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UNIVERSIDADE DA BEIRA INTERIOR  
C O V I L H Ã

# Effects of enzymatic treatment and refining on the properties of recycled pulp

Pala, H.<sup>1</sup>; Mota, M.<sup>1</sup>; Gama, F.M.<sup>1</sup>

## ABSTRACT

*The effect of refining and enzymatic treatment on secondary fibre properties is shown. The following treatment sequences were studied: (i) refining; (ii) refining + enzymatic treatment; (iii) enzymatic treatment + refining; (iv) enzymatic treatment. The effectiveness of the methods was evaluated by measuring physical and mechanical properties of pulp and paper (drainage rate, burst, tensile and tear indexes). The obtained data revealed that combined refining and enzymatic treatment can be considered as a valuable methodology to upgrade recycled pulps. The first is responsible for increasing burst and tensile resistance. The second, if developed under controlled conditions, allows better drainage, with only slight variation on paper mechanical properties.*

**Key words:** wastepaper, recycling, drainability, enzymes, refining

## INTRODUCTION

The use of waste paper in paper manufacturing has increased during the last years. Nevertheless, the incorporation of secondary fibre in production is low because of concerns about product quality. Recycled fibres are associated to significant limitations as they have low strength and high drainage resistance. As a matter of fact, the recovered fibre quality depends on the conditions imposed during the previous papermaking cycle: pulping, refining and drying. Strength losses are generally attributed to a loss in bonding potential, either in the strength of the inter-fibre bonds or in their number [1]. According to Ganapati et al. [1], some of the strength loss can be regained by making the recycled fibre more flexible, thereby increasing the surface area for bonding.

The main purpose of this work was to study the effects of both refining and enzymatic treatment on the physical properties of recycled pulps, as a possible strategy to improve the secondary fibre properties. Further work will follow, with the aim of analysing the fundamentals of both processes in terms of cellulose surface modification.

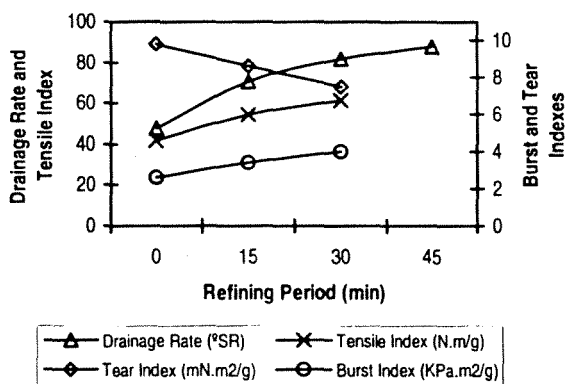
## RESULTS AND DISCUSSION

### *Evaluation of refining performance*

As shown in Figure 1, secondary pulp can recover some of its strength properties after refining (burst and tensile). Unfortunately, the process considerably reduces drainage, which negatively affects production capacity.

Considering previous reports, burst and tensile indexes improvement can be considered a result of increased fibrillation at the fibres surface, and consequent surface area enlargement, which enhances inter-fibre bonding [4]. These parameters usually increase to a maximum value and then decrease as a result of fibre breakage due to intensive refining.

Figure 1: Effect of refining on the physical properties of pulp and paper



In spite of the increased number of inter-fibre bonds, bounding strength is lower after refining. This fact can probably justify tear index reduction [1].

Drainage is essentially affected by the modification of fibre adsorptive capacities and by fines release. The fines created when secondary fibres are beaten, and consist largely of microfibrils that were strongly coupled to each other during the previous papermaking cycle. When liberated during refining, they start to behave as fillers, with a small effect on strength but a large effect on drainage properties [1].

**Evaluation of refining followed by enzymatic treatment performance**

The obtained results (Table I) revealed the effectiveness of the assayed enzyme preparation on increasing pulp drainage rate. This effect is generally attributed to the enzymes action on the surface of the fibres, which is responsible for peeling away cellulose fibrils with high affinity for water, and fines degradation [1].

Fibre fines and fibrils have a high specific surface area which contributes to water retention but very little to the hydrogen bonding potential of the fibre slurry. Thus, if their removal is selective, the pulp drains more easily and strength properties are not affected [1].

TABLE I: EFFECT OF DIFFERENT TREATMENT SEQUENCES ON PHYSICAL PROPERTIES OF PULP AND PAPER

Refining period (min)		0	15	30	45
<b>Refining</b>	<i>Drainability (°SR)</i>	47.5	70.5	81.8	87.8
	<i>Burst Index (Kpa.m²/g)</i>	2.6	3.4	4.0	--
	<i>Tensile Index (N.m/g)</i>	41.7	54.3	61.4	--
	<i>Tear Index (mN.m²/g)</i>	9.8	8.6	7.5	--
<b>Refining + Enzymatic treatment</b>	<i>Drainability (°SR)</i>	33.8	53.5	71.0	81.3
	<i>Burst Index (Kpa.m²/g)</i>	1.8	2.3	2.9	2.7
	<i>Tensile Index (N.m/g)</i>	33.3	42.8	48.5	51.5
	<i>Tear Index (mN.m²/g)</i>	7.1	6.4	5.4	4.1

Since cellulose degrading enzymes are being used, enzymatic reactions have to be carefully controlled so that selective removal is achieved. Data in Table I

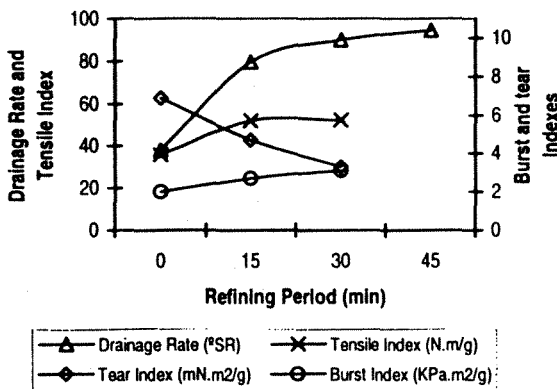
makes evidence of this fact. Under the applied conditions, extensive fibre attack could not be avoided, fibre length was probably altered and pulp strength was consequently reduced. Moreover, strength reduction was so strong that pulp improvement achieved through refining became worthless.

To validate this treatment sequence, enzymatic treatment should be performed under optimal conditions so that drainage can be increased, but cellulose degradation can be avoided and thereby strength losses.

### ***Evaluation of enzymatic treatment followed by refining performance***

Drainage reduction during refining is much faster for pulps that have been treated with enzymes than for plain pulps (Figure 1 versus Figure 2). The lower resistance to refining can be attributed to an attack on inter-fibre bounding areas through partial enzymatic hydrolysis or partial depolymerisation of carbohydrates on the fibres surface.[5]. This effect can be useful to the papermaking industry for energy saving during pulp upgrading processes.

**Figure 2: Effect of enzymatic treatment and refining on pulp and paper properties**



However, as the enzymatic treatment was performed under extreme conditions, considerable strength losses occurred in the pulp, that could not be recovered with beating. As a consequence, only after optimal enzymatic treatment application, energy savings could be conveniently evaluated.

### ***Evaluation of enzymatic treatment performance***

To avoid strength losses related to extensive enzymatic reactions, enzyme concentration was optimised. According to Table II, the enzymatic treatment of pulps with the lower enzymatic activity allows better drainage, with only slight variation on resistance.

As a matter of fact, the minimum drainage improvement took place at the maximum tested concentration (1.7 FPU/ g pulp o.d.). This fact can be explained as fibre surfaces are cleaned through removal of exposed fibrils, which can give rise more fines. If small particles are being produced faster than they are consumed, then drainage will decrease [1].

TABLE II: EFFECT OF ENZYME CONCENTRATION ON PHYSICAL PROPERTIES OF PULP AND PAPER

Enzymatic activity (g o.d. pulp)	Drainability (°SR)	Burst Index (Kpa.m <sup>2</sup> /g)	Tensile Index (N.m/g)	Tear Index (mN.m <sup>2</sup> /g)	Degradation (%)
Control	47.5	2.6	41.7	9.8	0.8
1.7 FPU	38	2.0	35.9	6.9	3.3
0.6 FPU	32.5	2.1	36.7	8.2	2.1
0.4 FPU	34	2.3	40.0	8.5	1.9

## CONCLUSIONS

The present work confirms that combined refining and enzymatic treatment can upgrade recycled pulp properties.

In spite of lowering drainage rate and tear index, refining can improve recycled pulp burst and tensile indexes. On the other hand, whenever reactions take place under optimised experimental conditions, enzymatic treatment can increase pulp drainage rate without affecting strength properties. It has also been demonstrated that drainage reduction during refining is much faster for pulps that have been previously treated with enzymes; this fact can probably allow considerable energy savings during the beating process.

However, complementary work should be developed so that upgrading processes can be better evaluated. Combined refining and enzymatic treatment should be performed, under optimal enzymatic reaction conditions. These experiments would allow more extensive conclusions about maximal fibre upgrading and possible energy savings during production.

## METHODS AND MATERIALS

To examine the effects of refining and enzymatic treatment on physical properties of recycled pulps, several experimental sequences were tested: (i) refining; (ii) refining + enzymatic treatment; (iii) enzymatic treatment + refining; (iv) enzymatic treatment. In order to conveniently estimate the enzymatic action, control assays (applying denatured enzyme) were made parallel to the enzymatic ones.

### Enzymatic Preparation

The enzymatic cocktail used was *Celluclast 1.5L*, a commercial preparation kindly supplied by Novo Nordisk. Its relevant activities are presented in Table III. Assay procedures used to determine enzymatic activities are the following:

TABLE III: ENZYMATIC ACTIVITIES

Activity	Activity (U/mL)
CMCase	56.5
FPase	26
Xylanase	680

### **Carboxymethylcellulase (CMCase)**

0,5 mL of the diluted enzyme was incubated in 0,5 mL of carboxymethylcellulose solution 1% (sodium citrate buffer, 0,05 M, pH = 5,0). The enzymatic reaction took place at 50°C, during 30 min. Released sugars were measured by DNS method, using glucose as standard.

### **Filter paper activity (FPase)**

0,5 mL of diluted enzyme was added to 1 mL of sodium citrate buffer containing 50 mg of Whatman n°1 filter paper. After incubating for 60 min at 50°C, released sugars were measured by DNS method, using glucose as standard.

### **Xylanase activity**

0,5 mL of diluted enzyme was incubated in 1,5 mL of oat spelt xylan solution 1% (sodium citrate buffer, 0,05 M, pH = 5,0). After incubating for 30 min at 65°C, released sugars were measured by DNS method, using glucose as standard.

### **Paper Furnish**

Paper furnish consisted on a secondary fibre sample kindly supplied by Portucel Viana.

### **Enzymatic Pulping**

After a 10 minutes fiberization in sodium citrate buffer 0,05 M, pH 5,0, the enzyme was added to the mixer and was allowed to react with the pulp during 30 minutes at 3% consistency. To finish up the trial, the enzyme was inactivated by boiling the pulp for 5 minutes.

### **Refining**

Pulp refining took place in a laboratory Valley beater (standard TAPPI procedure).

### **Samples dewatering**

Whenever samples had to be recovered, dewatering on a vacuum filter was applied.

### **Physical, Mechanical and Optical Properties determination**

After the handsheets preparation, properties of pulp and paper were determined, according to the usual standard TAPPI procedures. Parameters as drainage rate, burst, tensile strength, tear and brightness were measured.

### **ACKNOWLEDGEMENTS**

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