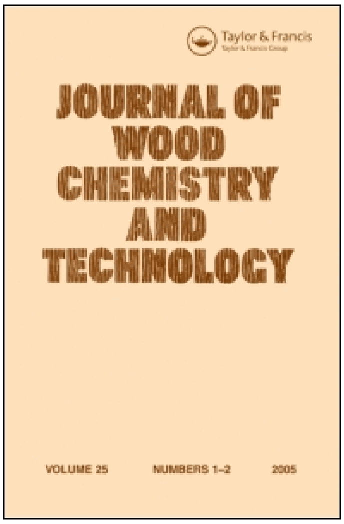

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EFFECT OF XYLANASE AND DOSAGE ON THE REFINING PROPERTIES OF UNBLEACHED SOFTWOOD KRAFT PULP

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ABSTRACT

Two xylanase preparations were compared at different doses for their abilities to enhance the refining properties of a never–dried, unbleached kraft pulp derived from radiata pine. These enzymes varied in their ability to solubilise xylan from pulp, and the selective removal of pulp xylan did not reduce intrinsic fibre strength. At low levels of xylan solubilisation, there were increases in sheet densification after PFI refining without change in tensile and tear strength at a given sheet density, indicating lower pulp refining requirements. At high levels of xylan solubilisation, the level of sheet densification was greater but there was a small decrease in tensile strength at a given sheet density and an increase in tear strength. One of the xylanases yielded a selective increase in tear strength at a given tensile strength.

INTRODUCTION

Improvements to the refining and papermaking properties of wood pulp would increase its value and versatility for use in the manufacture of a variety of end–products. A number of enzymes have been screened for this purpose on kraft[1–10] and mechanical pulps,[4,11–13] with greater modification of fibre properties achieved in the former. Although the treatment of kraft pulp with cellulolytic enzymes could enhance the tensile strength of the resultant sheets in certain cases,[2,9] specific conditions are required because these enzymes tend to degrade intrinsic fibre strength.[2–4,6,8,9] In contrast, the lignin–modifying laccase–mediator system preserves fibre strength but has limited effects on pulp properties.[10] Another enzyme that may be able to enhance sheet and fibre properties without loss in fibre strength is xylanase.[1,5–7]

To–date most of the work on xylanase, an enzyme that selectively hydrolyses xylan, in the pulp and paper sector has concerned the bleaching of kraft pulp.[14] Before pure xylanases were readily available, French researchers suggested that xylanase could improve pulp refining but their results were not completely reliable because cellulolytic action was indicated by a substantial loss in wet zero span breaking length.[1] Research in our laboratories indicated that treatment with very high doses of pure xylanase does not damage fibre strength and increases tear strength in unbleached and bleached kraft pulps derived from radiata pine.[6] Two other reports also suggested that xylanase treatment improves strength properties in unbleached kraft pulp[5,7] but one report suggested no improvement in bleached kraft pulp.[8] The present work provides a more thorough analysis of the effects that xylanase treatment has on the sheet properties of unbleached kraft pulp, particular when relatively low enzyme doses are used. Nearly all of the discussion is focused on the apparent density, tear index and tensile index of handsheets because these properties have been found to be the most important for the comparison of softwood kraft pulp.[15]

EXPERIMENTAL

Pulp

A medium coarseness (0.226 mg/m) kraft pulp derived from radiata pine, with kappa no. of 22.5, was obtained from Tasman Pulp and Paper Co. (Kawerau, New Zealand) in early 1995. It was produced by a conventional, continuous kraft cooking process, and no laboratory washing of this never–dried unbleached pulp was carried out before the experiments. The composition of the pulp was 0.8% ash, 3.2% Klason lignin, 0.5% acid soluble lignin, and 0.6% arabinosyl, 0.4% galactosyl, 78.6% glucosyl, 6.8% xylosyl and 5.2% mannosyl residues.

Enzymes

Xylanase E, purified from the fungus *Trichoderma reesei*, was provided by Genencor International (USA),[16] while the commercial Pulpzyme HC xylanase was provided by Novo Nordisk (Denmark). Xylanase activity was assayed using 0.9% birchwood xylan (Sigma, USA) as the substrate and a dinitrosalicylic acid reagent to measure the reducing ends generated.[17] Contaminating activities were evaluated using the same method except that 1.8% carboxymethyl-cellulose and 0.45% locust bean gum galactomannan and (Sigma) were used as substrates.

Enzyme Treatments

Enzyme treatments of pulp (5% consistency, 50°C, 2 h) were carried out in plastic bags after the pH of the slurry was stabilise at the desired level by adding H_2SO_4 (pH 5 for Xylanase E; pH 7 for Pulpzyme HC). After the enzyme treatment, the pulp sample was adjusted to pH 12 with NaOH, held at room temperature for 15 min and then well washed to neutral pH. The control pulp was treated in parallel in the same manner except that no enzyme was added.

Characterisation of Pulp and Treatment Filtrate

Handsheets were prepared from the pulp samples and physical testing was performed in accordance with APPITA standard procedures. The load applied during the refining of the pulp (10% consistency) in a PFI mill was 3.4 N/mm. All data from physical testing are reported on oven–dry bases.

TABLE 1
The activity of the Xylanase E and Pulpzyme HC, and the wet zero span tensile index (WZTI) of the pulp after xylanase treatment.

	Conditions		Activity (nkat/mg enzyme)			WZTI (N•m/g)[a]	
Xylanase	temp.	pH[b]	xylan	mannan	cellulose	control	treated
E	50°C	5	216	0	<0.26	143–148	138–146
HC	50°C	7	149	0	<0.14	137–146	135–144

[a] range of values observed in control and treated samples (all three xylanase doses examined) after the four levels of PFI refining.
[b] the 50 mM buffers for the assays at pH 5 and 7 were based on citrate and phosphate, respectively.

The carbohydrate in the treatment filtrate, expressed in the anhydro-form, was determined by high performance anion–exchange chromatography on a CarboPac PA-1 column (Dionex, USA) after secondary acid hydrolysis (4% H_2SO_4, 121°C, 103 kPa, 1 h). Net solubilisation of carbohydrate, determined by subtracting the carbohydrates in the control filtrates and the enzyme preparation, was expressed as a portion of the carbohydrate in the pulp.

RESULTS AND DISCUSSION

Both Xylanase E and Pulpzyme HC were highly purified xylanase preparations, with an activity ratio for cellulase:xylanase of about 0.001 (Table 1). Their action on pulp was highly selective for arabinoxylan, with > 98% of the solubilised carbohydrate composed of arabinosyl and xylosyl residues. The absence of cellulose degradation was confirmed by the retention of wet zero span tensile strength of the pulp fibres (Table 1). Based on equivalent activity on isolated birchwood xylan, xylanase E was more aggressive than Pulpzyme HC for the solubilisation of pulp xylan (Figure 1). Even when the dose of Pulpzyme HC was increased to 3.6 μkat/g pulp in a subsequent experiment, xylan solubilisation by this enzyme remained around 10%.

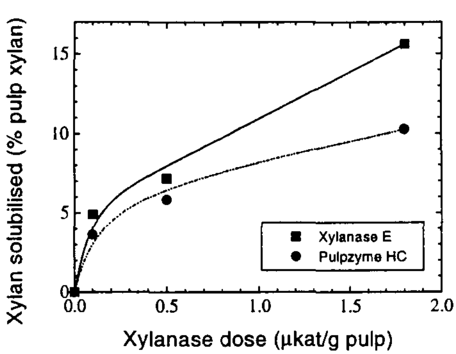

FIGURE 1. The solubilisation of xylan from kraft pulp during xylanase treatment.

Xylanase E was also more aggressive for increasing the apparent density of handsheets because it, but not Pulpzyme HC, provided maximal effects at a low dose (Figure 2). Similar patterns were reflected by the decrease in the freeness of pulp (data not shown), which is a measure of pulp drainability that is another indicator of pulp refining. At maximal levels of effects, the PFI refining revolutions required to achieve a sheet density of 700 kg/cm^3 was reduced by about 30%. Since no loss in fibre length was detected after xylanase treatment, the higher consolidation of the fibre network could be due to increases in fibre collapse or flexibility, decreases in fibre curl or changes to surface chemistry.

As would be expected, the tighter packing of the fibre network yielded higher tensile strength for given levels of refining. However, there was a 8% decrease in tensile strength at a given sheet density when the maximal increase in sheet density was achieved with all three doses of Xylanase E and the high dose of Pulpzyme HC (Figure 3). These results suggested that the intensity of interfibre bonding was decreased when high amounts of xylan were removed from pulp. With few exceptions, the development of the other tensile properties, such as tensile stiffness and T.E.A. indices, after xylanase treatment was in agreement with that of tensile index.

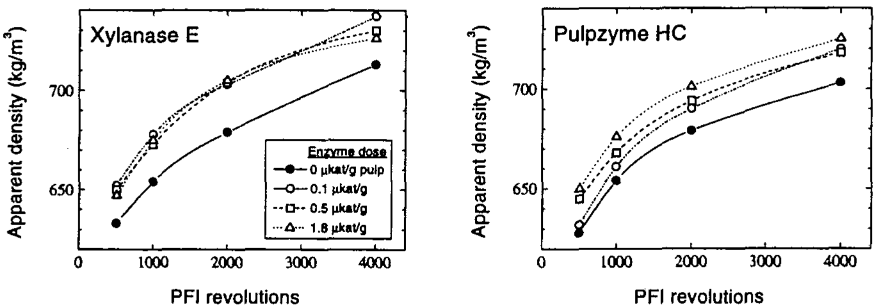

FIGURE 2. The development of sheet density with PFI refining after pulp treatment with xylanase.

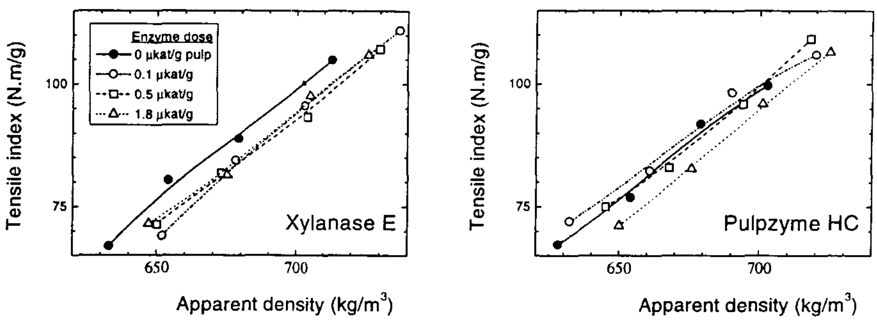

FIGURE 3. The tensile strength of kraft pulp after treatment with xylanase.

Xylanase treatment generally decreased the tear strength of the pulp at a given level of PFI refining. For the two lower xylanase doses examined, there was apparently no change to the relationship between tear strength and sheet density (Figure 4). For the high xylanase dose, there was an increase in tear strength at a given sheet density for sheet density below 700 kg/m^3. Only with the high dose of Xylanase E was there an increase in tear index at a given tensile index (Figure 5). This effect could not be achieved with Pulpzyme HC by increasing its dose to

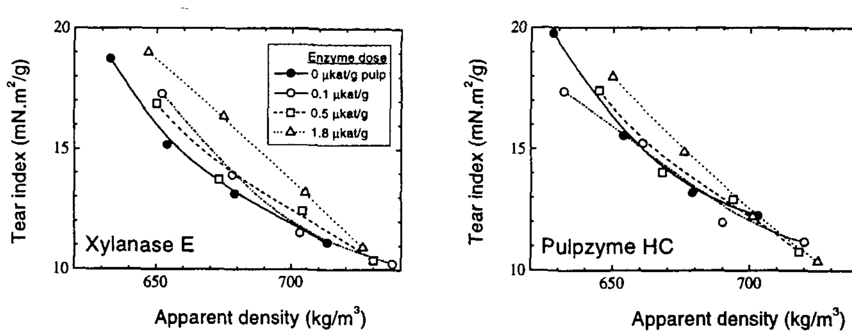

FIGURE 4. The tear strength of kraft pulp after treatment with xylanase.

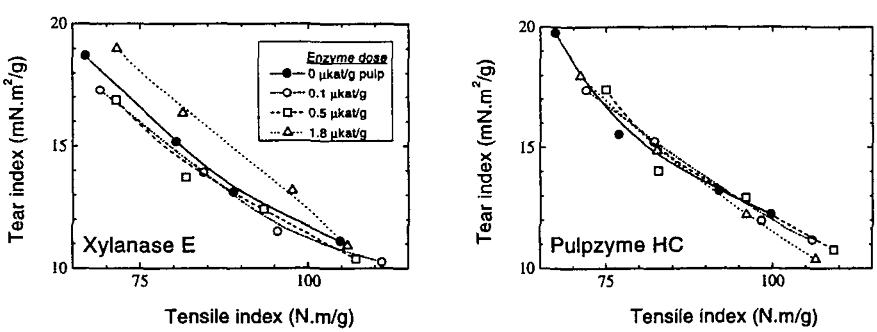

FIGURE 5. The tear–tensile relationship of kraft pulp after treatment with xylanase.

3.6 µkat/g pulp. Another commercial xylanase, Irgazyme 40 (Genencor), was also evaluated but the low levels of contaminating cellulase present (cellulase:xylanase ratio of ~0.005) was enough to decrease pulp strength at high enzyme doses.

Our results are in agreement with the general consensus that pulp hemicelluloses play important roles in pulp strength. The earlier evidence was gathered using pulp samples prepared by modifying pulping conditions,[18,19] by extracting with solvents such as alkaline solutions,[20,21] or by adding certain hemicellulose fractions or related polysaccharides to pulp.[22–24] For example, Mobarak *et al.*[23] found that hemicellulose added to extracted pulps increased strength properties to levels beyond those provided by the hemicellulose *in situ*. An advantage of using xylanase to study the roles of hemicelluloses is that xylan can be removed more selectively, leaving the other pulp components more or less unchanged. Although a small amount of lignin appears to be removed from pulp by xylanase treatment, the reported decrease in the kappa number of < 1 unit could be due to the removal of xylan carrying hexenuronic acid side–groups.[25]

The previously published data on the effects of enzymatic removal of pulp xylan on pulp properties have been sparse because most of the work involved pulp bleaching, xylanases with contaminating activities or comparisons using single refining levels.[1,5,7,8] The results of our detailed study indicate that a range of effects can be achieved in unbleached softwood kraft pulp. A selective increase in tear strength at a given tensile strength could be achieved when 15% of the pulp xylan was selectively removed using enzyme. Increases in sheet density after PFI refining was achieved in all cases, with the maximum effect obtained after the removal of about 5% and associated with a small decrease in tensile strength at a given sheet density. When less than 5% of pulp xylan was removed with xylanase, there was also an increase in pulp refinability without any changes to pulp quality. Since the amounts of commercial enzyme required to achieve this effect are similar to those currently recommended for bleaching applications, the economic feasibility of using xylanase to enhance pulp refining should be evaluated. It would be important to determine whether these effects could be achieved in other types of unbleached and bleached kraft pulp, in previously dried pulps, and in industrial scale refiners.

CONCLUSIONS

The xylan in an unbleached, never–dried, medium coarseness kraft pulp derived from radiata pine could be selectively solubilised by xylanase treatment.

When 3–5% of the xylan was selectively removed, the pulp showed: an increase in sheet density after PFI refining; no change in tear and tensile strength at a given sheet density; no change in the tear–tensile relationship of the pulp. As greater amounts of xylan were removed, the pulp showed: a larger increase in sheet density; a small decrease in tensile strength at a given sheet density; a small increase in tear strength at a given sheet density; no change in the tear–tensile relationship. When 15% of the xylan was removed, there was no further increase in sheet density or decrease in tensile strength at a given sheet density, but there was a selective increase in tear strength at given tensile strength values. The effects, achieved without any loss in fibre strength, could be solely attributed to the action of xylanase.

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