-known bleaching agent (*23*). Figure 7 shows that peracetic acid alone does not substantially enhance digestibility since it has no swelling action. However, when peracetic acid-treated softwood newspaper is subsequently AFEX treated, then the digestibility significantly increases in a synergistic, not additive, manner. (This synergy has previously been noted in the literature where peracetic acid was combined with steam explosion pretreatment [*28*]). Under the highest peracetic acid loading, the sugar yield increased from 220 to 579 mg Eq glucose/g softwood newspaper, a 163% increase. Unfortunately, peracetic acid is also too expensive to be economical. It is synthesized from acetic anhydride and hydrogen peroxide (*see* costs in Table 3). The chemical cost at a loading of 0.1 kg peracetic acid/kg dry softwood newspaper is \$210/t biomass (assuming stoichiometric peracetic acid yields from acetic anhydride and hydrogen peroxide).

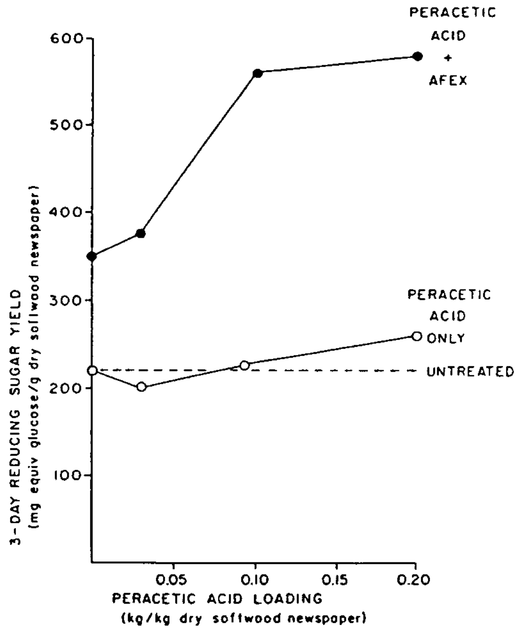

Fig. 7. Softwood newspaper experiment 6: Effect of peracetic acid and AFEX treatment on sugar yields.

Although neither the hydrogen peroxide nor the peracetic acid treatment appears to be economical, they indicate that lignin limits the reactivity of softwood newspaper. (An alternative explanation for low softwood newspaper digestion was that the Genencor 300P cellulase/hemicellulase was ineffective against mannan, a dominant hemicellulose component in softwoods. However, hydrolysis with high-mannanase Genencor M103S cellulase/hemicellulase failed to increase digestibility.) Previously, a mild organosolv delignification has been unsuccessfully attempted (*16*), but perhaps the conditions must be more severe to effect greater delignification.

Kenaf Newspaper

Kenaf is a fibrous annual plant that grows in the US Cotton Belt with annual yields of about 10–18 t/ha (*29*), about 4–7 times that of natural-stand softwoods and 1.5–2.6 times that of intensively cultivated softwoods (*30*). It is composed of about 25% "bast fibers" (long fibers) and 75% "kenaf core" (short fibers), all of which may be pulped to make paper. The *Bakersfield Californian* has successfully published a kenaf newspaper issue on July 13, 1987.

The normalized sugar yields for kenaf newspaper were very similar to those shown in Fig. 1 for softwood newspaper; about 50% of the sugars were released in 6 h and almost 80% in 24 h. Table 2 reports the treatment conditions used in each study.

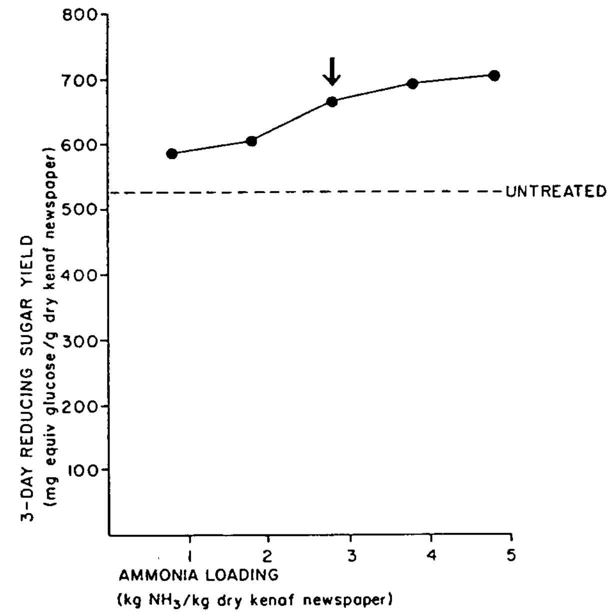

Fig. 8. Kenaf newspaper experiment 1: Effect of ammonia loading on sugar yield.

Figure 8 shows that increased ammonia loading makes kenaf newspaper steadily more digestible, whereas softwood newspaper digestibility declined slightly at high loadings. An ammonia loading of 2.8 kg NH_3/kg dry kenaf newspaper was selected, since it gives a good response and is less expensive to recover than the higher loadings.

Figure 9 shows that a 10-min reaction time at temperature is adequate, just as with softwood newspaper. Figure 10 indicates that, unlike some materials that are more digestible with 0.15–0.25 kg H_2O/kg dry biomass (*16,17*), kenaf newspaper is more digestible at very low water loadings. A loading of 0.08 kg H_2O/kg dry kenaf newspaper was selected, since this is the water level in air-dry material.

Figure 11 shows that temperature has a mild effect on reactivity with a slightly elevated plateau at 65–75°C. This temperature range is consistent with what was found for kenaf core in previous studies (*17*). The lower temperature (65°C) was selected to reduce pressure vessel costs.

AFEX increased the digestibility of raw kenaf newspaper from 525 to 750 mg Eq glucose/g dry kenaf newspaper, a 43% increase. Kenaf newspaper is substantially more digestible than softwood newspaper and requires no oxidative lignin cleavage.

MSW Components

The normalized sugar yields for MSW components were very similar to those for softwood newspaper shown in Fig. 1; 60% of the sugars were

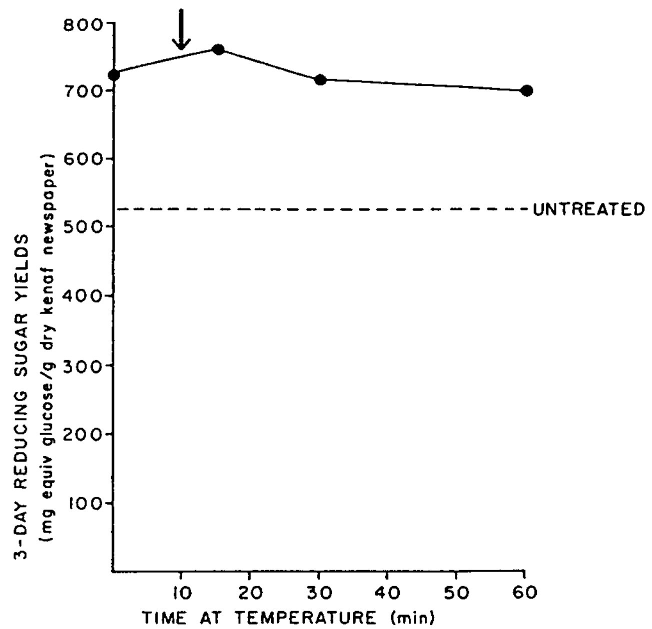

Fig. 9. Kenaf newspaper experiment 2: Effect of treatment time on sugar yield. (Note: Time-to-temperature is ca. 25 min.)

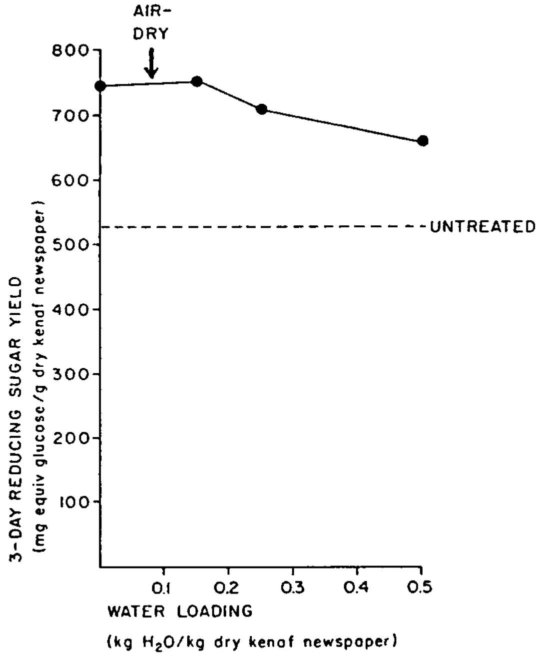

Fig. 10. Kenaf newspaper experiment 3: Effect of water loading on sugar yield.

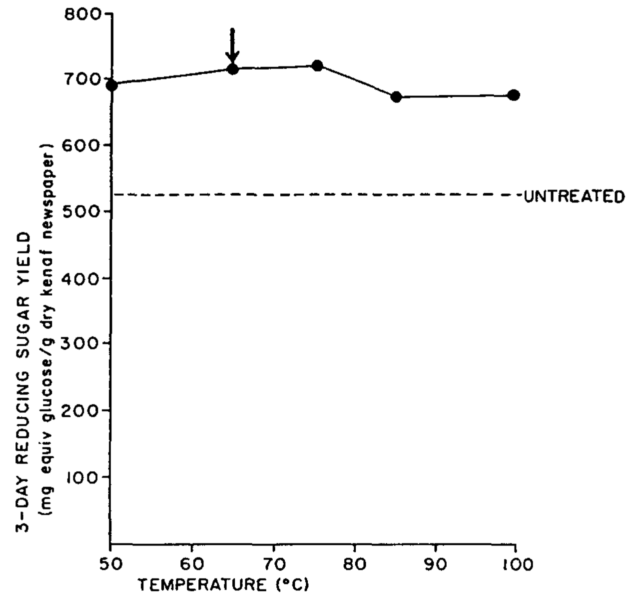

Fig. 11. Kenaf newspaper experiment 4: Effect of temperature on sugar yield.

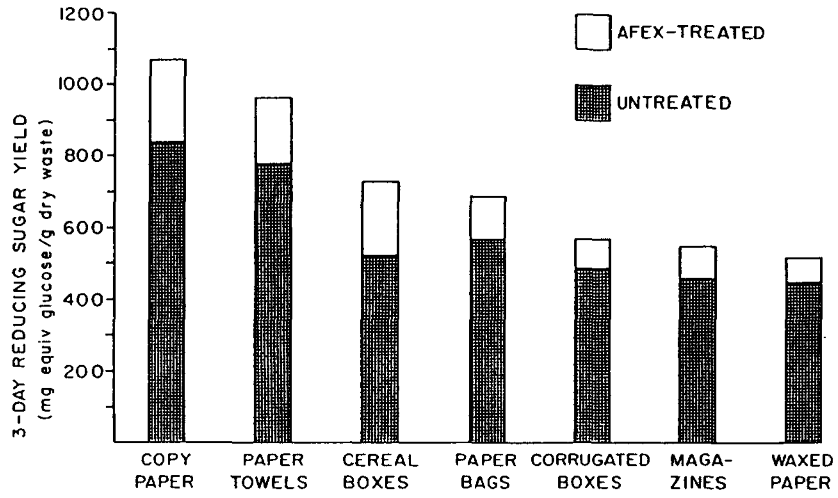

Fig. 12. Sugar yields from seven common MSW components.

released in the first 6 h and about 85% in 24 h. Table 2 shows the AFEX conditions used in this study.

Figure 12 shows the digestibility of seven common MSW components. AFEX-treated copy paper was completely converted to sugar (1070 mg Eq glucose/g dry paper) since it contains no lignin. AFEX increased its digestibility by 28%. The paper towels were brown hand towels from an institutional lavatory. AFEX-treated paper towels were almost completely converted to sugars (965 mg Eq glucose/g dry paper) with AFEX increasing the digestibility by 24%. AFEX-treated cereal boxes produced 726 mg Eq glucose/g dry paper and had the largest response to AFEX (40% increase). AFEX-treated paper grocery bags had high sugar yields (686 mg Eq glucose/g dry paper) with a 22% increase in digestibility from AFEX. AFEX-treated corrugated cardboard boxes gave moderate sugar yields (569 mg Eq glucose/g dry paper). AFEX-treated magazine paper yielded 544 mg Eq glucose/g dry paper, a good yield since magazine paper contains significant amounts of nondigestible groundwood and kaolin clay, a glossy coating that accounts for about 30–40% of the weight. AFEX-treated waxed paper yielded 514 mg Eq glucose/g dry paper, a good yield considering much of it is waxy coating. AFEX increased the digestibility of corrugated cardboard boxes, magazines, and waxed paper by 15–19%.

Using the increased digestibilities described above, AFEX should increase the digestibility of mixed MSW paper components (of the same composition as in Table 1) from 505 to 639 mg Eq glucose/g dry mixed MSW, a 25.5% increase. The expected hydrolysis residues from 1 t of dry MSW paper should decrease from 545 to 424 kg by using AFEX pretreatment, a 22% decrease. The dominant components of the residue are unreacted corrugated boxes and newspaper.

Mixed MSW

EG&G Idaho has developed a process for separating heavy components out of MSW. Table 4 compares the composition of this machine-sorted MSW to "typical" MSW. The yard waste, glass, and metal contents have been substantially reduced in the machine-sorted MSW. The machine-sorted, mixed MSW was subjected to the AFEX conditions listed in Table 2.

The biodegradable fraction of the machine-sorted MSW was AFEX treated and enzymatically hydrolyzed. Figure 13 shows the sugar yields at various enzyme loadings. These data show that little improvement in sugar yields can be expected above 5 IU/g, whereas substantial drops occur below 5 IU/g. Thus, the 5 IU/g loading used throughout these studies is very reasonable.

This particular mix of raw MSW paper components had a digestibility of 588 mg Eq glucose/g dry mixed MSW paper, which is higher than the 505 mg Eq glucose/g dry mixed MSW paper estimated from the study of individual MSW components. This is understandable, since the machine-sorted MSW was low in cardboard and newspaper, two very recalcitrant components. Since this particular mix was fairly digestible, AFEX treatment only increased the digestibility by 7% to 629 mg Eq glucose/g dry mixed MSW paper. (Note: In another sample of mixed MSW from EG&G

Table 4
Comparison of Machine-Sorted and Unsorted Air-Dried MSW

	Machine-sorted, %	Unsorted, %
Biodegradable		
Cardboard	11.8	11.1
Newspaper	6.3	7.4
Magazine, mail, wrapping paper	39.1	18.6 (combined)
Miscellaneous (tar, paper, gum, cigaret butts)	14.6	
*Yard waste	1.6	17.9
Textile	5.1	2.5
Subtotal	78.5	57.5
Refractory		
Plastic	12.0	7.2
*Glass	3.0	9.7
*Metal	2.0	9.6
Dirt	4.6	4.0
Subtotal	21.6	30.5

*Substantially reduced.

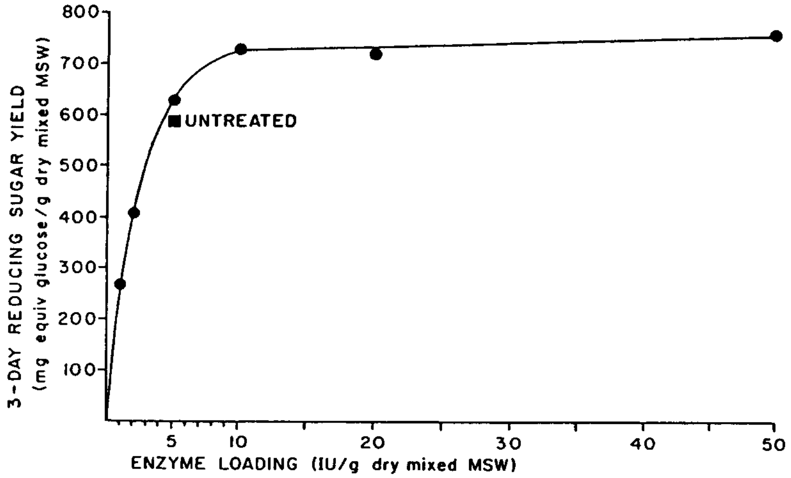

Fig. 13. Sugar yields from AFEX-treated mixed MSW at various enzyme loadings.

Idaho, the digestibility increased from 486 to 634 mg Eq glucose/g dry mixed MSW, a 30% improvement.) Assuming ethanol yields are 85% of theoretical, the sugars resulting from AFEX-treated mixed MSW would result in 165 L ethanol/wet t raw MSW or 73% of the estimated ethanol potential of raw MSW.

CONCLUSIONS

Most MSW components have been substantially delignified and pretreated in pulping operations. Therefore, MSW is much more digestible than agricultural residues, which have been previously studied (*14–17*). Whereas AFEX increases agricultural residue digestibilities by 100–1000%, it increases MSW component digestibility by only 15–50%. Since so much of the MSW readily hydrolyzes without pretreatment, only the unreacted residues from saccharification/fermentation should be AFEX treated, since much of the raw material is already digestible.

Most of the unreacted residues consist of cardboard and newspaper. These components are easily recycled, since they are readily identified and separated from mixed MSW. However, paper can only be recycled a limited number of times before the fibers become too short and must be purged. If the paper has significant amounts of ground softwood, it will be very difficult to digest. Although ground softwood can be rendered digestible by bleaching lignin prior to AFEX treatment, this is uneconomical. It would be more cost effective to use readily digestible paper, such as those derived from kenaf instead of softwoods. This may require legislation, since ground softwood dominates the US newsprint market.

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