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Biotechnological Application of Enzymes for making Paper Pulp from Green Jute/Kenaf

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Final report

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CHEMICAL PULP

I. INTRODUCTION

Jute is a bast fibre obtained from the ribbons of the plant through a process known as retting. It is traditionally used for packaging, transportation and storage of agricultural and industrial products. Because of the severe competition appeared in the last decades with the use of synthetics, it has become imperative for the survival of the jute economy to make it competitive and widen the fields of its application in non traditional areas.

The growing demand for pulp and paper and the increasing world concern for denudation of forest have promoted, jute/kenaf, which is the annual crop, as a potential raw materials for pulp and paper.

The interest for biotechnological application in the manufacture of pulp and paper is a consequence of the possibilities offered by the biotechnologies in improving the products quality at reduced cost and also in protecting the environment from pollution hazard. Biotechnological process is an environment-friendly process and reduces the production cost of pulp and paper by way of reducing energy consumption in refining, lowering chemical charges in cooking and bleaching and improving pulp properties compared to mechanical and chemical wood pulp.

In the framework of this project a preliminary work was consisting in optimizing the soda anthraquinone cooking process and comparing conventional, ECF(elementary chlorine free) and TCF(totally chlorine free) bleaching sequences. The effect of a pretreatment with enzyme was evaluated by testing a Xylanase and a Laccase.

In a second time the work was orientated on the bleaching in conventional and ECF bleaching sequences of pulps issued from soda anthraquinone process with and without pretreatment of the raw material with *C. Subvermispora*. Finally the activities of commercial enzymes and a IJSG developed one were compared on the kraft pulp.

II. MATERIAL AND METHOD

Material

The raw material used for the cooking was jute chips cut at 2 cm long.
Two pulps were produced by the soda anthraquinone process from jute chips with and without *C.Subvermispora* pretreatment and two pulp from the kraft process as well.

Cookings were performed in a rotary digester equipped with 4 liters autoclaves immersed in a oil bath temperature regulated.
After cooking, the pulps were defibered in a laboratory pulper and screened in a Vewerk screener equipped with 0.15mm slots screen.

The bleaching stages were performed using different ways :

C Chlorination at 3.5% consistency was carried out in a glass Erlenmeyer vessel.

X and L Enzyme treatment at 5% consistency, D Chlorine dioxide, E Alkaline extraction, P Hydrogen peroxide, H Hypochlorite at 10% consistency were performed in polyethylene bags immersed in a temperature regulated water bath.

Z Ozone stage at 30% consistency was carried out in a spherical rotary glass vessel in which an ozone gas flow was applied on the pulp.

O Oxygen stage at 10% consistency was performed in the same equipment used for cooking.

The chemical charges, temperature and time are given in the result tables.

The pulp were refined in a PFI mill

The analysis were measured according to the following standards :
Kappa number (NF ISO 302)
Viscosity nt (ISO 5351 – 1)
Brightness (NF Q 50012)
COD (NF T 90-101)
BOD (NF EN 1899-2)
AOX (NF EN 1485)
Basic weight (NF EN ISO 536)
Tensile resistance (NF EN ISO 1924-2)
Burst resistance (NF Q 03 053)
Tear resistance (NF EN 21 974)
Opacity (ISO 2471)
Absorption and diffusion coefficient (K and S) (ISO 9416)

III. COOKING OPTIMIZATION

The aim of this part was to optimize the caustic soda and caustic soda + anthraquinone cooking with regard to the main pulping characteristics.

The cooking trials were carried out in a digester containing 6 autoclaves of 4 liters immersed in an oil bath regulated in temperature. Each autoclave contained 300g of o.d. raw material.

The varying parameters for this optimization were :

Sodium hydroxide charge : 16 to 25%
Cooking time : 120 to 240 min at 170°C
Ratio liquor/wood : 8 and 4
Anthraquinone charge : 0 and 0.1%

The fixed parameters were :

The cooking temperature : 170°C
The time to reach 170°C : 90min

The aim was to obtain a pulp in which the long fibres were cooked enough leading to a homogeneous result. The best results were the highest yields with the highest screened yield leading to a pulp of kappa number around 20. (Table n°1).
Cooking n°5 and n°14 have been chosen as the best results, figure n°1 illustrate such a choice(figure 1).

Figure n° 2 and 3 illustrate the beneficial effect of anthraquinone on the chemical consumption : the best compromise between yields, screened yields and kappa number were obtained with 20 and 25% sodium hydroxide, respectively for cooking 14 and 5.

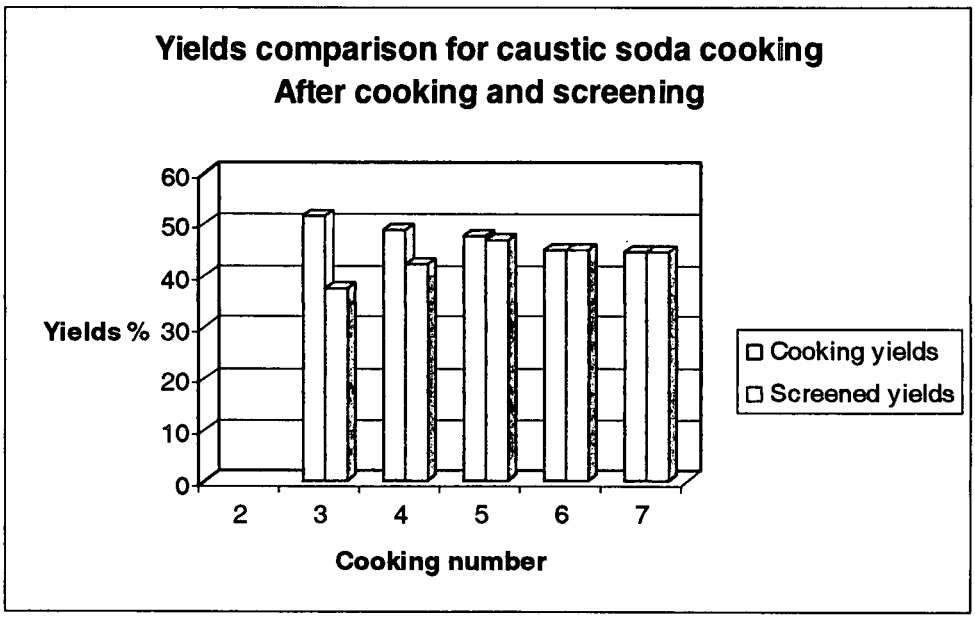
Table n°1

ALKALINE COOKING

REFERENCE 5 as **Pulp A**
REFERENCE 14 as **Pulp B**

Cooking conditions					Results					
REFERENCE	Cooking duration mn	Ratio liquor/jute	Caustic soda %	Anthraquinone %	Caustic soda consummed %	YIELD % unscreened	YIELD % screened	KAPPA number	Cellulose DP	Cellulose VISCOSITY mPa.s
1	120	8	16		14,9	58,3				
2	120	8	18		15,8	54,0				
3	120	8	20		16,4	51,6	37,6	56,0	1890	42,9
4	120	8	22		17,2	48,8	42,4	44,7	1740	34,4
5	120	4	25		19,8	45,2	45,0	19,9	1080	13,1
6	180	4	25		20,4	44,8	44,7	18,0		
7	240	4	25		21,1	43,1	42,9	15,7		
8	120	4	22		18,1	47,8	46,9	30,4	1420	21,6
9	120	4	25	0,1	19,6	45,5	45,3	15,5		
10	180	4	25	0,1	20,3	45,3	45,2	13,9		
11	240	4	25	0,1	20,8	43,6	43,4	13,3	770	8,2
12	120	4	23	0,1	18,9	46,8	46,7	16,4	1260	17,0
13	120	4	22	0,1	18,4	47,4	47,3	16,8	1320	18,7
14	120	4	20	0,1	17,7	48,3	48,1	18,3	1480	23,8
15	120	4	18	0,1	14,5	48,7	48,4	25,0	1730	34,0

Figure n°1



The screened yields indicated a few uncooked left in the pulp after cooking (0.2%). However the cellulose viscosity and the polymerization degree were rather low, especially for the selected caustic soda cook. A hardwood unbleached pulp at the same Kappa number level is 1800 to 2000.
The effect of anthraquinone was very significant in term of yield at the same kappa number level (3% more) (figure n° 3) and in term of cellulose viscosity or polymerization degree (400 units more)(figure n° 4).

Figure n°2

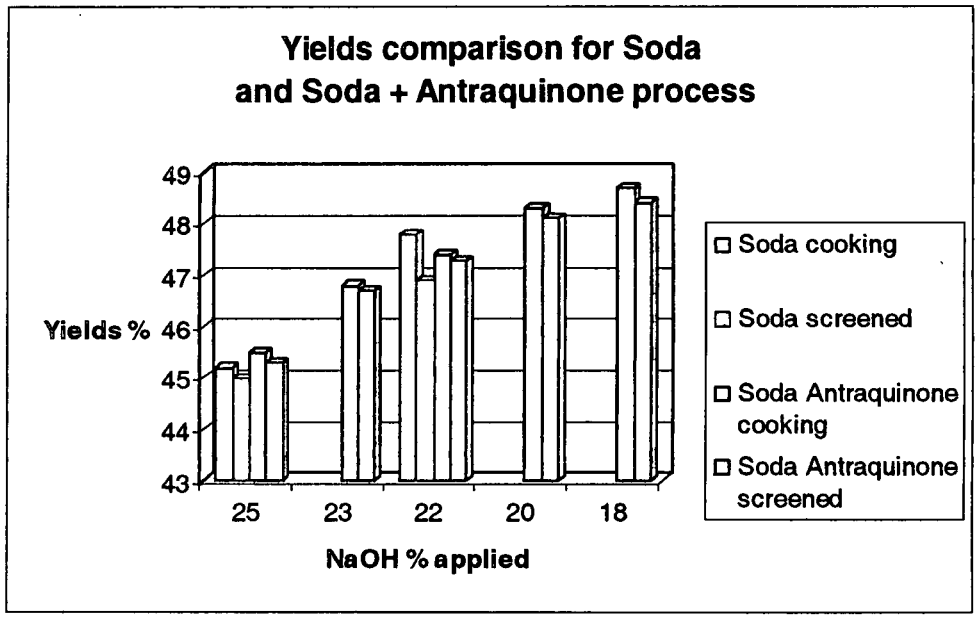


Figure n°3

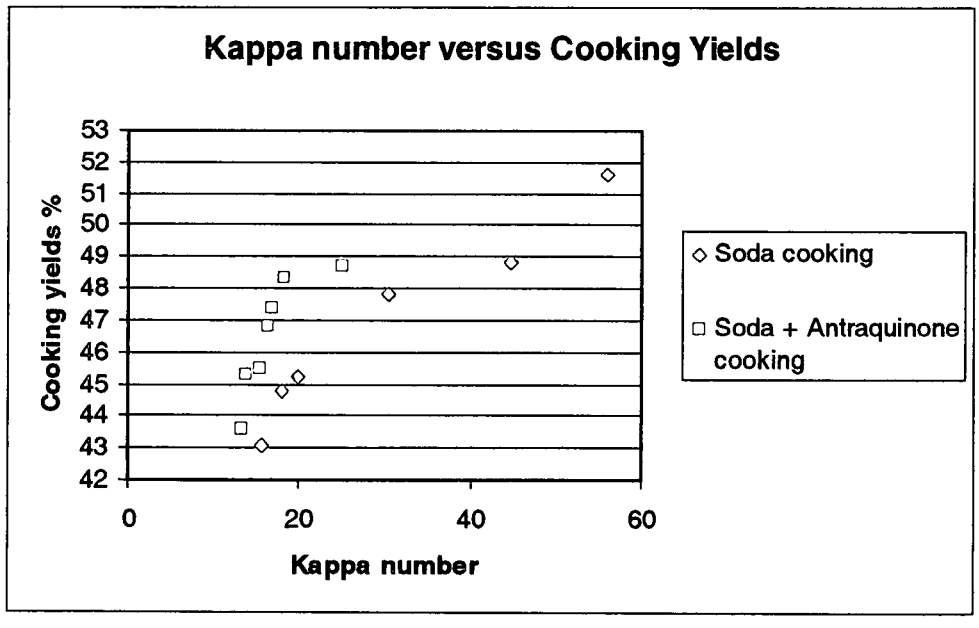
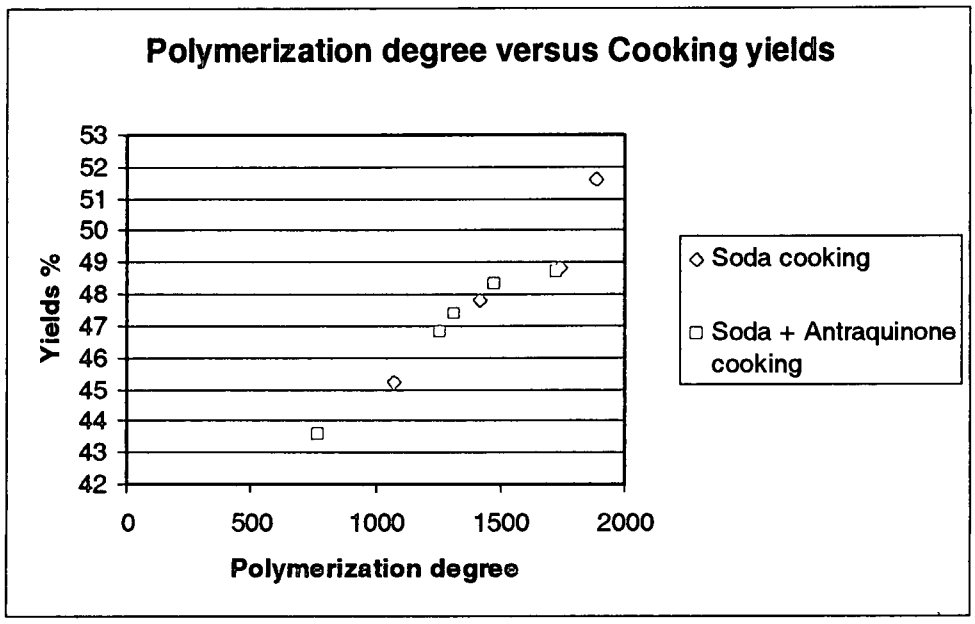


Figure n°4



Based on the selected pulping conditions, pulp A (cook n°5 with NaOH) and pulp B (cook n°13 with NaOH+AQ), were prepared to perform the bleaching tests with different sequences (CEH – DED – OP – OZP) and to test the mechanical properties of the pulpat different refining levels.

IV. BLEACHING

Comparison of Conventional, ECF and TCF bleaching sequences

On the prepared two pulp batches, conventional, ECF (Elementary chlorine free) and TCF (Totally chlorine free) bleaching sequences were tested to reach a final brightness of 80 % ISO.

The tested bleaching sequences were :

- CEH
- DED
- OP
- OZP

C for **C**hlorine, E for alkaline **E**xtraction, H for **H**ypochlorite, D for chlorine **D**ioxide, O for **O**xygen, P for hydrogen **P**eroxide, Z for o**Z**one.

a) CEH

The CEH sequence was carried out using conventional conditions, (table n°2).

The chlorine charge applied was based on the formula : Cl_2 % = 0.25*Kappa number.

The pulp A consumed more chlorine than pulp B. The first Hypochlorite stage showed a slightly higher consumption for pulp A but nevertheless only 50% of the chemical was consumed by both pulps. A time of 2 hours was appropriate with regard to the kinetic of the consumption (no more consumption after 1 hour)

Pulp A almost achieved the target. Pulp B looked to be more difficult to bleach.

In order to reach 80% ISO brightness a complementary Hypochlorite stage was carried out with 1% hypochlorite (as Chlorine). The chemical consumption was lower than for the H1 stage (20 – 30%).

The target was thenreached : The brightness were of 82.2 %ISO for pulp A and 80.9 %ISO forpulp B.

The bleaching yield was a bit lower for pulp B but the unbleached pulp B had a higher yield probably due to hemicelluloses saved during the cooking with anthraquinone. These hemicelluloses were dissolved during the bleaching.

The polymerization degree was very low. Hypochlorite stages are known to be damaging against cellulose, but this drop of viscosity was impressive.

b) DED

The DED sequence was carried out with usual conditions, chlorine dioxide was applied on the same basis as the chlorine step of the CEH sequence (table 3).

This sequence seemed to be well appropriate for this kind of pulp. The Chlorine dioxide was almost completely consumed in D1 and D2 stages. The brightness target was achieved : 81.3 and 81.7 %ISO respectively for pulp A and B, the Chlorine dioxide applied being respectively 2.9 and 2.74% as ClO_2.

It is important to note a slightly lower bleaching yield for pulp B; the comment was the same as for the CEH sequence.

With regard to the cellulose viscosity and the polymerization degree, this sequence did not have any effect on the cellulose degradation. The data was almost the same after bleaching for the unbleached pulp.

c) OP

As a TCF sequence, OP was tested (table n°4).

An Oxygen stage was carried out on the pulps A and B in order to decrease the Kappa number value.
The pulp B seemed to be less sensitive than the pulp A, the final Kappa number being 2 units higher.
The first P stage was applied with 5% Hydrogen peroxide, which was already an expensive sequence. All the peroxide was consumed and the results were very poor in term of brightness. A second P stage was tried with 3% more peroxide without any success. The peroxide consumption was probably due to cations such as iron or manganese in the pulp. A chelation stage could be safefull.
This bleaching sequence must be optimized, if it has some interest.

d) OZP

OZP was tested for being a sequence leading to usually proper results. The conditions and results are given in table n°5.
After the Oxygen stage previously described in the OP sequence a Z stage was carried out at high consistency with 1% Ozone applied. The Ozone consumption was almost 90% and the brightness target was almost achieved after the Z stage (78.6 and 76.1 %ISO). The Ozone seemed to be very reactive to ozone treatment and the charge should be reduced.
A P stage was carried out to reach the brightness aim. The 2% Hydrogen Peroxide charge was too high with regard to the low brightness gain to reach the target. All the peroxide was consumed and a high level of brightness was achieved (87.5 and 85.8 %ISO).
This TCF bleaching sequence seemed to be suitable to this kind of pulp. The chemical amounts had to be optimized especially for the Ozone charge.
Taking account of the cellulose viscosity and the polymerization degree, the fibres looked to be considerably degraded but these low values could be due to carbonyl groups generated on the cellulose leading to wrong viscosity data.

Table n°2

CEH

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

STAGES		Pulp A	Pulp B
C	Pulp consistency %	3.5	3.5
	Temperature °C	25	25
	Time min.	60	60
	Initial Kappa number	19.9	18.3
	Cl_2 %(0,25*K)	4.98	4.58
	Final pH	2	2.1
	Cl2 consumed %	4.32	3.7
E	Pulp consistency %	10	10
	Temperature °C	60	60
	Time min.	60	60
	NaOH %	2.49	2.29
	Final pH	11.8	11.7
H1	Pulp consistency %	10	10
	Temperature °C	50	50
	Time min.	120	120
	NaClO (as Cl_2 %)	2	2
	NaOH %	0.5	0.5
	Final pH	11.1	11.2
	NaClO consumed (as Cl_2 %)	1.05	0.93
	Brightness % ISO	79	77.2
H2	Pulp consistency %	10	10
	Temperature °C	50	50
	Time min.	120	120
	NaClO (as Cl_2 %)	1	1
	NaOH %	0.25	0.25
	Final pH	11.1	11.2
	NaClO consumed (as Cl_2 %)	0.29	0.23
	Brightness % ISO	82.2	80.9
	Bleaching yield %	94.4	93.7
	Polymerization degree	620	790
	Viscosity mPa.s	6.5	8.5

Table n°3

DED

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

STAGES		Pulp A	Pulp B
D1	Pulp consistency %	10	10
	Temperature °C	70	70
	Time min.	60	60
	Initial Kappa number	19.9	18.3
	ClO_2 %(0,25*K/2,63)	1.89	1.74
	Final pH	5.1	5.5
	ClO_2 consumed %	1.81	1.64
E	Pulp consistency %	10	10
	Temperature °C	60	60
	Time min.	60	60
	NaOH %	2.49	2.29
	Final pH	11.7	11.7
D2	Pulp consistency %	10	10
	Temperature °C	70	70
	Time min.	120	120
	ClO_2 %	1	1
	Final pH	3.1	3.3
	ClO_2 consumed %	0.89	0.91
	Brightness % ISO	81.3	81.7
	Bleaching yield %	95.4	94.3
	Polymerization degree	1060	1480
	Viscosity mPa.s	12.8	23.8

Table n°4

OP

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

STAGES		Pulp A	Pulp B
O	Pulp consistency %	10	10
	Temperature °C	100	100
	Time min.	60	60
	Initial Kappa number	19.9	18.3
	O_2 pressure bars	5	5
	NaOH %	3	3
	$MgSO_4,7H_2O$ %	0.3	0.3
	Final pH	11.5	11.7
	KAPPA number	7.5	9.6
P1	Pulp consistency %	10	10
	Temperature °C	80	80
	Time min.	120	120
	H_2O_2 %	5	5
	NaOH %	3	3
	DTPA %	0.5	0.5
	Final pH	11.4	11.5
	H_2O_2 consumed %	5	5
	Brightness % ISO	67.1	66.1
P2	Pulp consistency %	10	10
	Temperature °C	80	80
	Time min.	120	120
	H_2O_2 %	3	3
	NaOH %	2	2
	DTPA %	0.5	0.5
	Final pH	11.4	11.5
	H_2O_2 consumed %	3	3
	Brightness % ISO	71.5	69.9
	Bleaching yield %	95.9	95.3
	Polymerization degree	790	1130
	Viscosity mPa.s	8.5	14.2

Table n° 5

OZP

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

STAGES		Pulp A	Pulp B
O	Pulp consistency %	10	10
	Temperature °C	100	100
	Time min.	60	60
	Initial Kappa number	19.9	18.3
	O_2 pressure bars	5	5
	NaOH %	3	3
	$MgSO_4,7H_2O$ %	0.3	0.3
	Final pH	11.5	11.7
	KAPPA number	7.5	9.6
Z	Pulp consistency % (HC)	30	30
	pH	3	3
	O_3 charge %	1	1
	Final pH	3.3	3.4
	O_3 consumed %	0.89	0.86
	KAPPA number	1.56	2.07
	Brightness % ISO	78.6	76.1
P	Pulp consistency %	10	10
	Temperature °C	80	80
	Time min.	120	120
	H2O2 %	2	2
	NaOH %	2	2
	DTPA %	0.5	0.5
	Final pH	10.7	10.3
	H_2O_2 consumed %	2	2
	Brightness % ISO	87.5	85.8
	Bleaching yield %	89.5	90.1
	Polymerization degree	290	480
	Viscosity mPa.s	3.8	5.2

Introduction of an enzymatic pretreatment

Based on the results obtained during a study of biotechnologies applied on wheat straw pulp, an enzymatic pretreatmenthave been tested on pulps A and B before bleaching
Two commercial enzymes been tested :
A Xylanase : Pulpzyme HC commercialized by NOVO (X)
A Laccase : *Pycnoporus cinnabarinus* developped by INRA (Institut National de la Recherche Agronomique) (L).
Some trials were carried out with Xylanase (X) and Laccase (L) successively (X L).
After every enzyme treatment, an alkaline extraction (E) was carried out. The conditions and effect of this pretreatment on the Kappa number and the viscosities are given in table 6, 7 and 8 respectively for XE, LE and XLE stages.

Table 6

ENZYMATIC PRETREATMENT - XE

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

STAGES		Pulp A	Pulp B
X	Pulp consistency %	5	5
	Temperature °C	50	50
	Duration minutes	120	120
	Initial Kappa number	19,9	18,3
	Initial polymerisation degree	1080	1480
	Xylanase u/g	1	1
	Buffer pH7 mMole/l	25	25
	Final pH	7,1	7,14
	KAPPA number	18	16,4
E	Pulp consistency %	10	10
	Temperature °C	70	70
	Duration minutes	90	90
	NaOH %	2,5	2,5
	Final pH	11,5	11,65
	KAPPA number	**13,6**	**13,1**
	DP	**1180**	**1610**
	Viscosity mPa.s	15,3	28,6

Table 7

ENZYMATIC PRETREATMENT - LE

Pulp A: Cooked with caustic soda
Pulp B: Cooked with caustic soda + Anthraquinone

STAGES		Pulp A	Pulp B
L	Pulp consistency %	5	5
	Temperature °C	50	50
	Duration minutes	240	240
	Initial Kappa number	19,9	18,3
	Initial polymerisation degree	1080	1480
	Laccase u/g	25	25
	Buffer pH5 mMole/l	25	25
	HBT %	3	3
	O2 pressure bars	1,5	1,5
	Final pH	5,48	5,13
	KAPPA number	17,4	15,3
E	Pulp consistency %	10	10
	Temperature °C	70	70
	Duration minutes	90	90
	NaOH %	2,5	2,5
	Final pH	12,4	11,7
	KAPPA number	**13,4**	**13,4**
	DP	**1130**	**1510**
	Viscosity mPa.s	14,2	24,8

Table 8

ENZYMATIC PRETREATMENT - XLE

Pulp A: Cooked with caustic soda
Pulp B: Cooked with caustic soda + Anthraquinone

STAGES		Pulp A	Pulp B
X	Pulp consistency %	5	5
	Temperature °C	50	50
	Duration minutes	120	120
	Initial Kappa number	19,9	18,3
	Initial polymerisation degree	1080	1480
	Xylanase u/g	1	1
	Buffer pH7 mMole/l	25	25
	Final pH	7,1	7,14
	KAPPA number	18	16,4
L	Pulp consistency %	5	5
	Temperature °C	50	50
	Duration minutes	240	240
	Laccase u/g	25	25
	Buffer pH5 mMole/l	25	25
	HBT %	3	3
	O2 pressure bars	1,5	1,5
	Final pH	5,16	4,83
	KAPPA number	15,2	13,6
E	Pulp consistency %	10	10
	Temperature °C	70	70
	Duration minutes	90	90
	NaOH %	2,5	2,5
	Final pH	12,2	11,69
	KAPPA number	**10,7**	**10,8**
	DP	**1070**	**1520**
	Viscosity mPa.s	13	25,2

After all the enzymatic treatment a significant drop of the Kappa number was observed (30 to 35% for the XE and LE treatment and almost 50% for XLE)without any damages on the cellulose, the cellulose viscosity being not affected. The effect of the enzymatic pretreatment has then been proved to be not only efficient but also selective towards the delignification.

The bleaching abilities of the 3 enzymatic pretreated pulps were then compared by testing them in a conventional CEH bleaching sequence. The bleaching conditions and the results are given in table9, 10 and 11 respectively for XE, LE and XLE treatment.

Table 9

CEH on XE pretreated pulps

Pulp A: Cooked with caustic soda
Pulp B: Cooked with caustic soda + Anthraquinone

STAGES		Pulp A	Pulp B
C	Pulp consistency %	3,5	3,5
	Temperature °C	25	25
	Duration minutes	60	60
	Initial Kappa number	13,6	13,1
	Cl2 %(0,25*K)	3,4	3,28
	Final pH	2,43	2,6
	Cl2 consumed %	2,91	2,6
E	Pulp consistency %	10	10
	Temperature °C	60	60
	Duration minutes	60	60
	NaOH %	1,7	1,64
	Final pH	12,05	11,42
H1	Pulp consistency %	10	10
	Temperature °C	50	50
	Duration minutes	120	120
	NaClO (as Cl2 %)	2	2
	NaOH %	0,5	0,5
	Final pH	11,28	11,05
	NaClO consumed (as Cl2 %)	1,16	0,98
	Brightness % ISO	**80,4**	**80,2**
	Bleaching yield %	**91**	**91,5**
	DP	**720**	**900**
	Viscosity mPa.s	7,6	10

Table 10

CEH on LE pretreated pulps

Pulp A: Cooked with caustic soda
Pulp B: Cooked with caustic soda + Anthraquinone

STAGES		Pulp A	Pulp B
C	Pulp consistency %	3,5	3,5
	Temperature °C	25	25
	Duration minutes	60	60
	Initial Kappa number	13,4	13,4
	Cl2 %(0,25*K)	3,35	3,28
	Final pH	2,12	2
	Cl2 consumed %	2,82	2,7
E	Pulp consistency %	10	10
	Temperature °C	60	60
	Duration minutes	60	60
	NaOH %	1,68	1,64
	Final pH	12,03	11,45
H1	Pulp consistency %	10	10
	Temperature °C	50	50
	Duration minutes	120	120
	NaClO (as Cl2 %)	2	2
	NaOH %	0,5	0,5
	Final pH	11,24	11
	NaClO consumed (as Cl2 %)	1,21	0,98
	Brightness % ISO	**79,9**	**79,9**
	Bleaching yield %	**93,2**	**92,3**
	DP	**690**	**880**
	Viscosity mPa.s	7,3	9,6

Table 11

CEH on XLE pretreated pulps

Pulp A: Cooked with caustic soda
Pulp B: Cooked with caustic soda + Anthraquinone

STAGES		Pulp A	Pulp B
C	Pulp consistency %	3,5	3,5
	Temperature °C	25	25
	Duration minutes	60	60
	Initial Kappa number	10,7	10,8
	Cl2 %(0,25*K)	2,68	2,7
	Final pH	2,21	2,03
	Cl2 consumed %	2,25	2,15
E	Pulp consistency %	10	10
	Temperature °C	60	60
	Duration minutes	60	60
	NaOH %	1,34	1,35
	Final pH	11,94	11,38
H1	Pulp consistency %	10	10
	Temperature °C	50	50
	Duration minutes	120	120
	NaClO (as Cl2 %)	2	2
	NaOH %	0,5	0,5
	Final pH	11,43	11,16
	NaClO consumed (as Cl2 %)	1,04	0,75
	Brightness % ISO	**81,7**	**81,9**
	Bleaching yield %	**91,1**	**89**
	DP	**670**	**930**
	Viscosity mPa.s	7	10,4

The use of less hypochlorite led to less cellulose degradations (the viscosity values were slightly higher than the reference pulps) and should have an incidence on the mechanical properties.
From the enzymatic pretreated pulps the 80%ISO brightness target was obtained easily in the 3 different cases. It is usefull to remind that the bleaching of the pulp without enzymatic pretreatment required a CEHH sequence to reach the target brightness
Moreover, the chlorine amount needed to reach the target was considerably decrease as shown in table n°17, which compares the chlorine amount applied and reacted for the bleaching of the non pretreated and of the enzymatic pretreated pulps.

Table n°12

	Chlorine applied %		Chlorine reacted %	
Pulp	A	B	A	B
Non pretreated	7.98	7.58	5.66	4.86
After XE	5.40	5.28	4.07	3.58
After LE	5.35	5.28	4.03	3.68
After XLE	4.68	4.70	3.29	2.90

This reduction of chlorine consumption due to enzymatic treatment will have a large incidence on the generated AOX level.
If we compare the 3 pretreatments in term of brightness, XE and LE gave similar results, whereas XLE which generated more delignification after enzymes treatment allowed to achieve a better value with less chlorine consumption.
The XLE pretreatment seemed to be the most interesting one.
The bleaching ability of the XLE pretreated pulp was then tested in ECF and TCF sequences

ECF : D E D bleaching sequence (table n°13).
TCF : Q P on pulp A and a comparison with O Q P on pulp B (table n°14).

Looking at the results obtained, the D E D sequence led to very good brightness levels : 82.5%ISO (A) and 83.0%ISO (B). The chlorine dioxide consumption of 5.18% used in these trials (as Chlorine) could be reduced to reach the final brightness of 80%ISO.
It is also to note that the viscosity measurement indicated that this sequence did not have any effect on the cellulose degradation(same values as the unbleached pulp)

For the TCF bleaching, because of short stock of XLE pretreated pulps, a Q P sequence was carried out on the A XLE pulp and a O Q P was tested on the B XLE pulp (these two trials are comparable in term of chemical consumption and brightness results).
The O Q P sequence achieved the brightness target on pulp B. If we assume that the 2 pulps have a quite similar behavior it looked necessary to perform an oxygen stage after the enzymatic treatment in order to improve the delignification because the QP sequence led to 69.6 %ISO brightness only.

It can be recalled that with the O P sequence carried out on the A and B pulp without enzymatic treatment, it was not possible to reach 80%ISO brightness. The brightness achievement with 5% Hydrogen peroxide was 66 – 67%ISO.
This demonstrates again the efficiency of an enzymatic pretreatment.
Concerning the effect of bleaching on the quality of the pulp, it seems that, compared to the CEH bleaching sequence, the ECF and TCF sequences led to less cellulose degradations, the cellulose viscosity was comparable to the unbleached pulp for the DED sequence and less degraded for the OQP pulp than for CEH pulps.

Table 13

DED on XLE pretreated pulps

Pulp A: Cooked with caustic soda
Pulp B: Cooked with caustic soda + Anthraquinone

STAGES		Pulp A	Pulp B
D1	Pulp consistency %	10	10
	Temperature °C	70	70
	Duration minutes	60	60
	Initial Kappa number	10,7	10,8
	ClO2 %(0,25*K/2,63)	1,02	1,03
	Final pH	3,1	2,93
	ClO2 consumed %	1,02	1,03
E	Pulp consistency %	10	10
	Temperature °C	60	60
	Duration minutes	60	60
	NaOH %	1,34	1,35
	Final pH	11,5	11,64
D2	Pulp consistency %	10	10
	Temperature °C	70	70
	Duration minutes	120	120
	ClO2 %	1	1
	Final pH	3	2,88
	ClO2 consumed %	0,95	0,87
	Brightness % ISO	**82,5**	**83**
	Bleaching yield %	**93,1**	**90,1**
	DP	**1120**	**1470**
	Viscosity mPa.s	14	23,4

Table 14

OQP on XLE pretreated pulps

Pulp A: Cooked with caustic soda
Pulp B: Cooked with caustic soda + Anthraquinone

STAGES		Pulp A	Pulp B
O	Pulp consistency %		10
	Temperature °C		100
	Duration minutes		60
	Initial Kappa number	10,7	10,8
	O2 pressure bars		5
	NaOH %		3
	MgSO4,7H2O %		0,3
	Final pH		11,66
	KAPPA number		6
Q	Pulp consistency %	10	10
	Temperature °C	90	90
	Duration minutes	60	60
	EDTA %	0,5	0,5
	Final pH	6,61	7,32
P	Pulp consistency %	10	10
	Temperature °C	80	80
	Duration minutes	120	120
	H2O2 %	5	5
	NaOH %	2	2
	Final pH	11,52	10,91
	H2O2 consumed %	4,84	2,03
	Brightness % ISO	**69,6**	**80**
	Bleaching yield %	**91,2**	**90**
	DP	**770**	**1170**
	Viscosity mPa.s	8,1	15

Figure n°5 and n°6 illustrate the viscosity and brightness results comparing the bleaching sequences with and without a XLE pretreatment.

Figure n°5

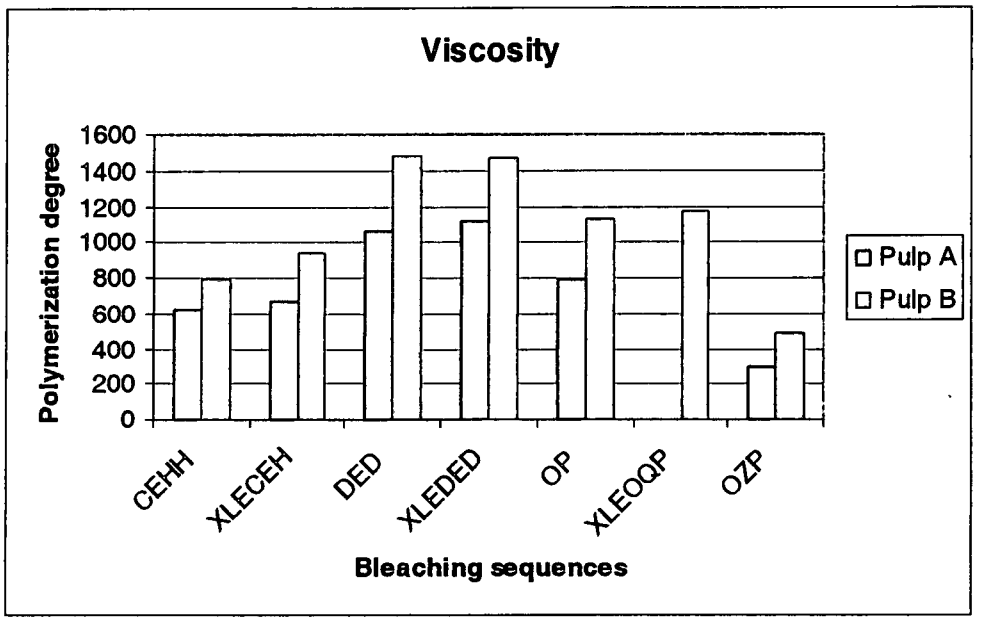


Figure n°6

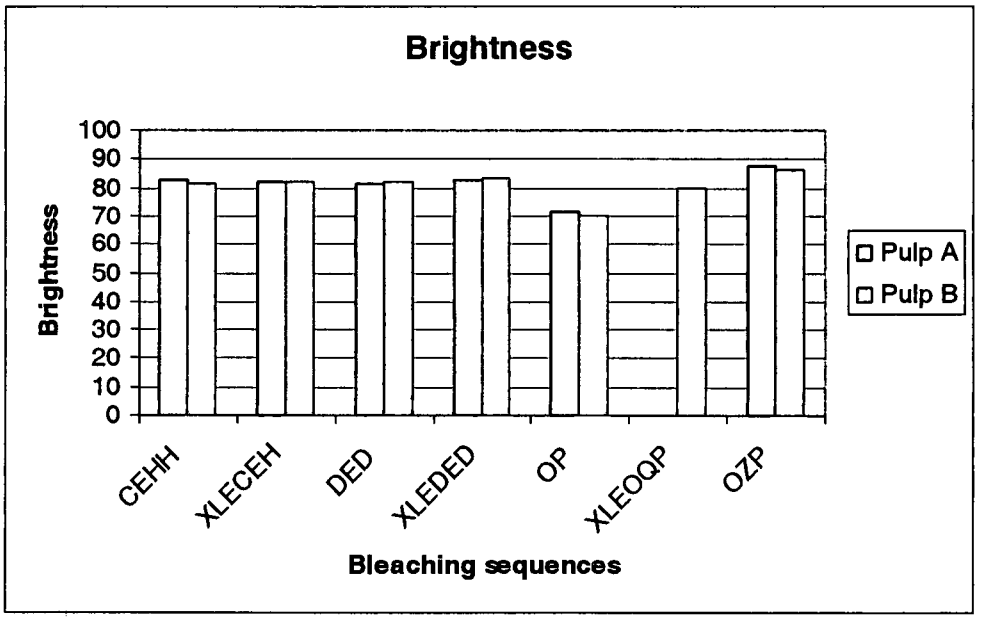


Figure n°5 shows that the enzymatic treatment do not have any effect on the pulp viscosity.

Figure n°6 shows that the 80%ISO brightness target is obtained in any cases except for a OP stage which is really improved by a enzymatic pretreatment.

Bleaching yields

In term of yield it was interesting to compare the total yields after bleaching with and without an enzymatic pretreatment for the two pulps (Table n°15).

Table n°15

Pulp	A		B	
Cooking Yield	45.0		48.1	
Bleach.sequence	Bleaching yield	Final pulp yield	Bleaching yield	Final pulp yield
CEHH	94.4	42.5	93.7	45.0
DED	95.4	42.9	94.3	45.4
OZP	89.5	40.3	90.1	43.3
XE CEH	91.0	40.9	91.5	44.0
LE CEH	93.2	41.9	92.3	44.4
XLE CEH	91.1	41.0	89.0	42.8
XLE DED	93.1	41.9	90.1	43.3
XLE QP	91.2	41.0		
XLE OQP			90.0	43.3

The bleaching yields obtained for the sequences involving enzymes are slightly lower than for the conventional bleaching except for the OZP sequence which needs an optimization of the Z charge. However , this drawback can be counter balanced on an industrial point of vue by the reduction of the chemical charges needed, especially for chlorine, as previously seen; an improvement in term of effluent toxicity is to be expected.

V. MECHANICAL PROPERTIES

To characterize the different pulps obtained with the different bleaching sequences, beating was carried out in a PFI mill at 3 freeness levels.
The results are presented respectively in tables 16, 17 and 18 for the CEH, DED and OZP bleaching sequences without enzymatic treatment.

Table n°16

CEH bleached pulps

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

Pulp	A				B			
PFI revolution number	0	500	1000	1900	0	300	900	1400
Drainage index,°SR	23	31	38	50	27	32	44	52
Bulk, cm3/g	1.6	1.47	1.4	1.32	1.47	1.39	1.32	1.27
Breaking length, km	5.28	6.52	6.72	6.93	6.13	7.4	8.03	7.49
Burst index, kPa,m²/g	2.81	3.33	3.28	3.67	3.81	3.85	4.09	4.27
Tear index, mNm²/g	7.88	7.31	6.81	6.38	8	7.31	7	6.55
Brightness %ISO	82.2				80.9			
Opacity fond papier %	86.1	83	82.3	80.8	82.5	81.7	79.8	78
Opacity fond blanc %	82.1	78.8	78.1	75.9	78.2	76.9	74.8	73
Scattering coefficient S m²/kg	42.8	36.9	34.4	32	35.4	32.8	30.2	27.8
Absorption coefficient K m²/kg	0.33	0.28	0.28	0.28	0.31	0.31	0.29	0.26

Table n°17

DED bleached pulps

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

Pulp	A				B			
PFI revolution number	0	500	2000	3700	0	500	2000	3200
Drainage index,°SR	23	28	37	53	24	29	39	49
Bulk, cm3/g	1.55	1.47	1.37	1.28	1.47	1.39	1.33	1.23
Breaking length, km	5.67	6.56	7.45	7.32	6	7.88	8.34	7.93
Burst index, kPa,m²/g	3.11	4.16	4.19	4.03	3.52	4.7	5	5.58
Tear index, mNm²/g	11.55	10.97	9.86	9.62	12.51	11.85	10.64	10.06
Brightness, %ISO	81.3				81.7			
Opacity fond papier, %	87.3	86	84.3	82.9	85.1	83	79.7	78.8
Opacity fond blanc, %	82.4	80.7	78.7	76.4	80.1	77.5	74.8	72.6
Scattering coefficient S, m²/kg	41.3	37.6	33.6	29.9	37.3	33.3	28.4	26.7
Absorption coefficient K, m²/kg	0.55	0.51	0.52	0.53	0.43	0.42	0.39	0.38

Table n°18

OZP bleached pulps

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

Pulp	A				B			
PFI revolution number	0	200	500	1000	0	200	600	950
Drainage index,°SR	25	31	38	52	25	30	40	50
Bulk, cm3/g	1.54	1.46	1.4	1.32	1.49	1.4	1.33	1.31
Breaking length, km	5.23	5.85	6.28	6.35	5.74	6.5	6.94	6.53
Burst index, kPa,m²/g	2.68	3.59	3.46	3.23	3.15	3.65	3.8	3.87
Tear index, mNm²/g	6.12	5.45	4.95	4.44	7.42	6.53	5.91	5.29
Brightness, %ISO	87.5				85.8			
Opacity fond papier, %	83.7	82.9	81.8	80.8	82.4	80.7	79.2	80.6
Opacity fond blanc, %	80	79	77.8	76.2	78.8	76.9	75.2	74
Scattering coefficient S, m²/kg	43.7	37.6	36	33.3	38.1	34.6	31.8	32.6
Absorption coefficient K, m²/kg	0.25	0.25	0.25	0.25	0.43	0.42	0.39	0.38

These tables showed that the mechanical properties seemed to be optimal around

40°SR freeness. In order to compare easily the diverse bleached pulps, the 40°SR refined trials were regrouped in table n°19 and illustrated in figure n°7

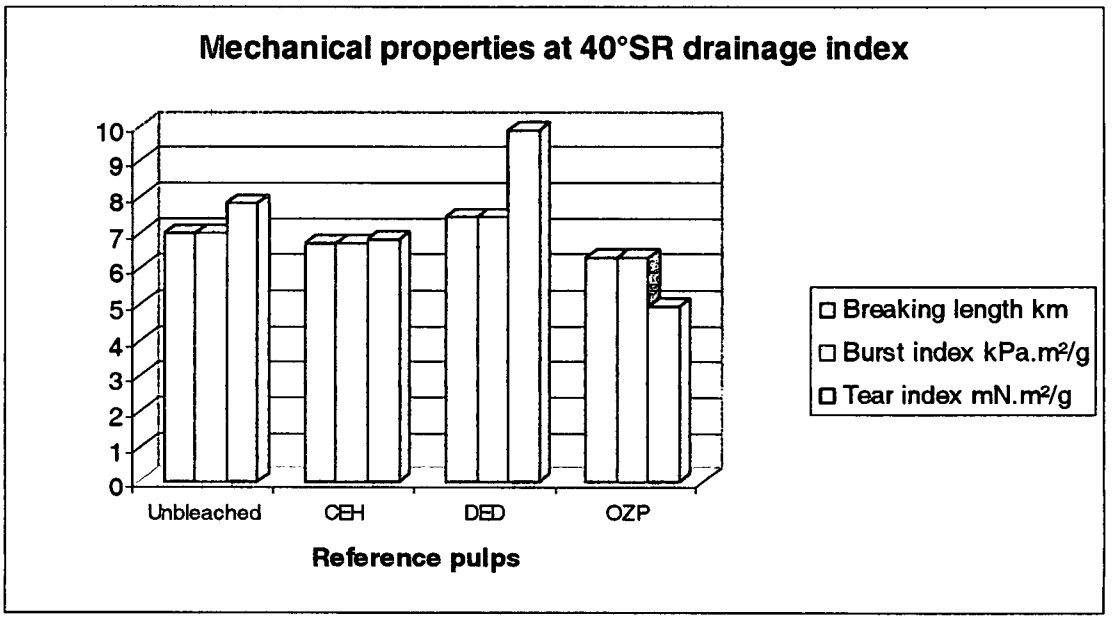
Table n°19

Mechanical properties at 40°SR

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

Pulp	A				B			
	Unbleached	CEH	DED	OZP	Unbleached	CEH	DED	OZP
PFI revolution number	3000	1000	2000	500	2300	900	2000	600
Drainage index,°SR	39	38	37	38	41	44	39	40
Bulk, cm3/g	1.4	1.4	1.37	1.4	1.32	1.32	1.33	1.33
Breaking length, km	6.99	6.72	7.45	6.28	7.96	8.03	8.34	6.94
Burst index, kPa,m²/g	3.85	3.28	4.19	3.46	4.76	4.09	5	3.8
Tear index, mNm²/g	7.8	6.81	9.86	4.95	7.91	7	10.64	5.91
Brightness, %ISO		82.2	81.3	87.5		80.9	81.7	85.8
Opacity fond papier, %		82.3	84.3	81.8		79.8	79.7	79.2
Opacity fond blanc, %		78.1	78.7	77.8		74.8	74.8	75.2
Scattering coefficient S, m²/kg		34.4	33.6	36		30.2	28.4	31.8
Absorption coefficient K, m²/kg		0.28	0.52	0.25		0.29	0.39	0.39

Figure n°7



The analysis of these results showed that the mechanical properties of the jute pulps looked like properties of hardwood pulps. A result of 1 mm for the fibre length

analysis confirm that jute could be compared to a good hardwood such as poplar or eucalyptus. The pretty good tear index seemed to be due to the presence of long fibres in relation with the bast fibres part of the plant.
As expected from the viscosity values, the best bleaching sequence in terms of strength of the pulp was D E D, on the contrary, the C E H sequence led to some degradations of the fibres due to hypochlorite stages. The sequence performed with ozone also generated degradations but the high achieved brightness demonstrates that the ozone quantity could be minimized leading to less degradations.
Pulp B issued from cooking with anthraquinone gave better results. This improvement was due to more hemicelluloses saved during the cooking and protecting cellulose during the bleaching. The counter part is that the bleaching yields of pulp B were always lower than pulp A.

In order to appreciate the effect of the enzymatic treatment, the mechanical properties of the biobleached pulps were measured at 40 °SR freeness and compared to the unpretreated pulps (table n°20).

Table 20
Biobleached pulps refined at 40 °SR

Pulp A: Cooked with caustic soda,
Pulp B: Cooked with caustic soda + Anthraquinone,

Pulp	A					B				
	XE CEH	LE CEH	XLE CEH	XLE DED	XLE QP	XE CEH	LE CEH	XLE CEH	XLE DED	XLE OQP
PFI revolution number	1100	1100	1050	2400	1150	800	800	700	2000	1000
Drainage index,°SR	40	42	41	41	41	40	43	39	40	42
Bulk, cm3/g	1.4	1.38	1.39	1.36	1.4	1.33	1.34	1.36	1.3	1.29
Breaking length, km	7.16	7.26	6.94	6.69	6.77	7.57	7.66	7.27	7.98	7.88
Burst index, kPa,m²/g	3.81	3.8	3.76	3.99	3.62	4.27	4.76	4.25	4.56	4.59
Tear index, mNm²/g	8.92	7.94	8.35	9.89	7.87	8.96	8.68	9.42	10.82	9.04
Brightness, %ISO	80.4	79.9	81.7	82.5	69.6	80.2	79.9	81.9	83	80
Opacity fond papier, %	83.7	82.6	82	82.9	86.2	80.3	80.2	80.6	78.8	78.9
Opacity fond blanc, %	78.2	77.6	77.3	77.6	79.7	75.4	75.2	75.9	73.9	73.9
Scattering coefficient S, m²/kg	34.6	34.4	34.4	34.1	33.8	30.9	31	31.6	29.5	29.6
Absorption coefficient K, m²/kg	0.43	0.35	0.29	0.37	0.8	0.29	0.3	0.26	0.26	0.25

These results did not show big differences between the diverse biobleaching sequences nevertheless it was to note a very good tear index especially for the DED type bleaching sequences (what has been already noticed on the unpretreated pulps).
The comparison with the conventional bleached pulps did not underline large differences, thus the conclusion is that enzymatic pretreatment did not bring any detrimental effects.

VI. EFFLUENT ANALYSIS

A comparison of the bleaching effluents was carried out from the 2 pulps (A&B) obtained by the 2 cooking processes, bleached in each case by conventional, ECF and TCF sequences.

COD, AOX, Dry matters and color were analyzed. For the biopretreated sequence, only COD was measured (table n°22)

Table n°21 summarizes the comparison of the conventional, ECF and TCF bleaching sequences.

Table n°21

EFFLUENT ANALYSIS

Based on 1 od mt

	COD kg/T	Total COD kg/T	AOX kg/T	Total AOX kg/T	Dry matter %	Color kg/T Pt/Co	Total color kg/T Pt/Co
Pulp A							
C E H H							
Chlorine step	22,8		3,03		0,32	13,4	
Extraction	22				0,53	41,6	
Hypochlorite 1	5		0,37		0,87	0,85	
Hypochlorite 2	3	52,8	0,17	3,57	0,46	0,36	56,21
D E D							
Chlorie Dioxide 1	19,8		0,59		1,08	8,68	
Extraction	10				0,41	6,03	
Chlorie Dioxide 2	5,9	35,7	0,14	0,73	0,39	1	15,71
O Z P							
Oxygen	40				0,6	42,57	
Ozone	13				0,05	2,4	
Hydrogen Peroxide	26,1	79,1			0,69	1	45,97
Pulp B							
C E H H							
Chlorine step	18,6		1,95		0,34	12,9	
Extraction	20,3				0,56	35,8	
Hypochlorite 1	5		0,39		0,87	0,85	
Hypochlorite 2	5	48,9	0,14	2,48	0,47	0,72	50,27
D E D							
Chlorie Dioxide 1	18,9		0,86		0,99	8,37	
Extraction	6,3				0,37	2,7	
Chlorie Dioxide 2	5,9	31,1	0,16	1,02	0,44	0,85	11,92
O Z P							
Oxygen	35,6				0,5	29,79	
Ozone	15				0,05	2	
Hydrogen peroxide	22	72,6			0,63	0,9	32,69

Figure N°8 and 9 illustrate respectively the COD and AOX values in comparison of the 2 pulps quality and the different bleaching processes.

Figure n°8

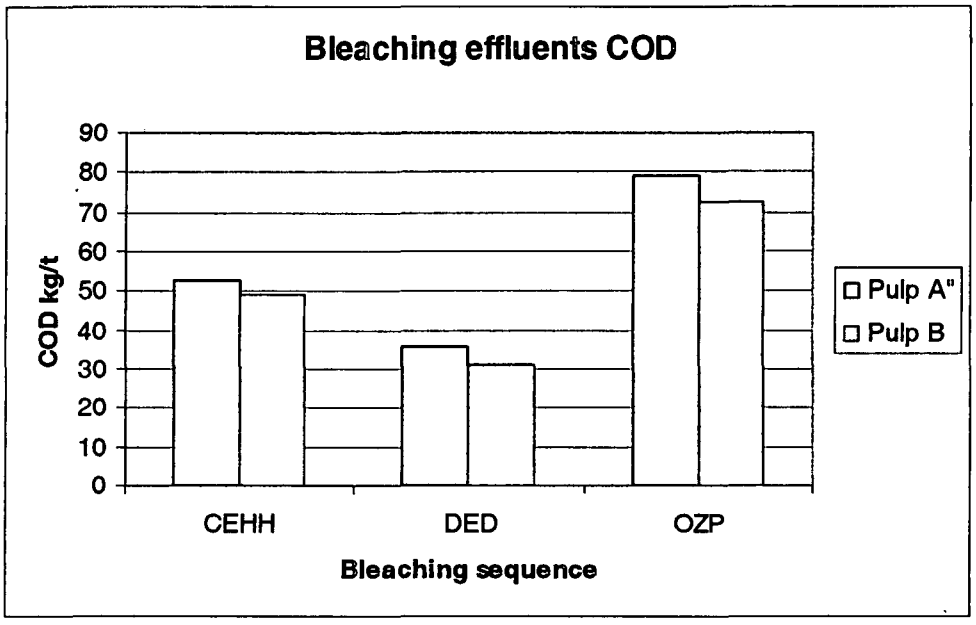


Figure n°9

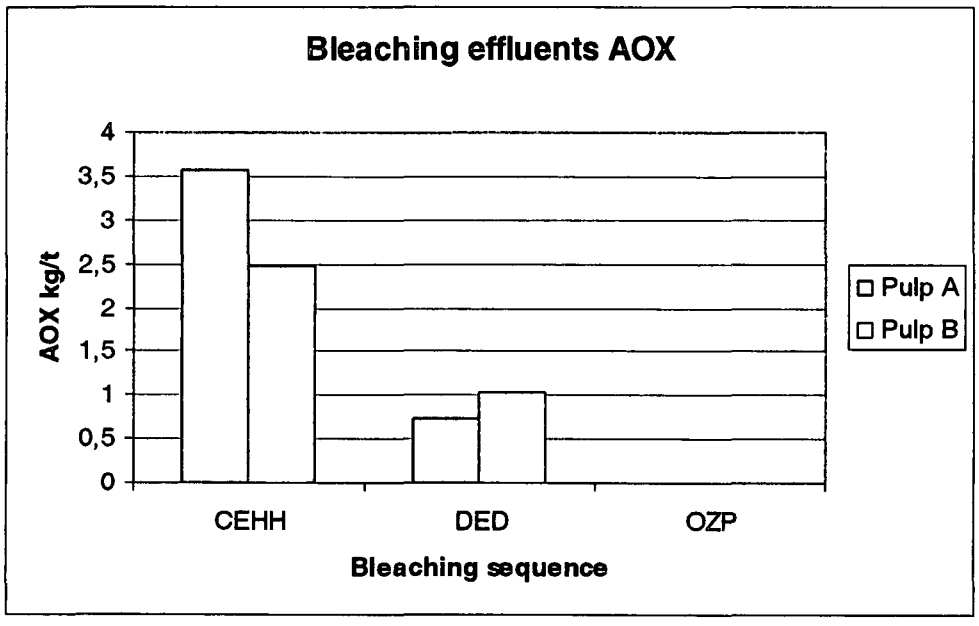


Table n° 22

EFFLUENT ANALYSIS Based on 1 od mt	
	COD kg/T
Pulp A	
C E H H	52.8
D E D	35.7
O Z P	79.1
Pulp B	
C E H H	48.9
X E C E H	54.3
XLE C E H	145.6
D E D	31.1
XLE D E D	140.9
O Z P	72.6
XLE O Q P	168.3

The Soda anthraquinone pulp generated less pollution than the Soda pulp.
Table n°21 showed a positive effect of the ECF bleaching sequence compared with the conventional one on the COD and the AOX content. The TCF bleaching sequence (OZP) led to a higher COD value, but this sequence needed to be optimized because of its higher brightness.

Table 22 was mainly to compare the COD charges issued from bleaching sequences with and without enzymatic pretreatment. A slight increasing was to note for the XE CEH compare to the CEHH conventional bleaching sequence.
The XLE pretreatment led to an important increasing in COD. These results can be explained by the presence of HBT (mediator use in laccase treatment) in the solution. However the behavior of the COD generated by the X, L and E stages should be different than the other stage and should be easily degradable.

VII. FIRST CONCLUSION

The preparation of a reference pulp by a soda process needed an optimization of the cooking. The best selected pulp was the compromise between the lowest kappa number for the highest screened yield.
The selection led to a pulp of kappa number 20.

The effect of the addition of anthraquinone was an improvement of the yield (3% more), of the cellulose polymerization degree (400 units) and of the mechanical properties.

The bleaching sequences tested on the pulps led to the target brightness of 80%ISO. However the CEH sequence needed a double H stage to reach 80%ISO. The ECF sequence (DED) with a good efficiency produced a strong pulp. The TCF sequence (OZP) produced a pulp at high brightness level with some degradations on the cellulose inducing lost of mechanical properties, but the ozone stage should be optimized leading to less degradations.

On an environmental point of view the TCF and ECF sequences generate less toxicity than the conventional one.

The first tests carried out with available commercial enzymes (Xylanase commercialized by NOVO as PULPZYME HC and Laccase developed by INRA as *Pycnoporus cinnabarinus*) demonstrate a good feasibility. The first biobleaching results showed an improvement of the bleachability :

Less chlorine (30 to 40%) was needed to achieve high brightness levels with the CEH sequence.

The ECF and TCF sequences performed on the Xylanase - Laccase treated pulp led to good results either in term of brightness or mechanical properties.

The effluent analysis did not show any improvement : The same COD value was obtained after a xylanase pretreatment followed by a CEH sequence than after a CEHH sequence. The strange results obtained on the Xylanase – Laccase pretreatment seemed due to the use of the mediator in the Laccase step and must be confirmed.

The pulp cooked with caustic soda anthraquinone led to higher final pulp yields whatever the chosen bleaching sequence.

The jute pulp mechanical properties obtained in these trials are suitable for printing and writing paper grades.

CHEMICAL PULP BLEACHING - SECOND PART

VIII. INTRODUCTION

The aim of this second part was to compare two pulps from whole jute issued from soda anthraquinone process

1 : A whole jute soda anthraquinone reference pulp

2 : A pulp from whole jute pretreated with *Ceriporiopsis subvermispora* (as described in the following paragraph) and cooked with the soda anthraquinone process.

Fungal pretreatment experiment :

Fresh Culture of three strains of ***Ceriporiopsis subvermispora***, four strains of ***Phanerochaete chrysosporium*** and ***Fomes lignosus*** (locally isolated strain) was used.

The composition of the inoculum media was 0.3% malt extract, 0.3% yeast extract and 1% glucose. The strain was allowed to grow in stationary conditions for 7 days. Normally strains are inoculated in several conical flasks. Weight of biomass of one or two conical flasks was taken to be used for the determination of required biomass for particular quantity of jute chips.

For bio-pulping initially dried jute plants (***Corchorus olitorius***) were cut into small pieces (2-3 cm), 350 gm of these dried samples were put in polythene bags and 700 ml of water containing 35 mg of KH_2PO_4, 70mg of NaH_2PO_4, 150 mg of $MgSO_4$, 35µg of $CaCl_2$, 35µg $FeSO_4$, 3µg $ZnSO_4$, 7µg $CuSO_4$, and 3µg $MnSO_4$ were added. The bags were autoclaved at 121°C for 15 minutes. Each polythene bag was inoculated separately with 3 strains of ***Ceriporiopsis subvermispora*** (1, 2 & 3), ***Phanerochaete chrysosporium*** and ***Fomes lignosus*** with 10 plugs of fresh culture of these strains. In each bag,, 14g of glucose were also added. The bags were fitted with inlet and outlet tubes for aeration and incubated at 30° C for 14 days. Total moisture content of jute chips including the media was 66.6%. Chips were treated with microbial strains for 15 days. We used 0.8gm of biomass per kg of jute chips. Control experiments were carried out without fungus under the same condition.

Laboratory scale pulping trials were carried out after harvesting, control and chips treated with fungal strain

The two pulps, were then bleached by conventional and ECF bleaching sequence.

An oxygen delignification was also performed in order to test a possible upscaling of the process.

Finally, a xylanase pretreatment was tested on the unbleached pulps to improve the delignification and decrease the chemical charge to apply.

In a second time two pulps issued from kraft cooking process were bleached with three commercial enzymes to be compared and one developed by IJSG. A CEH bleaching was applied on the resulting pretreated pulps to obtain fully bleached ones.

The following tables regroup the cooking conditions and the results.

Soda Anthraquinone cooking

	Normal Chips	*C. subvermispora* pretreated
Cooking Temperature, °C	170	170
Time, min.	90	90
Na_2O Charge, %	17	17
Anthraquinone, %	0.05	0.05
Yield, %	48	48
Kappa number	19.5	16.2
Viscosity, mPa.s	12.6	11.9
Polymerization degree	1050	1020

Kraft cooking

	Normal Chips	*C. subvermispora* pretreated
Cooking Temperature, °C	170	170
Time, min.	120	120
Na_2O Charge, %	17	17
Sulfidity, %	22	22
Yield, %		
Kappa number	17.8	16.5
Viscosity, mPa.s	23.8	15.7
Polymerization degree	1480	1200

These four pulps were supplied by BCIC and IJSG to CTP for bleaching.

IX. COMPARISON OF CONVENTIONAL AND ECF BLEACHINGS ON THE UNBLEACHED PULPS AND AFTER AN OXYGEN DELIGNIFICATION

With regard to an easy industrialization of the project the bleaching study was in a first step conducted on the two pulps using the conventional (CEH) and ECF (DED) bleaching sequences without and with a complementary oxygen delignification stage.. All these treatment were performed on the Soda anthraquinone pulps issued from whole jute and *C Subvermispora* pretreated jute.
The Conventional (CEH), the ECF (DED), the O CEH and O DED bleaching sequences conditions and results are given respectively in table n° 28, 29, 30 and 31.

Table n°28

CEH

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvermispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
C	Pulp consistency, %	3.5	3.5
	Temperature, °C	25	25
	Time, minutes	60	60
	Initial Kappa number	19.5	16.2
	Cl_2 %(0,25*K)	4.88	4.05
	Final pH	1.8	1.82
	Cl_2 consumed %	4.3	3.55
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Time, minutes	60	60
	NaOH, %	2.44	2.03
	Final pH	11.4	11.41
H	Pulp consistency, %	10	10
	Temperature, °C	50	50
	Time, minutes	120	120
	NaClO,%(as Cl_2)	2	2
	NaOH, %	0.5	0.5
	Final pH	11.04	11.09
	NaClO consumed (as Cl_2 %)	1.08	1.01
	Brightness, % ISO	80	83.3
	Bleaching yield, %	94.5	94.7
	DP	680	630
	Viscosity, mPa.s	7.1	6.6

Table n°29

DED

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvermispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
D1	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Time, minutes	60	60
	Initial Kappa number	19.5	16.2
	ClO_2, %(0,25*K/2,63)	1.86	1.54
	Final pH	3.3	3.4
	ClO_2 consumed, %	1.84	1.54
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Time, minutes	60	60
	NaOH, %	2.44	2.03
	Final pH	11.74	11.65
D2	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Time, minutes	120	120
	ClO_2 %	1	1
	Final pH	3.4	3.1
	ClO_2 consumed, %	0.88	0.9
	Brightness, % ISO	83.6	85.2
	Bleaching yield, %	96.8	95.3
	DP	1020	960
	Viscosity, mPa.s	12	10.9

Table n°30

OCEH

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvernispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
O	Pulp consistency, %	10	10
	Temperature, °C	100	100
	Time, minutes	60	60
	Initial Kappa number	19.5	16.2
	O2 pressure, bars	5	5
	NaOH, %	3	3
	$MgSO_4,7H_2O$, %	0.3	0.3
	Final pH	11.67	11.68
	KAPPA number	8.2	9
C	Pulp consistency, %	3.5	3.5
	Temperature, °C	25	25
	Time, minutes	60	60
	Initial Kappa number	8.2	9
	Cl_2 %(0,25*K)	2.05	2.25
	Final pH	2.2	2.33
	Cl_2 consumed %	1.66	1.84
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Time, minutes	60	60
	NaOH, %	1.03	1.13
	Final pH	11.26	11.42
H	Pulp consistency, %	10	10
	Temperature, °C	50	50
	Time, minutes	120	120
	NaClO,%(as Cl_2)	2	2
	NaOH, %	0.5	0.5
	Final pH	11.08	11.19
	NaClO consumed (as Cl_2 %)	0.94	0.82
	Brightness, % ISO	84.8	86.8
	Bleaching yield, %	92.2	92
	DP	570	520
	Viscosity, mPa.s	5.9	5.5

Table n°31

ODED

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvernispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29.7	Pulp 02 Brightness 33.6
O	Pulp consistency. %	10	10
	Temperature. °C	100	100
	Time. minutes	60	60
	Initial Kappa number	19.5	16.2
	O2 pressure. bars	5	5
	NaOH. %	3	3
	MgSO4.7H2O. %	0.3	0.3
	Final pH	11.67	11.68
	KAPPA number	8.2	9
D1	Pulp consistency. %	10	10
	Temperature. °C	70	70
	Time. minutes	60	60
	Initial Kappa number	8.2	9
	ClO2. %(0.25*K/2.63)	0.78	0.86
	Final pH	4.63	6.3
	ClO2 consumed. %	0.76	0.72
E	Pulp consistency. %	10	10
	Temperature. °C	60	60
	Time. minutes	60	60
	NaOH, %	1.03	1.13
	Final pH	11.61	11.56
D2	Pulp consistency. %	10	10
	Temperature. °C	70	70
	Time. minutes	120	120
	ClO_2 %	1	1
	Final pH	3.52	3.6
	ClO_2 consumed %	0.92	0.98
	Brightness %, ISO	87.1	87.4
	Bleaching yield, %	90.7	91.7
	DP	910	900
	Viscosity, mPa.s	10.2	10

Table n°32 summarizes the total chlorine charges applied during the different bleaching sequences, table n°33 regroups the obtained final brightness.

Table n°32
Chlorine charge % applied

	Pulp 01	Pulp 02
CEH	6.0	5.1
O CEH	3.0	3.1
DED	7.2	6.4
O DED	4.5	4.8

Table n°33
Final brightness %ISO

	Pulp 01	Pulp 02
CEH	80	83.3
O CEH	84.8	86.8
DED	83.6	85.2
O DED	87.1	87.4

An oxygen delignification led to 50% and 40% chlorine charge reduction on the CEH bleaching sequence respectively for pulp 1 and pulp 2. In spite of this reduction, the final brightness increase from 80.0 to 84.8%ISO and 83.3 to 86.8 %ISO respectively.
The biopretreatment of the jute led to 15% chlorine reduction charge, only if oxygen stage was not used.
The oxygen stage seemed to minimize this effect leading almost to the same chlorine charge, however the brightness was improved by 2 units (85.2 to 87.4 %ISO for DED and 83.3 to 86.8 %ISO for CEH). Fungal pretreatment helped to remove lignin during cooking and oxygen delignification

Regarding the DED sequence, the final brightness was higher than for the CEH sequence, but the equivalent chlorine charge was higher, due to a supplementary consumption in the D2 stage compared to the hypochlorite stage.
The same remarks could be done about the effect of the biopretreatment of the chips and on the use of oxygen on the chlorine charge, however these effects were less marked than for the CEH sequence (11% chlorine reduction charge by the bio pretreatment and 38% reduction by the oxygen stage).
The brightness gain due to the oxygen stage was also comparable with the CEH trials and the final brightness achieved at 87.1 %ISO and 87.4 %ISO respectively for pulp 01 and pulp 02. These brightness were far from the target (7 units) inducing that a reduction of the chlorine or chlorine dioxide charge could be envisaged for industrial application.

Table n° 34 represents the bleaching yields

Table n°34
Bleaching yields %

	Pulp 01	Pulp 02
CEH	94.5	94.7
O CEH	92.2	92.0
DED	96.8	95.3
O DED	90.7	91.7

There was no significant differences between pulp 01 and pulp 02. The oxygen stage seemed to reduce significantly the bleaching yield and consequently the total yield. The oxygen stage oxidized hemicelluloses and lignin left in the pulp leading to soluble compounds and also part of the cellulose underlined by the viscosity drop, about 100 units of polymerization degree (see table n°35).
The CEH sequence was more detrimental to cellulose than DED.

Table n°35
Pulp polymerization degree

	Pulp 01	Pulp 02
CEH	680	630
O CEH	570	520
DED	1020	960
O DED	910	900

X. XYLANASE PRETREATMENT

Enzymatic pretreatment

During the previous work (cf chemical pulp 1st part), an enzymatic pretreatment was associated to an alkaline extraction. It was necessary to compare the effect of a X (Enzyme), X E (enzyme followed by an extraction stage) and E (alkaline extraction stage) to determine exactly the efficiency of enzymes.

The treatment conditions and the results are given respectively in table 36, 37, and 38 for the X CEH, XE CEH and E CEH bleaching.

Table n°36

XCEH

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvermispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29.7	Pulp 02 Brightness 33.6
X	Pulp consistency, %	5	5
	Temperature, °C	50	50
	Duration, minute	120	120
	Initial Kappa number	19.5	16.2
	Xylanase, u/g (Pulpzyme)	1	1
	Buffer pH7, mMole/l	25	25
	Final pH	6.93	7
	KAPPA number	17.2	14.9
C	Pulp consistency, %	3.5	3.5
	Temperature, °C	25	25
	Duration, minute	60	60
	Initial Kappa number	17.2	14.9
	Cl_2, %(0,25*K)	4.3	3.73
	Final pH	1.82	1.93
	Cl_2 consumed, %	4.1	3.5
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Duration, minute	60	60
	NaOH, %	2.15	1.87
	Final pH	11.42	11.37
H	Pulp consistency, %	10	10
	Temperature, °C	50	50
	Duration, minute	120	120
	NaClO,%(as Cl_2)	2	2
	NaOH, %	0.5	0.5
	Final pH	10.94	11.06
	NaClO consumed (as Cl_2 %)	0.97	0.88
	Brightness, % ISO	80.6	83.2
	Bleaching yield, %	93.23	93.09
	DP	630	620
	Viscosity, mPa.s	6.5	6.5

Table n°37

XECEH

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvernispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
X	Pulp consistency, %	5	5
	Temperature, °C		50
	Duration, minute	120	120
	Initial Kappa number	19.5	16.2
	Xylanase u/g (Pulpzyme)	1	1
	Buffer pH7 mMole/l	25	25
	Final pH	6.93	7
	KAPPA number	17.2	14.9
E	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	90	90
	NaOH %	2.5	2.5
	Final pH	11.49	11.53
	KAPPA number	12.1	12.3
C	Pulp consistency, %	3.5	3.5
	Temperature, °C	25	25
	Duration, minute	60	60
	Initial Kappa number	12.1	12.3
	Cl_2 %(0,25°K)	3.03	3.08
	Final pH	1.98	2.1
	Cl_2 consumed, %	2.78	2.8
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Duration, minute	60	60
	NaOH, %		
	Final pH	11.33	11.42
H	Pulp consistency, %	10	10
	Temperature, °C	50	50
	Duration, minute	120	120
	NaClO,%(as Cl_2)	2	2
	NaOH, %	0.5	0.5
	Final pH	10.86	11.02
	NaClO consumed (as Cl_2 %)	1.05	0.92
	Brightness %, ISO	82.5	85.5
	Bleaching yield, %	94.44	93.72
	DP	620	580
	Viscosity, mPa.s	6.5	6.1

Table n°38

ECEH

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvernispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
E	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	90	90
	Initial Kappa number	19.5	16.2
	NaOH %	2.5	2.5
	Final pH	11.41	11.47
	KAPPA number	15.9	14.3
C	Pulp consistency, %	3.5	3.5
	Temperature, °C	25	25
	Duration, minute	60	60
	Initial Kappa number	15.9	14.3
	Cl_2 %(0,25*K)	3.98	3.58
	Final pH	1.82	1.98
	Cl_2 consumed %	3.94	3.34
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Duration, minute	60	60
	NaOH, %	1.99	1.79
	Final pH	11.47	11.48
H	Pulp consistency, %	10	10
	Temperature, °C	50	50
	Duration, minute	120	120
	NaClO,%(as Cl_2)	2	2
	NaOH, %	0.5	0.5
	Final pH	11	10.97
	NaClO consumed (as Cl_2 %)	1.01	0.95
	Brightness %, ISO	81	84.1
	Bleaching yield, %	96.16	95.37
	DP	590	580
	Viscosity, mPa.s	6.2	6

Results

These results showed a drop of Kappa number after the pretreatment (table n°39) and consequently a reduction of the chlorine charge (table n°40.

Table n°39

Kappa number drop along bleaching sequences

	Untreated		*C.Subvermispora*	
X	2.3	Total 5.9	1.3	Total 3.6
E	3.6		1.9	
XE	7.4		3.9	

The xylanase stage induced a lower kappa number drop than extraction stage (2.3 and 3.6 respectively), but the XE stage was more efficient than the total of the two stages (7.4 compare to 5.9). That meant that the xylanase reacted on the lignin leading to more soluble compounds removed by alkaline extraction. The pretreatment applied on the pulp issued from the chips pretreated with *C.Subvermispora* had lower results, but the pretreatment led to the same Kappa number (12.1, 12.3 respectively for the untreated chips and the pretreated).The pulp issued from the pretreated chips had initially a lower kappa number, and the enzymes reacted selectively on only the residual xylans linked to lignin inducing some breakage of the bonds. The effect of the biotreatment of the chips seemed not to be cumulative with the xylanase stage in the bleaching sequence

Table n°40

Chlorine charge % applied

	Untreated	*C.Subvermispora*
Reference CEH	4.9	4.1
X CEH	4.3	3.7
E CEH	4.0	3.6
XE CEH	3.0	3.1

A Xylanase stage led to 12 and 10% chlorine charge savings respectively for pulp 01 and pulp 02.

An alkaline extraction led to 18 and 12% chlorine charge reduction respectively for pulp 01 and pulp 02.

The combination of the two stages(XE pretreatment) led to 39 and 25% chlorine charge reduction respectively for pulp 01 and pulp 02.

The XE pretreatment demonstrated the efficiency of xylanase treatment of pulps to reduce chlorine charge beside the brightness was increased by 2.5 units (table n°41).

Table n°41

Brightness, %ISO

	Untreated	*C.Subvermispora*
Reference CEH	80	83.3
X CEH	80.6	83.2
E CEH	81.0	84.1
XE CEH	82.5	85.5

The bleaching yields were comparable, however 1 point in bleaching yield was noticed in case of pretreated chips comparing CEH and XE CEH but it will not affect the fully bleached pulp yield.
A slight drop in viscosity was noticed for the XE CEH sequence compare to the reference (table n°42).

Table n°42
Viscosity results

	Untreated	*C.Subvermispora*
Reference CEH	680	630
X CEH	630	620
E CEH	590	580
XE CEH	620	580

It could be noticed that this drop was mainly due to alkaline extraction and not the xylanase stage, revealing the attack of sodium hydroxide to cellulose
Following all these results, the most interesting biobleaching sequence was a sequence including a XE stage.

XI. COMPARISON OF CONVENTIONAL AND ECF BLEACHING SEQUENCES AFTER A BIOPRETREATMENT

The subject of this part was to compare the efficiency of the conventional and the ECF bleaching sequence on pulps pretreated by XE
The XE pretreatment was perform on the 2 pulps as described previously. The pretreatment was also applied after the oxygen stage.
The CEH and DED part of the sequences were applied in the same conditions as the conventional and the ECF sequences previous trials, the chemical rate applied were based on the kappa number after XE.

Bleaching sequences :

XE CEH
XE DED
O XE CEH
O XE DED

All the results are given respectively in table n° 43 to 46 for XE CEH, XE DED, O XE CEH, O XE DED

Table n°43

XECEH

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvernispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
X	Pulp consistency, %	5	5
	Temperature, °C		50
	Duration, minute	120	120
	Initial Kappa number	19.5	16.2
	Xylanase u/g (Pulpzyme)	1	1
	Buffer pH7 mMole/l	25	25
	Final pH	6.93	7
	KAPPA number	17.2	14.9
E	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	90	90
	NaOH %	2.5	2.5
	Final pH	11.49	11.53
	KAPPA number	12.1	12.3
C	Pulp consistency, %	3.5	3.5
	Temperature, °C	25	25
	Duration, minute	60	60
	Initial Kappa number	12.1	12.3
	Cl_2 %(0,25*K)	3.03	3.08
	Final pH	1.98	2.1
	Cl_2 consumed, %	2.78	2.8
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Duration, minute	60	60
	NaOH, %		
	Final pH	11.33	11.42
H	Pulp consistency, %	10	10
	Temperature, °C	50	50
	Duration, minute	120	120
	NaClO,%(as Cl_2)	2	2
	NaOH, %	0.5	0.5
	Final pH	10.86	11.02
	NaClO consumed (as Cl_2 %)	1.05	0.92
	Brightness %, ISO	82.5	85.5
	Bleaching yield, %	94.44	93.72
	DP	620	580
	Viscosity, mPa.s	6.5	6.1

Table n°44

XEDED

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvernispora* and Cooked with caustic soda + Anthraquinone.

STAGES		Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
X	Pulp consistency, %	5	5
	Temperature, °C	50	50
	Duration, minute	120	120
	Initial Kappa number	19.5	16.2
	Xylanase, u/g (Pulpzyme)	1	1
	Buffer pH7, mMole/l	25	25
	Final pH	6.93	7
	KAPPA number	17.2	14.9
E	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	90	90
	NaOH, %	2.5	2.5
	Final pH	11.49	11.53
	KAPPA number	12.1	12.3
D1	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	60	60
	Initial Kappa number	12.1	12.3
	ClO_2, %(0,25*K/2,63)	1.15	1.17
	Final pH	4.11	4.58
	ClO_2 consumed, %	1.15	1.14
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Duration, minute	60	60
	NaOH, %	1.51	1.54
	Final pH	11.72	11.82
D2	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	120	120
	ClO_2, %	1	1
	Final pH	3.38	3.83
	ClO_2 consumed, %	0.94	0.97
	Brightness %, ISO	83.9	85.1
	Bleaching yield, %	93.1	94.4
	DP	1160	1190
	Viscosity, mPa.s	14.9	15.5

Tablen°45

OXECEH

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvemispora* and Cooked with caustic soda + Anthraquinone.

STAGES	O step before XECEH	Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
X	Pulp consistency, %	5	5
	Temperature, °C	50	50
	Duration, minute	120	120
	Kappa number after O step	8.2	9
	Xylanase, u/g (Pulpzyme)	1	1
	Buffer pH7, mMole/l	25	25
	Final pH	7	6.98
E	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	90	90
	NaOH, %	2.5	2.5
	Final pH	11.46	11.48
	KAPPA number	6.3	6.7
C	Pulp consistency, %	3.5	3.5
	Temperature, °C	25	25
	Duration, minute	60	60
	Initial Kappa number	6.3	6.7
	Cl_2, %(0,25*K)	1.58	1.68
	Final pH	2.5	2.78
	Cl_2 consumed, %	1.32	1.35
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Duration, minute	60	60
	NaOH, %	0.79	0.84
	Final pH	10.92	11.04
H	Pulp consistency, %	10	10
	Temperature, °C	50	50
	Duration, minute	120	120
	NaClO,%(as Cl_2)	2	2
	NaOH, %	0.5	0.5
	Final pH	11.15	11.16
	NaClO consumed (as Cl_2 %)	0.76	0.7
	Brightness, % ISO	84.8	87.4
	Bleaching yield, %	91	89
	DP	630	580
	Viscosity, mPa.s	6.5	6.1

Table n°46

OXEDED

Pulp 7M008-C09-01: Cooked with caustic soda + Anthraquinone.
Pulp 7M008-C09-02: Treated with *C. subvernispora* and Cooked with caustic soda + Anthraquinone.

STAGES	O step before XEDED	Pulp 01 Brightness 29,7	Pulp 02 Brightness 33,6
X	Pulp consistency, %	5	5
	Temperature, °C	50	50
	Duration, minute	120	120
	Kappa number after O step	8.2	9
	Xylanase, u/g (Pulpzyme)	1	1
	Buffer pH7, mMole/l	25	25
	Final pH	7	6.98
E	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	90	90
	NaOH, %	2.5	2.5
	Final pH	11.46	11.48
	KAPPA number	6.3	6.7
D1	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	60	60
	Initial Kappa number	6.3	6.7
	ClO_2, %(0,25*K/2,63)	0.6	0.64
	Final pH	5.82	6.21
	ClO_2 consumed, %	0.5	0.55
E	Pulp consistency, %	10	10
	Temperature, °C	60	60
	Duration, minute	60	60
	NaOH, %	0.79	0.84
	Final pH	11.7	11.74
D2	Pulp consistency, %	10	10
	Temperature, °C	70	70
	Duration, minute	120	120
	ClO_2, %	1	1
	Final pH	3.9	4.6
	ClO_2 consumed, %	0.97	0.98
	Brightness, % ISO	86.1	87
	Bleaching yield, %	92.5	93.1
	DP	970	1060
	Viscosity, mPa.s	11.1	12.8

Table n°47summarizes the Kappa number evolution after the diverse treatments.
The effect of fungi pretreatment and of the other ones on kappa number are never cumulative, except for the X stage.
The XE pretreatment induced a drop in Kappa number, 19.5 to 12.1 for the reference pulp (01) and 16.2 to 12.3 for the pulp with fungal pretreatment (02). The kappa number after XE pretreatment was equivalent, underlining that the benefit of the chips pretreatment was reduced on the unbleached pulp. This meant that the xylanase only react on xylans linked to the lignin. An oxygen pretreatment already reduced the effect of biopretreatment of the chips leading to kappa number 8.2 and 9.0 respectively for pulp 01 and pulp 02. A XE pretreatment on the pulp after oxygen did not generate differences on the 2 pulps, the final kappa achieved at 6.3 and 6.7 respectively for pulp 01 and pulp 02.

Table n°47

	Initial	X	XE	O	OXE
Kappa number pulp 01	19.5	17.2	12.1	8.2	6.3
Kappa number pulp 02	16.2	14.9	12.3	9.0	6.7

Nevertheless, these decreases in kappa number consequently reduced the chemical charges to apply for the rest of the bleaching sequences. To illustrate this, the chlorine charges applied (as Cl_2 reacted for all the bleaching sequences) are represented in table n°48. Table n° 49 give the corresponding brightness.

Table n° 48
% of Cl_2 charge

	CEH	DED	XECEH	XEDED	OCEH	ODED	OXECEH	OXEDED
Pulp 1	6.0	7.2	4.1	5.5	3.0	4.5	2.3	4.1
Pulp 2	5.0	6.4	4.0	5.6	3.1	4.8	2.4	4.3

Table n° 49
Brightness %ISO

	CEH	DED	XECEH	XEDED	OCEH	ODED	OXECEH	OXEDED
Pulp 1	80.0	83.6	82.5	83.9	84.8	87.1	84.8	86.1
Pulp 2	83.3	85.2	85.5	85.1	86.8	87.4	87.4	87

The XE pretreatment induced 32% chemical saving in case of conventional CEH bleaching and 24% saving in case of ECF bleaching.
An oxygen stage reduced 50% and 38% the chemical consumption respectively for the conventional and the ECF bleaching sequence.
The combination of oxygen followed by an enzyme treatment led to 62% and 43% reduction charge respectively for the conventional and the ECF bleaching sequences.
The enzyme treatment after the oxygen stage reduced by 23% and 9% respectively for conventional and ECF bleaching.
Concerning the brightness results the chemical charges could be reduced some more for a brightness target of 80% ISO.
In all cases, the achieved final brightness was higher than the target and we could appreciate a benefit of XE, Oxygen and OXE pretreatment. In the same way, the

pulp issued from the *C. subvermispora* chips achieved in every cases at 2 or 3% higher brightness than the pulp without chip pretreatment. The similarity of the kappa number of the two pulps after the pretreatment and the final higher brightness for pulp 02 underlines a modification of the lignin by the fungal pretreatment, making it easier to remove by the bleaching chemicals.

XII. EFFLUENT ANALYSIS

COD, BOD and AOX analysis were carried out on the total effluent of each bleaching sequence, (table n°50)

Table n°50

	COD Kg/t	BOD Kg/t	COD/BOD	Bleaching Yields	AOX Kg/t
CEH pulp 01	53.20	8.80	6,00	94,50	4.13
CEH pulp 02	45.77	8.61	5,30	94,70	3.09
DED pulp 01	36.94	6.48	5,70	96,80	0.41
DED pulp 02	23.22	6.29	3,70	95,30	0.24
O CEH pulp 01	59.94	9.08	6,60	92,20	1.13
O CEH pulp 02	54.27	10.58	5,10	92,00	1.16
O DED pulp 01	51.57	9.21	5,60	90,70	0.09
O DED pulp 02	43.27	8.65	5,00	91,70	0.09
XE CEH pulp 01	66.49	12.45	5,30	94,40	2.06
XE CEH pulp 02	72.73	12.87	5,60	93,70	1.88
XE DED pulp 01	67.21	11.13	6,00	93,10	0.10
XE DED pulp 02	62.06	11.06	5,60	94,40	0.13
OXE CEH pulp 01	77.68	12.76	6,00	91,00	1.46
OXE CEH pulp 02	69.52	13.05	5,30	89,00	1.73
OXE DED pulp 01	71.70	13.73	5,20	92,50	0.09
OXE DED pulp 02	62.20	14.35	4,30	93,10	0.07

It was observed an increase in COD and BOD with biobleaching sequence. This was correlated with the bleaching yields. However the XE pretreatment with higher bleaching yields generate more COD than the oxygen treatment. Nevertheless these COD and BOD levels were quite low and acceptable.
In all cases, the ECF bleaching sequences generated less COD and BOD.
The pulp issued from the pretreated chips generates also less COD and BOD comparatively to the normal pulp.
In term of biodegradability, these effluents were comparable with mill effluents. Some ratio COD/BOD lower than 6 indicated an easier biodegradability.
Globally, the ECF pulps had a lower ratio compared to conventional bleaching and pulp 2 with chips pretreatment was also in a good position compared to pulp 1.
The AOX content was considerably decreased, due to the chemical reduction charges and especially in the bleaching sequences with oxygen. The XE pretreatment looked less efficient than the oxygen stage.
Regarding the AOX content, the ECF bleaching sequence was preferable.

XIII. MECHANICAL PROPERTIES

During the first part of this work, the mechanical properties evolution during refining was tested. The optimum was found at 40°SR. The tested pulps were refined at 40°SR in a PFI mill.
The bleaching sequences selected were all those including a CEH part, and only the DED and the OXEDED for the ECF bleaching.
The results are given in table n°51.

No significant differences were underlined for all the tested pulps.
The mechanical properties were quite good and these pulps were suitable for printing and writing paper grades.

All the pulps issued from pretreated chips had a slightly lower mechanical properties and especially the tear index.

The ECF pulps had comparable mechanical properties but the tear index was higher than for bleached pulps with CEH containing sequences. It seemed to be correlated with the viscosity.
The use of a xylanase pretreatment did not bring any improvement of the mechanical properties in spite of the chemical charge reduction they allowed.

Table n°51

Mechanical properties

	CEH		DED		OCEH		XECEH		OXECEH		OXEDED	
	Pulp 1	Pulp 2	Pulp 1	Pulp 2	Pulp 1	Pulp 2	Pulp 1	Pulp 2	Pulp 1	Pulp 2	Pulp 1	Pulp 2
Bulk, cm^3/g	1.47	1.43	1.38	1.39	1.45	1.46	1.46	1.45	1.42	1.48	1.42	1.41
Tensile, kN/m	4.79	4.9	5.41	5.33	4.99	4.76	4.83	4.84	5.32	4.76	5.35	5.12
Tensile index, Nm/g	61.7	62.1	69	69.3	65.1	62.5	61.8	61.5	67.1	62.5	67.7	65
Breaking length, m	6290	6330	7040	7060	6630	6370	6300	6270	6840	6370	6900	6630
Stretch, %	3.6	3.9	2.6	2.8	2.8	2.8	2.9	3	3.5	2.8	3.1	3.5
Burst index, $kPa.m^2/g$	3.26	3.43	3.99	3.83	3.35	3.24	3.3	3.73	3.69	3.32	4.08	3.77
Tear index, $mN.m^2/g$	8.14	7.48	9.12	8.86	7.46	7	8.05	7.8	8.89	7.98	9.5	8.87
Opacity, %ISO	85.7	85.3	83.4	84.2	82.9	83.2	86.2	86.4	83.5	83.9	84.5	84.9
Absorption coefficient K, m^2/kg	0.44	0.33	0.34	0.34	0.23	0.2	0.42	0.35	0.2	0.19	0.31	0.28
Scattering coefficient S, m^2/kg	40.4	41.7	36.7	39.6	40.2	42.6	42.1	44.4	41	43.9	40.2	42.2

XIV. COMPARISON OF 3 COMMERCIAL ENZYMES AND 1 LABORATORY DEVELOPED ENZYME.

On kraft pulps with and without jute chip biopretreatment, the efficiency of 3 commercial enzymes and a specific enzyme developed by IJSG was studied.
In each case, the enzyme pretreatment was followed by an alkaline extraction.
Prior to the trials, the enzyme activity was measured at pH 7 on all the enzymes.
2 units/g of pulp was applied at pH 7 during 1 hour at 50°C.
A E stage was carried out on the kraft pulps in order to control the efficiency of XE treatment.
Table n° 52 summarizes the enzyme pretreatment results.

Table n°52
Comparison of the enzyme efficiency
Kappa number

	Initial pulp	After E		Pulpzyme	Biobrite	Ecopulp	IJSG
Pulp 1	17.3	15.8	After X	15.3	15.2	15.9	16
			After XE	12.4	12.2	12.9	14
Pulp 2	16.5	15.1	After X	14.6	14.2	15	15.4
C.Subvermispora			After XE	12.4	12.2	12.9	13.9

In the applied conditions, the best enzymes were Pulpzyme from NOVO and Biobrite from IOGEN.
The Kappa number after XE treatment achieved the same values for the 2 pulps, suppressing the kappa number difference observed after cooking.
Table 54 and 55 give the results respectively for the jute kraft pulp and for the biopretreated jute kraft pulp.

After bleaching, no differences was underlined. The 2 pulps achieved higher level than the 80%ISO brightness target. However the pulp obtained with the pretreated jute reached a higher brightness (82.3 % to 83.9 %ISO compared to 80.6 % to 81.7 %ISO on the normal kraft pulp).
The final viscosities were lower (600 to 620 and 750 to 780 polymerization degree) on the pretreated jute kraft pulp, but it was a consequence of the viscosities obtained after cooking (1200 instead of 1480). Bleaching did not recover the initial loss in viscosity observed during cooking.

In comparison with the soda AQ pulps, the kraft pulps seemed more difficult to bleach, achieving lower brightness even with a lower kappa number on the unbleached pulps (table n°53).

Table n°53
Final brightness after XE CEH, %ISO

	Soda AQ	Kraft
Normal process	82.5	81.4
C.Subvermispora treated	85.5	83.7

Table n°54

Reference Kraft pulp

Initial Brightness 26 **Viscosity mPa.s** 23.8
DP 1480

STAGES		1 Pulpzyme	2 Biobrite	3 Ecopulp	4 IJSG	5 Control
X	Pulp consistency, %	5	5	5	5	
	Temperature, °C	50	50	50	50	
	Time, min.	60	60	60	60	
	Initial Kappa number	17.8	17.8	17.8	17.8	17.8
	Enzyme, u/g	2	2	2	2	
	Buffer pH7 mMole/l	25	25	25	25	
	Final pH	6.82	6.89	6.85	6.9	
	KAPPA number	15.3	15.2	15.9	16	
E	Pulp consistency, %	10	10	10	10	10
	Temperature, °C	70	70	70	70	70
	Time, min.	90	90	90	90	90
	NaOH, %	2.5	2.5	2.5	2.5	2.5
	Final pH	11.51	11.66	11.64	11.69	11.57
	KAPPA number	12.4	12.2	12.9	14	15.8
C	Pulp consistency, %	3.5	3.5	3.5	3.5	3.5
	Temperature, °C	25	25	25	25	25
	Time, min.	60	60	60	60	60
	Initial Kappa number	12.4	12.2	12.9	14	15.8
	Cl_2, %(0,25*K)	3.1	3.05	3.22	3.5	3.95
	Final pH	2.29	2.31	2.27	2.16	2.08
	Cl_2 consumed, %	2.82	2.72	2.83	2.95	3.56
E	Pulp consistency, %	10	10	10	10	10
	Temperature, °C	60	60	60	60	60
	Time, min.	60	60	60	60	60
	NaOH, %	1.55	1.52	1.61	1.75	1.97
	Final pH	11.44	11.4	11.54	11.42	11.49
H	Pulp consistency, %	10	10	10	10	10
	Temperature, °C	50	50	50	50	50
	Time, min.	120	120	120	120	120
	NaClO (as Cl_2) %	2	2	2	2	2
	NaOH %	0.5	0.5	0.5	0.5	0.5
	Final pH	11.12	11	11.05	11.04	10.95
	NaClO consum (as Cl_2) %	1.01	1.03	0.95	1.01	1.1
	Brightness, % ISO	81.4	81.7	81.4	80.6	80.9
	Bleaching yield, %					
	DP	753	768	753	758	782
	Viscosity, mPa.s	8	8.2	8	8	8.3

Table n°55

Biopretreated Kraft pulp

Initial Brightness 30 **Viscosity mPa.s** 15,7
DP 1200

STAGES		1 Pulpzyme	2 Biobrite	3 Ecopulp	4 IJSG	5 Control
X	Pulp consistency, %	5	5	5	5	
	Temperature, °C	50	50	50	50	
	Time, min.	60	60	60	60	
	Initial Kappa number	16.5	16.5	16.5	16.5	16.5
	Enzyme, u/g	2	2	2	2	
	Buffer pH7 mMole/l	25	25	25	25	
	Final pH	6.95	6.9	6.86	6.91	
	KAPPA number	14.6	14.2	15	15.4	
E	Pulp consistency, %	10	10	10	10	10
	Temperature, °C	70	70	70	70	70
	Time, min.	90	90	90	90	90
	NaOH, %	2.5	2.5	2.5	2.5	2.5
	Final pH	11.57	11.63	11.49	11.72	11.52
	KAPPA number	12.4	12.2	12.9	13.9	15.1
C	Pulp consistency, %	3.5	3.5	3.5	3.5	3.5
	Temperature, °C	25	25	25	25	25
	Time, min.	60	60	60	60	60
	Initial Kappa number	12.4	12.2	12.9	13.9	15.1
	Cl_2, %(0,25*K)	3.1	3.05	3.22	3.48	3.78
	Final pH	2.36	2.38	2.32	2.18	2.18
	Cl_2 consumed, %	2.66	2.58	2.72	2.98	3.42
E	Pulp consistency, %	10	10	10	10	10
	Temperature, °C	60	60	60	60	60
	Time, min.	60	60	60	60	60
	NaOH, %	1.55	1.52	1.61	1.74	1.89
	Final pH	11.44	11.5	11.47	11.4	11.41
H	Pulp consistency, %	10	10	10	10	10
	Temperature, °C	50	50	50	50	50
	Time, min.	120	120	120	120	120
	NaClO (as Cl_2) %	2	2	2	2	2
	NaOH %	0.5	0.5	0.5	0.5	0.5
	Final pH	11.05	10.97	10.96	11.13	11.03
	NaClO consum (as Cl_2) %	0.97	0.99	0.96	0.97	1.02
	Brightness, % ISO	83.7	83.9	83.2	82.6	82.3
	Bleaching yield, %					
	DP	612	615	596	622	621
	Viscosity, mPa.s	6.4	6.4	6.2	6.5	6.5

XV. CONCLUSION

Without any enzyme pretreatment on unbleached pulps, a DED bleaching sequence led to better brightness, bleaching yield and viscosity than CEH sequence.
An oxygen delignification prior to the bleaching sequence would be an alternative to reduce the chemical consumption, a decrease of 50% less chlorine in case of CEH and 38% in case of DED. The brightness with less chlorine or chlorine dioxide were increased in 3 to 4 points in brightness.
A Xylanase stage followed by an alkaline extraction induced a reduction of the chemical consumption of 32% in case of conventional bleaching and 24% in case of ECF bleaching.
The OXE pretreatment led to 62% and 43% reduction respectively to conventional and ECF pulps.

The pulp produced with the chips pretreated with *Ceriporiopsis subvermispora* was bleached to higher final brightness with comparable chemical charges.
A pretreatment of the unbleached pulp (O, XE, OXE) decreased the effect of the fungal treatment whatever it is, leading to equivalent kappa number before bleaching.

With regard to the brightness target of 80%ISO, the chemical charges could be reduced in every cases, except for the CEH bleaching sequence.

On an environmental point of vue, the COD and BOD charges in the effluents increased when applying a pretreatment before the bleaching sequence (O ,XE and OXE)
However these values remained in an acceptable level, and the biodegradability was not affected.
It was important to underline that the ECF bleaching sequence generated less COD and BOD charges as well as the pulp issued from the pretreated chips.
The XE bleaching pretreatment led to slightly better bleaching yields.

The AOX content was considerably decreased in case of conventional bleaching (4.1 and 3.1 kg/t to 1.1- 1.2 kg/t with O stage, 2.1 – 1.9 kg/t with XE stage and 1.5 – 1.7 kg/t with OXE stage).
The mechanical properties were similar for all the pulps after bleaching leading to rather acceptable pulp qualities. A slightly better tear index for the ECF pulps.

The oxygen stage seemed to be the most interesting first bleaching stage but at mill scale it will need more investment and more maintenance.

The enzyme stage had be followed with an alkaline extraction for a better efficiency in delignification.

In the studied conditions, the commercial enzymes, and the IJSG one led to better results (Pulpzyme from NOVO and Biobrite from IOGEN). However all the enzymes had a positive effect. The IJSG proposed enzyme would have been tested in diverse conditions of pH for probably a better efficiency.
These trials underlined a better bleachability of the Soda AQ pulps in comparison with the kraft pulps.

JUTE HIGH YIELD PULPS

XVI. INTRODUCTION

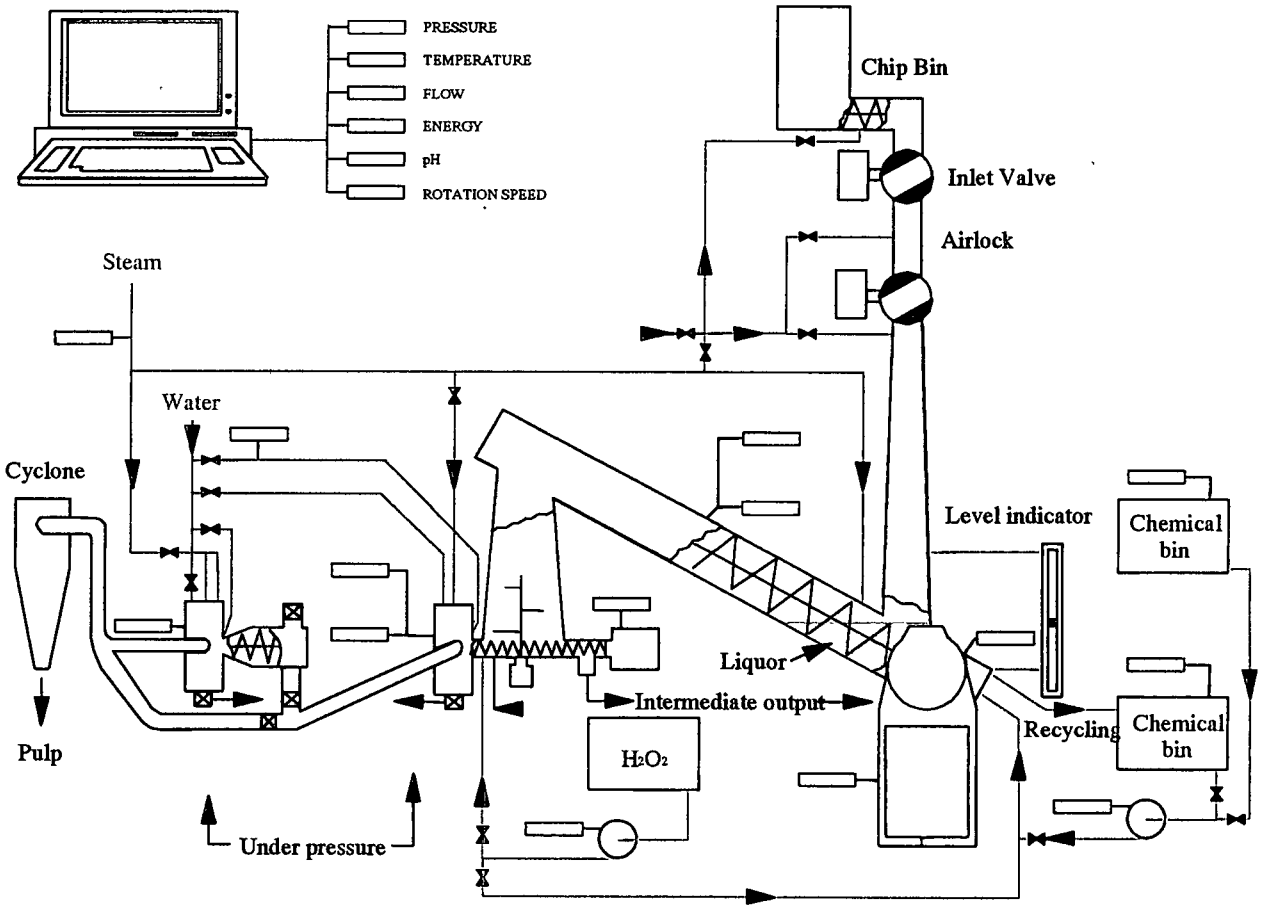
Jute as a fibre resource had to be tested in mechanical pulping such as TMP and APMP processes. The association of long fibres and short fibres would produce some interesting pulps good enough for low paper grades.

The CTP's High Yield Pulp Pilot plant facility can operate under industrial conditions and simulate a mill process. The results obtained on this pilot plant can be upscaled directly.

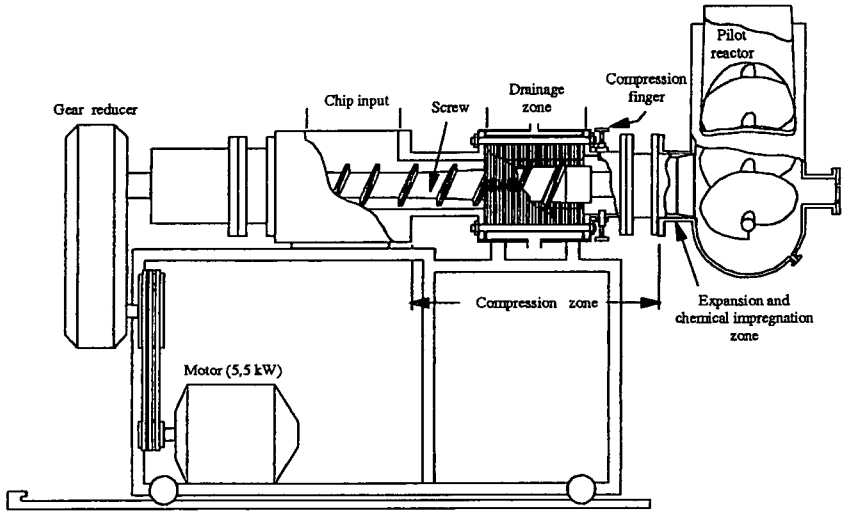
Pilot plant description

The High Yield Pilot Plant is described in figure n°1

Figure n°1



The Modular Screw Device 6" Pressafiner



A very flexible pilot plant combining plug screw, adjustable speed primary refiner and secondary refiner in line to simulate refiner mechanical pulping processes : RMP, TMP, CTMP, APP.

A laboratory facility operating under industrial conditions (continuously and under pressure) and producing high yield mechanical pulps having the characteristics of industrial pulps.

- Capacity : 10 to 40 kg per hour.
- Adjustable speed (1500 to 5000 rpm), 12 inches diameter disk refiner under pressure (motor power : 90 kW).
- Pressure : up to 5 bars.
- Chemical processing time after impregnation of raw material : 5 to 30 minutes.
- 12 inches diameter single disk refiner under pressure or at atmospheric pressure (motor power : 45 kW).
- Measurements of pH, pressure, power consumptions, steam consumption, dilution water flows, ... are followed by a data chain. Temperature and pressure are automatically regulated.
- Possibility of impregnation of the raw materiel in a plug screw feeder : the MSD 6" Pressafiner.

The MSD Pressafiner can be used alone or linked to the pilot plant reactor and therefore to all the pilot equipments.
For the simulation of new high yield mechanical pulping processes associating production of high quality pulps and respect of environment, a plug screw feeder, the MSD 6" Pressafiner (Modular Screw Device Pressafiner) allows to improve the impregnation of raw materials by chemicals. It ensures the following functions :

- Drainage of the chips with possibilities of residual recovery chemicals from the previous impregnation stages.
- Mechanical compression of the chips.
- Improvement of chemical absorption by the chips at the output of the compression zone.

XVII. TMP

Pulp preparation – Trial description

The whole jute chips (cutted at 2 to 5 cm long) were fed in the impregnation screw at 15 kg/h,
the steam pressure was maintained at 2 bar during about 5 minutes before the primary refiner.
The first stage pulp was blown to the secondary refiner.
During this trial, 4 pulps were produced at different freeness levels. The energy consumption were measured on the primary and secondary refiners. The results are given in table n°1.

Table n°1

Reference	1	2	3	4
Freeness, ml CSF	77	90	122	165
Primary refiner energy, kWh/t	940	830	790	630
Secondary refiner energy, kWh/t	1240	1040	1010	970
Total energy consumption kWh/t	2180	1870	1800	1600

These results were perfectly comparable with energy consumption measured during softwood TMP pulping on the pilot plant. As expected, specific energy .increased when freeness decreased
In figures 2, 3 and 4 are shown the energy consumption versus freeness for the 4 trials

Figure n°2

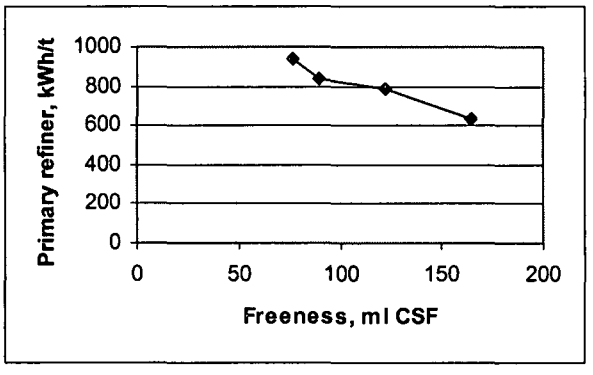


Figure n°3

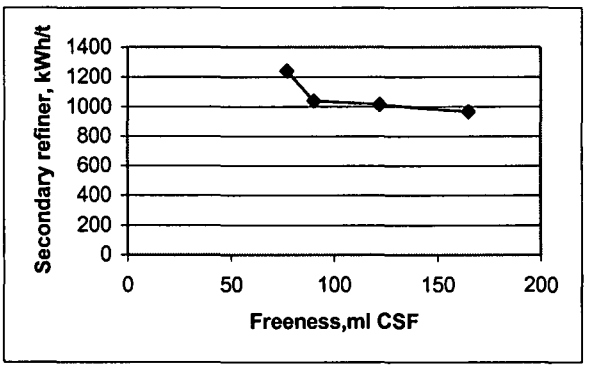
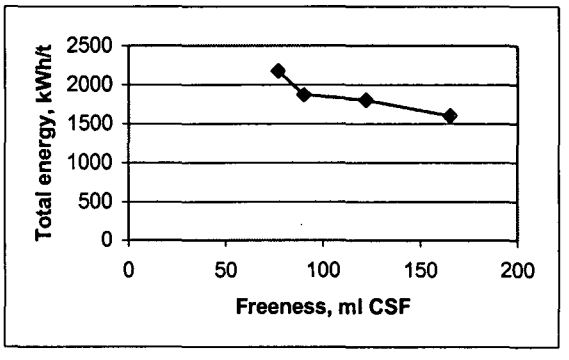


Figure n°4



Pulp analysis

a) Unbleached pulp physical properties

The pulp physical properties were measured on the 4 freeness levels after a pulp screening on the CTP pulper screener Lam'RCF equipped with 3 mm hole screen and 0.3 mm slot screen in order to remove the shives left in the pulps.
The results are given in table n°2

Table n°2

Reference	1	2	3	4
Freeness, ml CSF	77	90	122	165
Bulk, cm^3/g	3.01	2.98	3.06	3.09
Breaking length, km	1.25	1.16	1.36	1.29
Tear index, $mN.m^2/g$	1.51	1.47	1.67	1.89

In figure 5, 6 and 7 are shown the mechanical properties evolution versus freeness

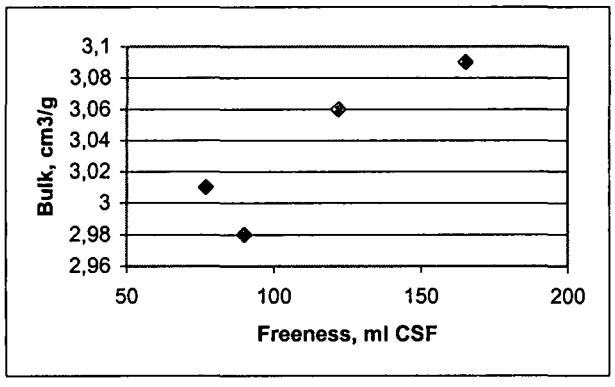


Figure n°5

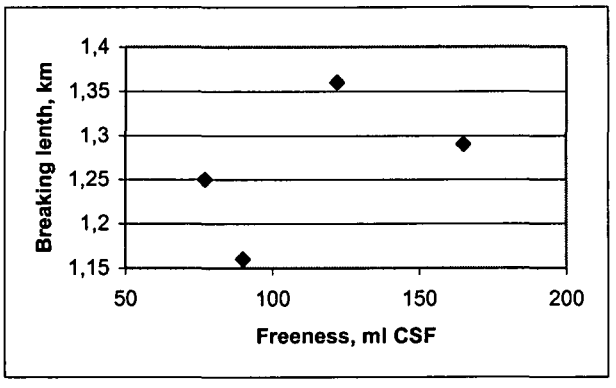


Figure n°6

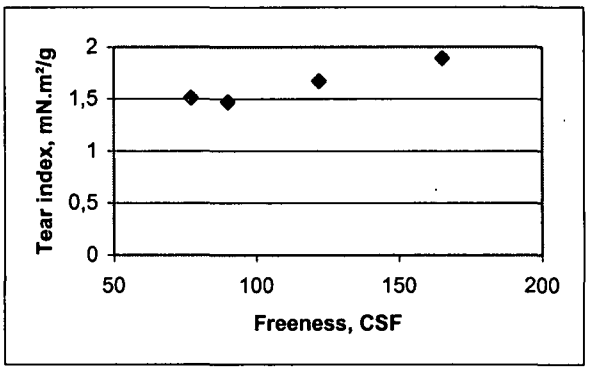


Figure n°7

The figure n°6 show that the breaking length was optimum around 100 – 120 ml CSF. These tensile and tear results were rather low. The bulk values were quite high. The high bulk value revealed that fibres were quite rigid. This explained partly the low physical properties.

This is also correlated with the weak tensile and tear.

b) Morphological analysis

The 4 samples were analyzed by the MORFI analyzer (table n°3).

Table n°3

Reference	1	2	3	4
Freeness, ml CSF	77	90	122	165
Length weighted fibre length, mm	0,515	0,576	0,578	0,672
Area weighted fibre length, mm	0,495	0,550	0,555	0,630
Width, µm	25,4	26,0	25,4	25,9
Coarseness, mg/m	0,348	0,341	0,335	0,314
Fines, % in length	82,1	77,9	78,4	76,7
Fines, % in surface	55,7	49,2	50,1	44,1
Shives, number/g	1388	1793	2366	1857
Shives, length, mm	1,77	0,91	1,08	1,42
Fibre length distribution % by class				
0.2 – 0.35 mm	39,0	31,5	32,0	26,0
0.35 – 0.5 mm	25,5	25,0	25,0	22,0
0.5 – 0.75 mm	21,0	24,0	25,0	26,0
0.75 – 1.00 mm	7,0	9,0	8,0	10,5
1.00 – 2.00 mm	6,5	9,5	10,0	13,0
2.00 – 3.00 mm	1,0	0,5	0,5	2,0

The mean fibre length was low, and comparable with a hardwood stone ground wood pulp.
The high fine contents and the short fibre length explained the weak physical properties.
The fibre length distribution indicate that the bast fibres contained in the bark were cutted, a few fibres were analyzed in the classes between 0.75 and 3.00 mm and no fibres were longer than 3 mm.
A lot of shives were present in the pulps even after screening. This indicated that fibre separation mechanisms were quite difficult for such a raw material

Bleaching trials

c) Preliminary trials

The bleaching trials were carried out on the pulp sample n°2.
A mechanical pulp suitable bleaching sequence with hydrogen peroxide was applied
The unbleached pulp brightness was 34.7 %ISO.
A first series of trials was devoted to find out the best bleaching conditions.
Regarding mechanical pulp bleaching, the hydrogen peroxide charge was fixed at 3 and 5%,
The sodium hydroxide charge was varied with regard to the final pH and the hydrogen peroxide consumption. As buffer and stabilizer, 4 % sodium silicate and 0.5 % DTPA were applied.

The temperature was fixed at 70°C, the reaction time : 120 minutes. A ERWEKA high consistency mixer allowed the mixing of the pulp at 28 %. The consistency was fixed at 28 %.
The 4 preliminary trials, with a totally negative results indicated that the mixing of the pulp and the chemicals led to a non homogeneous result without any bleaching effect. This could be due to the high shives contents consuming all the peroxide.

The consistency parameter was changed to 15 %.
A manual mixing seemed to be more appropriate and a preliminary chelating stage was tested. See table n°4.
The high consistency mixer was tested again on a 15% consistency pulp without any good results (a non homogeneous mixing with the chemicals). The study was carried on at 11% consistency.

Table n°4

Preliminary Chelation			0.5% DTPA
Consistency, %	15	15	15
H_2O_2 %	3	3	3
NaOH %	2	3	2
Silicate %	4	4	4
DTPA %	0.5	0.5	0.5
Final pH	8.0	8.3	7.2
Residual peroxide %	0	0	0.3
Brightness %ISO	42.3	47.6	57.2

These results indicated that a preliminary chelating stage was necessary and that the sodium hydroxide charge could be increased to a ratio H2O2/NaOH of 1/1. such as hardwoods, jute mechanical pulp seemed to need more alkali during bleaching

d) One stage bleaching trials

To carry out these bleaching trials the pulp samples were pretreated with a chelating agent in the following conditions :

Consistency : 5 %
Temperature : 70 °C
Time: 25 min.
DTPA : 0.5%

After the chelating stage the pulps were dewatered.
A new series of tests were carried out with 3 and 5 % Hydrogen peroxide and varying sodium hydroxide charges in the following conditions :

Consistency : 11 %
Temperature : 70 °C
Time: 120 min.

See table n°5

Table n°5

Trial number	14	11	12	13
H2O2, %	3	3	5	5
NaOH, %	2	3	4	5
Silicate, %	4	4	4	4
DTPA, %	0.5	0.5	0.5	0.5
Final pH	7.9	8.1	8.1	8.4
Residual peroxide, %	1.1	0.6	1.3	1.2
Initial brightness, %ISO	32.1	32.1	32.1	32.1
Final brightness, %ISO	48.4	51.9	59.4	62.6
Brightness gain, %ISO	16.2	19.8	27.3	30.5

It was interesting to note that it was possible to have very high brightness gain and if it is possible to increase the initial brightness level the brightness target of 80 %ISO would be approached. The poor initial brightness was due to the bark color.

A presteaming of the chips and the pass through the pressafiner or extracting part of the bark in the effluent of a screw press or a bivis machine or with appropriate chemical or biological treatment would lead to better results.
This raw material had a behavior comparable to a hardwood in term of sodium hydroxide consumption, during bleaching.
The mechanical properties were measured in order to evaluate the effect of the bleaching stage. See table n°6

Table n°6

Reference	2	13
	Unbleached	Bleached
Freeness, ml CSF	90	90
Bulk, cm^3/g	2,98	2,45
Breaking length, km	1,16	1.85
Tear index, $mN.m^2/g$	1,47	2.11

These results showed an important improvement of the mechanical properties. The bleached pulp mechanical properties were comparable with a SGW softwood pulp. But they were always too low for newsprint production. A reinforcement of this pulp with a chemical softwood pulp will be necessary for an appropriate paper production.

e) Enzymatic pretreatment before the P stage

Three xylanases were tested during this trial series :
ECOPULP TX 200C from AB Enzymes (Germany)
PULPZYME from NOVOZYMES (Denmark)
BIOBRITE from IOGEN (Canada)
As previously the pulp samples were preliminary chelated.
A standard P bleaching stage and a 2 stages P sequence were carried out to compare the results with and without enzymatic treatment. See table n° 7
These results showed that it was possible to increase the sodium hydroxide charge to 6% for 5 % hydrogen peroxide applied with an improvement of the brightness

(62.6 %ISO in table 5 for 5% NaOH and 5 % H_2O_2 and 66.1 %ISO for 6 % NaOH and 5 % H_2O_2).
The enzyme treatment showed a slight improvement of the unbleached brightness (about 5 to 5.5 %ISO). The 3 enzymes tested led to the same results.
The one P stage bleaching sequence led to 34 % brightness gain for the reference and 37 – 37.5 % with xylanase pretreatment.
An increase in time (3 hours instead of 2 hours) increased the brightness (68.9 to 69.7 %ISO) for the bleaching.
An other repartition of the H2O2 and NaOH charges increased the brightness result (68.9 to 71.8 %ISO)
The two P stage bleaching sequences led to a better brightness for the reference with 37 % brightness gain but the effect of the enzyme pretreatment was lower with 38 % gain.
The enzyme pretreatment was efficient on the one P stage bleaching, however the improvement was low (3 %ISO).
The brightness of 70 %ISO was reached.

f) Mechanical properties
g)

The mechanical properties were measured on the single and 2 P stage bleached pulps without and with enzyme pretreatment. See table n°8

Table n°8

				Enzyme (Pulpzyme)	
	Unbleached	1 P stage	2 P stages	1 P stage	2 P stages
Bulk, cm3/g	2.98	2.41	2.13	2.25	2.09
Breaking Length, km	1.16	2.02	2.57	2.29	2.58
Burst index, kPa.m²/g		0.92	1.20	1.08	1.23
Tear index, mN.m²/g	1.51	2.49	2.85	2.70	2.83
Opacity, %ISO		96	94.1	94.4	93.4
Absorption coefficient K, m²/kg		1.84	1.21	1.23	1.07
Scattering coefficient S, m²/kg		60.2	57.6	58.8	57.1

The bleaching stages improved considerably the mechanical properties due to the high sodium hydroxide charge applied. The two stages P bleaching led to slightly better results than one P stage. The enzyme pretreatments had a small incidence especially on the one stage P bleaching.
Despite of a strong increase in the mechanical properties, the results were weak and this pulp needed a reinforcement pulp to be use in news print paper making.

However the optical properties were rather good especially the opacity and scattering

Table n°7

Bleaching optimization without and with enzymatic treatment												
References	C02/1.2/20	C02/1.2/21	C02/1.2/22	C05/1.2/29	C05/1.2/30	C05/1.2/31	C02/1.2/23	C02/1.2/26	C02/1.2/24	C02/1.2/27	C02/1.2/25	C02/1.2/28
CHELATION STAGE DTPA												
Consistency, %	**5**											
Temperature, °C	**70**											
Time, min	**25**											
DTPA, %	**0.5**											
TRAITEMENT ENZYMATIQUE												
Consistency, %							**5**					
Temperature, °C							**50**					
Time, min							**120**					
ECOPULP TX 200C 158g/100kg pâte							**158mg/100g**					
PULPZYME 1000U/g									**100mg/100g**			
BIOBRIGHT 3595U/ml											**10U/g**	
initial pH							6.96		7.09		7.12	
final pH							6.71		6.74		6.6	
Blancheur, %ISO							**37.6**		**37.4**		**37.3**	
P Stage or P1 (APMP)		**"APMP"**										
Consistency, %	10	10	10	10		10	10	10	10	10	10	10
Temperature, °C	70	70	70	70		70	70	70	70	70	70	70
Time, min	120	120	120	120		120	120	120	120	120	120	120
H2O2, %	5	1	1	1		2	5	1	5	1	5	1
NaOH, %	6	3	4	4		4	6	4	6	4	6	4
Silicate, %	4	4	4	4		4	4	4	4	4	4	4
DTPA, %	0.5	0.5	0.5	0.5		0.5	0.5	0.5	0.5	0.5	0.5	0.5
pH final	8.5	8.61	8.95	9.08		8.64	8.65	9.17	8.62	9.08	8.45	9.03
H2O2 consumed, %	4.78	1	1	1		2	4.66	1	4.66	1	4.69	1
Brightness, %ISO	**66.1**	**41.5**	**40.9**	**40.6**		**49.3**	**69.2**	**43**	**69.6**	**42.9**	**69.2**	**42.9**
Brightness pH 5				**42.1**		**50.8**						
P2 STAGE(APMP)												
Consistency, %		10	10	10	10	10		10		10		10
Temperature, °C		70	70	70	70	70		70		70		70
Time, min		120	120	120	180	120		120		120		120
H2O2, %		4	4	4	4	3		4		4		4
NaOH, %		2	2	2	2	2		2		2		2
Silicate, %		4	4	4	4	4		4		4		4
DTPA, %		0.5	0.5	0.5	0.5	0.5		0.5		0.5		0.5
final pH		8.22	8.88	8.85	8.46	8.91		9.16		9.04		9.07
H2O2 consumed, %		2.56	2.68	2.77	2.88	1.88		2.39		2.47		2.39
Brightness, %ISO		**68.5**	**69**	**68.3**	**69.6**	**71.8**		**70.4**		**70.2**		**70**
Brightness pH 5				**68.9**	**69.7**	**71.8**						

XVIII. APMP FEASIBILITY

APMP1

A APMP trial was carried out on the High yields pilot plant to compare with the TMP process.

The jute chips were first impregnated with water during one night and presteamed before the trial.

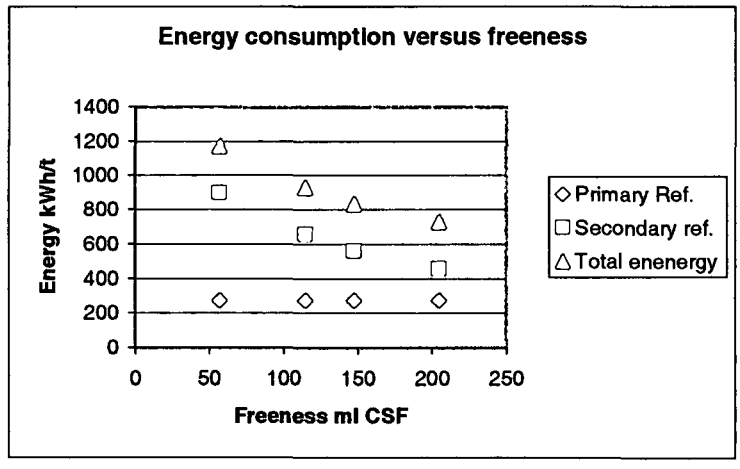
a) Trial description

The Pilot plant was fed through the MSD pressafiner to the impregnation screw. The first stage liquor was introduced at the bottom of the screw. The expected chemicals applied were 4 % NaOH , 1% H2O2 , 4% Silicate and 0.5% DTPA. The temperature was maintained at 80°C and the pressure 2 bar with pressurized air before the 1st refining stage.

The energy consumption was measured on the primary and on the secondary refiner. The results are given in table n°9

Table n°9

Reference	21	22	23	24
Freeness, ml CSF	57	115	148	205
Primary refiner energy, kWh/t	271	271	271	271
Secondary refiner energy, kWh/t	899	659	562	458
Total energy consumption kWh/t	1170	930	830	730



The total energy consumption showed an important decrease compared to the TMP pulp(800 kWh/t instead of 1700 for 150 ml CSF). The effect of the sodium hydroxide charge led to a low energy consumption at the primary refiner, the fibre separation was helped by dissolution of some wood components. The refining energy was also considerably decreased because of the fibrillation of the fibres instead of a cutting effect

The pulp was washed after the 1st refining stage and refined at 4 freeness levels to evaluate the mechanical properties. The 2nd P stage bleaching was carried out at laboratory scale.

The unbleached mechanical properties are given in table n°10 and illustrated in figure n° 9 and 10

During the feeding in the pressafiner a dark liquor was extracted from the raw material (mainly bark particles and water soluble extracts).
The COD measured on the pressafiner effluent was 22.8 kg/t
The COD measured on the pulp effluent was 390 kg/t revealing an important dissolved matter and then a decreased yield correlated with the mechanical properties increase

Table n°10

Reference	1	2	3	4
Freeness, ml CSF	57	115	148	205
Bulk, cm^3/g	1.45	1.47	1.55	1.53
Breaking length, km	6.72	6.40	6.59	6.13
Tear index, $mN.m^2/g$	5.12	5.64	6.32	6.25

Figure n°9

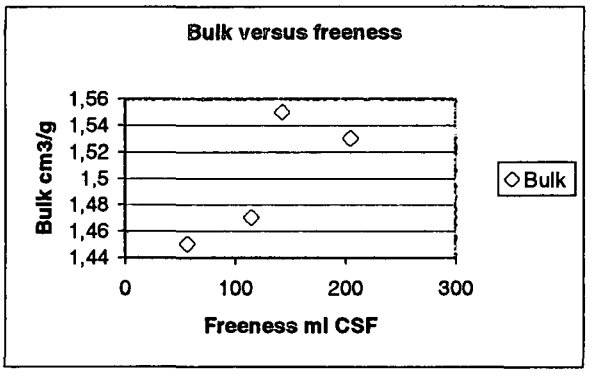
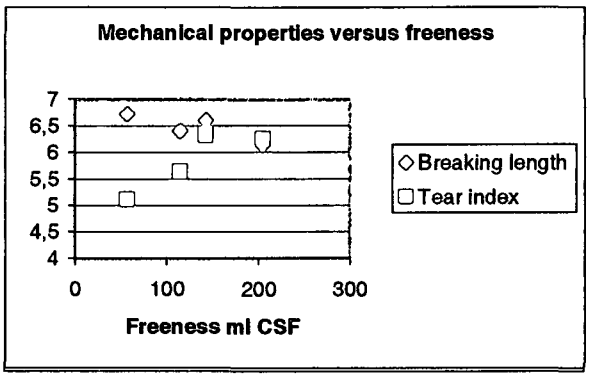


Figure n°10



These so high mechanical property values and the behavior of the pulp after the primary refiner led us to check the absorption rate of the chips after the pressafiner. The absorption was so high that three time chemicals were applied on the raw material leading to 12% NaOH and 3% H2O2 in the first stage.

Such an amount of chemicals decreased the pulp yields improving the pulp mechanical properties.

b) Morphological analysis

Table n°11

Reference	21	22	23	24
Freeness, ml CSF	57	115	148	205
Length weighted fibre length, mm	0.753	0.806	0.824	0.855
Area weighted fibre length, mm	0.735	0.770	0.780	0.820
Width, µm	25.7	25.9	26.2	26.0
Coarseness, mg/m	0.208	0.206	0.206	0.210
Fines, % in length	52.4	51.1	49.8	48.6
Fines, % in surface	24.9	23.0	21.3	21.4
Shives, number/g	10890	8640	10570	12520
Shives, length, mm	1.60	1.66	1.77	1.52
Fibre length distribution % by class				
0.2 – 0.35 mm	13.15	11.45	11.75	10.4
0.35 – 0.5 mm	17.65	16.2	16.1	15.65
0.5 – 0.75 mm	33.4	33.6	33.35	33.05
0.75 – 1.00 mm	19.25	19.75	19.45	18.40
1.00 – 2.00 mm	13.8	15.75	15.55	17.35
2.00 – 5.00 mm	3.55	3.25	3.8	5.2

As previously remarked on the energy consumption the morphological analysis indicated an increase in the fibre length meaning that the fibres were not damaged during the pulping compared to a TMP pulp. The coarseness decrease showed a dissolution of part of the material meaning a decrease in the yield.
In spite of this wrong chemical rate, bleaching trials were carried out on the pulp. Sample n°2 with 115 ml CSF was selected for the bleaching tests The bleaching results are given in table n°12.

The brightness results were lower than for the TMP test but reached 67%ISO which was closed to the results obtained during the TMP pulp tests.
It was to note that a preliminary chelating treatment was not necessary and led to slightly lower results.
The enzyme treatments led to slight improvement of the brightness (1.3 to 3%)
These interesting and unexpected brightness results obtained were probably due to the extraction of bark and water soluble extracts which generally consume chemicals and generate poor unbleached brightness results.
The high pH measured on the enzyme pretreatment would have been adjusted to a neutral or slightly acidic medium to improve the enzyme effect.

c) Mechanical properties

The mechanical properties were measured on the bleached pulp without chelating treatment
The results are given in table n° 13

Table n°13

Reference	Unbleached pulp	Reference bleaching	Ecopulp	Pulpzyme	Biobright
Bulk cm^3/g	1.47	1.68	1.61	1.67	1.58
Breaking length km	6.40	6.00	5.51	5.34	5.62
Burst index	5.64	4.70	4.86	5.37	5.32

A decrease in the mechanical properties was observed comparing the bleached pulp and the unbleached one. The enzyme pretreated pulps gave also lower results. This was probably due to the modifications of the fibre repartition after diverse inter stages washing.

Table n°12

CHELATION STAGE DTPA												
Consistency %	**5**								**5**			
Temperature °C	**70**								**70**			
Time min	**30**								**30**			
DTPA %	**0.5**								**0.5**			
ENZYMATIC TREATMENT												
Consistency %			**5**						**5**			
Temperature °C			**50**						**50**			
Time min			**120**						**120**			
ECOPULP TX 200C 158g/100kg pulp			**158mg/100g**						**158mg/100g**			
PULPZYME 1000U/g					**100mg/100g**						**100mg/100g**	
BIOBRIGHT 3595U/ml							**10U/g**					**10U/g**
initial pH												
final pH			10.3		10.3		10.3		10.1		10.1	10.2
Brightness %ISO			**40**		**40.5**		**39.7**		**41**		**40.1**	**40.1**
Brightness at pH 5												
P2 STAGE (APMP)												
Consistency %	10	10	10		10		10		10		10	10
Temperature °C	70	70	70		70		70		70		70	70
Time min	120	120	120		120		120		120		120	120
H2O2 %	4	4	4		4		4		4		4	4
NaOH %	2	2	2		2		2		2		2	2
Silicate %	4	4	4		4		4		4		4	4
DTPA %	0.5	0.5	0.5		0.5		0.5		0.5		0.5	0.5
final pH	10.7	10.6	10.3		10.1		10.2		10.5		10.4	10.6
H2O2 consumed %	2.1	2.05	2.3		2.3		2.2		2.3		2.3	2.3
Brightness %ISO	**61.6**	**59.1**	62.6		**63.5**		**63.5**		**62**		**62.8**	**63.8**
Brightness at pH 5	**61.8**	**64**	**65.3**		**66.5**		**67**		**64.1**		**64.6**	**65**

Initial brightness 37.5

APMP 2

After checking the absorption capacity of the material after the pressafiner, an other test was carried out with the right amount of chemicals (4% NaOH, 1% H_2O_2) in the conditions previously described on the jute chips

.

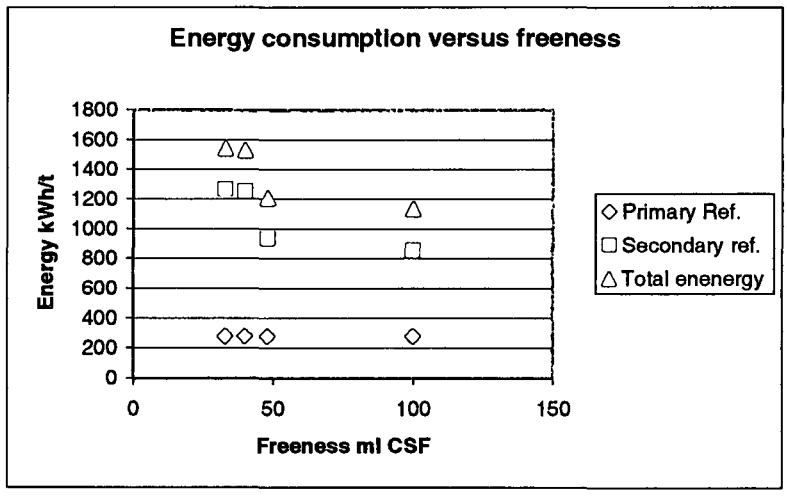
The pulp was collected after the primary refiner. And refined separately at 4 freeness levels.

The results are given in table n°14 and illustrated in figure n°11

Table n°14

Reference	31	32	33	34
Freeness, ml CSF	33	40	48	100
Primary refiner energy, kWh/t	276	276	276	276
Secondary refiner energy, kWh/t	1267	1254	931	857
Total energy consumption kWh/t	1543	1530	1207	1133

Figure n°11



The total energy consumption was intermediate between TMP pulp and the first APMP test

The COD measured on the pressafiner effluent was 22.8 kg/t
The COD measured on the pulp effluent was 60 kg/t
This low COD value measured on the pulp revealed a lower effect of the chemicals applied on the impregnation stage compared with the previous APMP trial.
However this amount of COD needed to be treated

d) Morphological analysis

Table n°15

Reference	31	32	33	34
Freeness, ml CSF	33	40	48	100
Length weighted fibre length, mm	0.587	0.643	0.648	0.695
Area weighted fibre length, mm	0.585	0.635	0.640	0.680
Width, µm	26.3	26.9	26.4	27.0
Coarseness, mg/m	0.243	0.244	0.247	0.242
Fines, % in length	65.85	65.2	63.25	61.25
Fines, % in surface	33.68	31.48	29.80	27.43
Shives, number/g	2804	3800	2549	5594
Shives, length, mm	1.23	1.10	1.63	1.521.27
Fibre length distribution % by class				
0.2 – 0.35 mm	24.15	20.25	18.25	16.95
0.35 – 0.5 mm	24.15	22.45	21.95	20.00
0.5 – 0.75 mm	30.1	31.75	33.30	32.40
0.75 – 1.00 mm	11.95	13.25	14.55	15.30
1.00 – 2.00 mm	8.4	10.25	10.60	13.00
2.00 – 5.00 mm	1.3	2.0	2.0	2.35

The comparison of the pulps at about 100 ml CSF showed that that the impregnation stage led to an increase of the fibre length. The decrease of coarseness indicated a yields drop.
This pulp analysis led to intermediate result between TMP and the previous APMP pulps.

e) Mechanical properties analysis

The mechanical properties was measured on the pulp after the impregnation stage. The results are given in table n°16 and illustrated in figure n° 12 and 13

Table n° 16

Reference	31	32	33	34
Freeness, ml CSF	33	40	48	100
Bulk, cm^3/g	1.52	1.50	1.55	1.64
Breaking length, km	5.99	5.90	5.75	5.33
Tear index, $mN.m^2/g$	2.88	3.12	3.24	3.63

These mechanical properties were improved by the impregnation stage and led to values suitable for news print paper manufacturing.

The bleaching step was carried out on the 100 ml CSF freeness pulp. The results are given in table n°17

Figure n°12

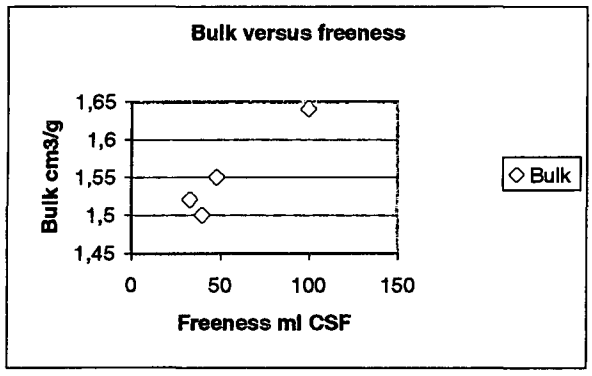


Figure n° 13

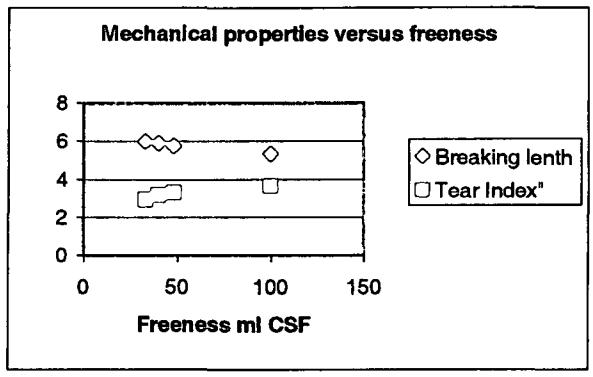


Table n° 17

	A	B (I)	C	*F*	D	*G*	E	*H*
DTPA								
DTPA, %		0,5	0,5	*0,5*	0,5	*0,5*	0,5	*0,5*
Consistency, %		5	5	*5*	5	*5*	5	*5*
Temperature, °C		70	70	*70*	70	*70*	70	*70*
Time, min.		25	25	*25*	25	*25*	25	*25*
				Washing		*Washing*		*Washing*
ENZYME								
ECOPULP TX 200C			158mg/100g	*158mg/100g*				
PULPZYME 1000 U/g					100mg/100g	*100mg/100g*		
BIOBRIGHT 3595 U/ml							10 U/g	10 U/g
Consistency, %			5	*5*	5	*5*	5	*5*
Temperature °C			50	*50*	50	*50*	50	*50*
Time min,			120	*120*	120	*120*	120	*120*
Initial pH			9,3	*6,3*	9,3	*6,5*	9,3	*6,6*
Final pH			9	*8,2*	9	*7,4*	8,9	*8,1*
H2O2 Stage								
H2O2, %	4	4	4	*4*	4	*4*	4	*4*
Na OH, %	2	2	2	*2*	2	*2*	2	*2*
Silicate, %	4	4	4	*4*	4	*4*	4	*4*
DTPA, %	0,5	0,5	0,5	*0,5*	0,5	*0,5*	0,5	*0,5*
Consistency, %	10	10	10	*10*	10	*10*	10	*10*
Temperature °C	70	70	70	*70*	70	*70*	70	*70*
Time min,	120	120	120	*120*	120	*120*	120	*120*
Initial pH			10,4	*10,8*	11	*10,8*	10,9	*10,8*
Final pH	10,8	10,35	10,45	*10,1*	10,6	*10,3*	10,5	*10,3*
H2O2 consumption, %	99,3	81,7	89	*83*	84,3	*70*	89,6	*82*
Brightness %ISO	**45**	**60**	**57,5**	***64,2***	**57,2**	***64,5***	**58,7**	***63,5***
Brightness %ISO at pH 5		61,8	59	*66*	58,6	*66,5*	59,4	*65,3*

These bleaching trials showed that a preliminary chelation stage was necessary, that was not the case in the previous APMP trial. During the refining, some iron ions can be transferred to the pulp leading to hydrogen peroxide decomposition .
The effect of the chelation stage was remarked on the reference bleaching tests (A and B) with a lower hydrogen peroxide consumption and a better brightness.

The enzyme pretreatment conducted without pH adjustment did not generate interesting results but when the pH was adjusted to pH 6 – 7 the brightness results were improved by 4 units. An acidification of the pulp after bleaching improved the brightness with 2 units more.

The final pulp brightness achieved at 66 %ISO. This values were slightly lower than the TMP bleached pulp, however it is suitable for news print paper production.

f) Mechanical properties

Mechanical properties were measured on the best bleached pulps, the results are given in table n°18

Table n°18

Reference	Unbleached pulp	Reference bleaching	Ecopulp	Pulpzyme	Biobright
Bulk cm^3/g	1.64	1.66	1.61	1.63	1.64
Breaking length km	5.33	5.37	5.62	5.50	5.38
Tear index	3.63	4.34	4.40	4.72	4.81
Opacity		91.6	91.5	90.2	90.3

A slight increase in the physical properties was observed on the bleached pulp, especially the tear index. The enzyme pretreatment did not have influence.
The opacity were rather good and no changes were observed with enzyme treatment.

Conclusion

It was possible to produce TMP pulp using whole jute. The bleaching with hydrogen peroxide reached 70 %ISO brightness with 5 % Hydrogen peroxide and 6% sodium hydroxide.
Whenever after the bleaching sequence, the pulp mechanical properties were still low and the pulp would need reinforcement pulp to be used in newsprint paper manufacturing.
The TMP pulp preparation led to short fibres. The relatively high energy consumption cut drastically the bast fibres.
Prior to bleaching a chelating stage was necessary. In case of a single P stage applying all the chemical charge, the enzyme pretreatment was slightly efficient (about 3 brightness unit gain). In case of an "APMP" bleaching trial, enzyme pretreatment was not interesting. The repartition of Hydrogen peroxide and sodium hydroxide could be optimized in the first and second stage. Perhaps, because alkali removed the xylans in the pulp during impregnation.
APMP pulp was produced on the TMP CTP pilot plant processing a preliminary impregnation before the primary refiner and bleaching after the secondary refiner.

A first pulp produced with a high chemical charge led to interesting results in term of mechanical properties and bleachability.
The application of enzyme as pretreatment before the bleaching stage had a poor efficiency. The alkalinity left in the pulp increased the pH treatment to 10 which was not really suitable for enzyme treatment.
A second APMP pulp was prepared with the jute chips left with the right chemical rate impregnation. The pulp obtain was more difficult to bleach, however with a chelating stage the final brightness achieved at 66 – 67%ISO with good mechanical properties.
The pretreatment in the APMP process inducing a slight dissolution of compounds led to a lower need in energy to produce the pulp and then improve the fibre properties.

Some more trials would have to be conducted in APMP pulping to confirm the present results and to optimize the chemicals for achieving mechanical properties suitable for news paper manufacturing and a brightness target of 60 – 65 %ISO.

XIX. APMP OPTIMIZATION

Regarding the previous results obtained on the APMP process applied on the whole jute, it was interesting to optimize the chemical charges to reach 60 – 65%ISO brightness as a target and appropriate mechanical properties for news-print paper manufacture.

Trial description

The jute chips were first impregnated with water during one night and presteamed before the trial at atmospheric pressure.

The pilot plant was fed through the MSD pressafiner to the reactor screw. The first stage liquor was introduced at the bottom part of the screw. The chemical charges were introduced based on the material absorption at the out put of the MSD pressafiner.
The impregnation liquor concentration was calculated based on the total liquor absorbed by the chips after the press.
The reactor was pressurized to 2 bar with pressurized air, the temperature was maintained at 80 °C and the reaction time was 20 minutes.
The impregnated jute was defibered in a primary refiner equipped with D2 505 pattern discs(12-inch diameter). The rotation speed was 3500 rpm. After the primary refiner, the pulps were blown in a cyclone and stored before refining tests with a Sprout Waldron 12-inch equipment with C 2976 discs.
Three chemical charges were tested in the impregnation step and four freeness levels were reached to evaluate the pulp quality.
The energy consumption was measured on the primary and on the secondary refiner.

The trial conditions and the main mechanical properties are given in table 19 for trials 1, 2 and 3

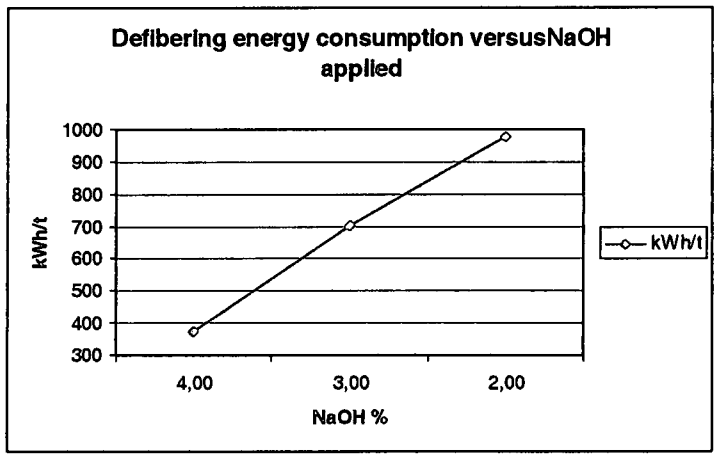
Table n°19

Trial n°	NaOH 4%				NaOH 3%				NaOH 2%			
Freeness, CSF	60	115	180	257	68	108	170	245	73	98	136	250
Trial conditions												
Temperature, °C	80	80	80	80	80	80	80	80	80	80	80	80
Time, mn	20	20	20	20	20	20	20	20	20	20	20	20
Primary refiner speed ,rpm	3500	3500	3500	3500	3500	3500	3500	3500	3500	3500	3500	3500
Chips dry matter content, %	33,30				30,91				32,00			
Defibered pulp dry matter content,	17,64	17,64	17,64	17,64	17,80	17,80	17,80	17,80	24,07	24,07	24,07	24,07
Defibering energy, kWh/t	372	372	372	372	701	701	701	701	975	975	975	975
Refining energy, kWh/t	1217	1145	1099	759	1171	1155	1133	1019	1262	1256	1098	842
MSD energy, kWh/t	100	100	100	100	100	100	100		100	100	100	100
Total energy consumption, kWh/t	1689	1617	1571	1231	1972	1956	1934	1720	2337	2331	2173	1917
Chemical charges												
NaOH, % - g/l	4 - 23				3 - 17.3				2 - 11.5			
H_2O_2, % - g/l	1 - 5.8				1 - 5.8				0.5 - 2.9			
Sodium silicate, %												
DTPA, %												
Mechanical properties												
Freeness, CSF	60	115	180	257	68	108	170	245	73	98	136	250
Bulk, cm^3/g	1,36	1,39	1,46	1,56	1,33	1,37	1,43	1,46	1,46	1,49	1,51	1,61
Tensile index, Nm/g	71,2	66,3	65,3	62,9	78,6	72,7	66	62,9	67,8	64,4	64,7	57,7
Breaking length, m	7258	6758	6659	6413	8010	7413	6727	6411	6914	6562	6596	5881
Tear index, $mN.m^2/g$	4,7	5,33	5,9	6,29	4,74	5,69	5,97	6,45	5,9	6,03	6,34	6,62

Energy consumption

The defibering step was conducted in order to get the same freeness level of about 800 ml CSF for the three chemical charges. The energy consumption in the defibering step versus the NaOH charge is illustrated in figure n°14.

Figure n°14



This figure underline that the energy consumption was well correlated with the sodium hydroxide rate applied. Sodium hydroxide induced an important effect on the jute stick softening, leading to an easier fibre separation during the defibering.

Figures 15, 16 and 17 illustrate the energy consumption versus freeness.

Figure n°15

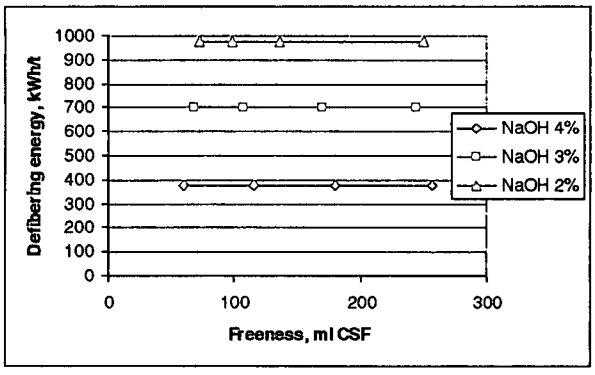


Figure n°16

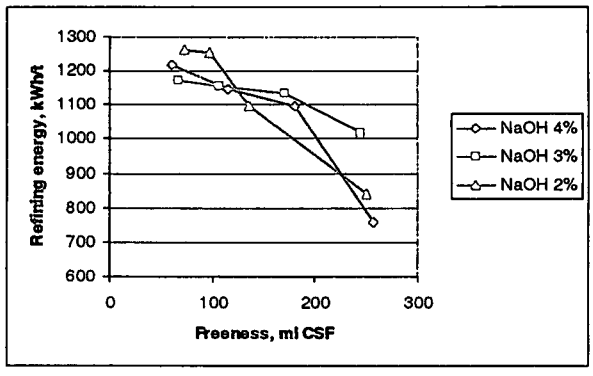


Figure n°17

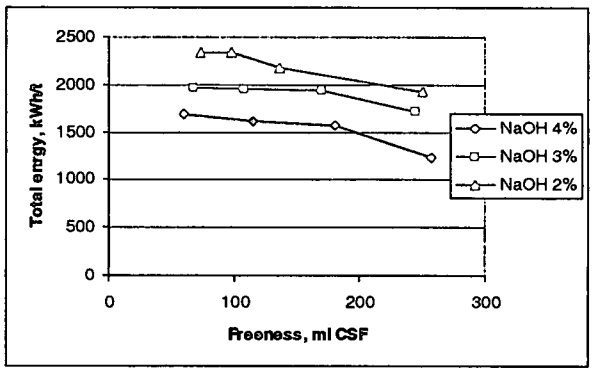


Figure n°16 underline that the refining energy consumption was similar for the 3 trials. Total energy consumption was well correlated with the sodium hydroxide rate applied and the difference come from defibering behaviour (figure n°17).

Mechanical properties

Figure 18, 19and 20 illustrate the main mechanical properties versus freeness respectively, bulk, breaking length and tear index.

Figure n°18

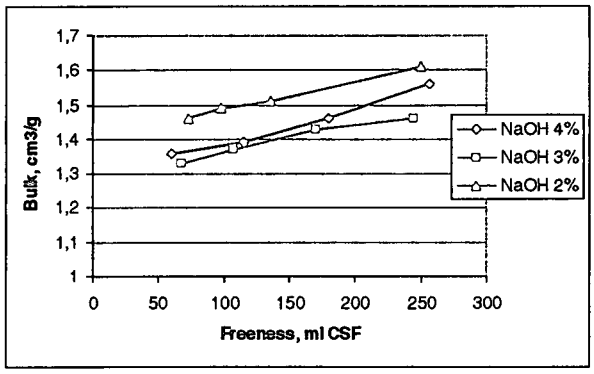


Figure n°19

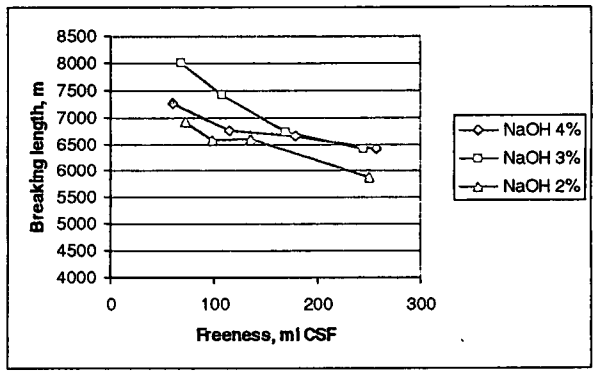
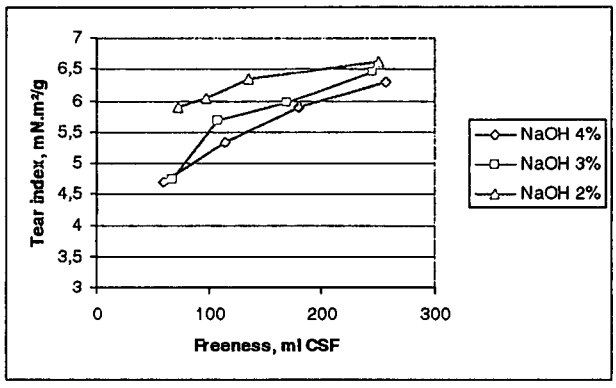


Figure n°20



Bulk was higher for the 2% NaOH trial, indicating more rigid fibres. Breaking length was slightly lower for the 2% NaOH trial but the tear index was better than the others. The pulps obtained with 3% NaOH and 1% H_2O_2 (proportionally more than the other trials 4/1 and 2/0. 5) seemed to have slightly better properties, especialy breaking length, indicating that the fibres were more delignified and more flexible. The internal cohesion improved by the hydrogen peroxide reaction generated more hydrogen bonds between fibers.

Morphological analysis

The pulps were analysed by the Morfi analyzer (table n°20).

Table n°20

Trial n°		1				2				3			
Freeness, CSF		60	115	180	257	68	108	170	245	73	98	136	250
Number of fibres analysed		9684	8599	8557	8691	8115	8262	8733	8319	8155	8290	8133	7526
Length pondered in length, mm		0,635	0,752	0,777	0,821	0,729	0,745	0,794	0,832	0,762	0,797	0,815	0,859
Length pondered in area, mm		0,630	0,730	0,745	0,775	0,710	0,720	0,765	0,795	0,735	0,765	0,785	0,820
Length arithmetical, mm		0,496	0,565	0,579	0,602	0,556	0,568	0,596	0,616	0,583	0,598	0,609	0,632
Width, µm		26,5	26,8	26,7	26,5	26,0	26,1	26,1	26,1	25,8	25,6	25,7	25,7
Coarsness, mg/m		0,150	0,149	0,149	0,143	0,161	0,153	0,142	0,142	0,155	0,149	0,147	0,157
Kink angle, °		124,5	128,5	127,5	130,5	127,0	130,5	132,0	132,5	130,0	131,0	129,0	128,5
Kinked fibres, %		14,70	10,65	10,05	10,40	12,70	10,90	9,80	10,35	10,60	12,15	10,70	9,90
Nb of kink by fibres		1,12	1,10	1,12	1,11	1,11	1,11	1,12	1,10	1,10	1,12	5,72	1,14
Courbure, %		5,85	4,90	4,75	4,70	5,20	4,70	4,55	4,55	4,60	4,90	4,75	4,70
Repartition by class in length, %													
	0,20 - 0,35	19,20	14,05	13,00	12,30	14,55	13,40	11,95	11,45	12,90	12,15	11,50	10,35
	0,35 - 0,50	23,30	19,50	18,75	18,15	18,80	19,00	17,50	16,70	17,35	16,95	16,30	16,20
	0,50 - 0,75	32,55	34,15	33,85	32,70	34,70	34,95	35,00	33,80	34,75	33,75	33,85	33,10
	0,75 - 1,00	13,40	15,15	16,20	16,90	15,85	16,75	17,45	17,40	18,00	18,10	18,60	18,40
	1,00 - 2,00	10,15	13,55	14,70	15,90	13,80	13,40	14,60	16,45	14,30	15,20	15,85	17,10
	2,00 - 3,00	1,20	3,30	3,20	3,20	2,15	2,35	3,20	3,75	2,55	3,50	3,40	4,30
	3,00 - 5,00	0,25	0,25	0,30	0,80	0,20	0,15	0,40	0,40	0,20	0,40	0,50	0,50
	5,00 - 7,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
	7,00 - 10,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
	> 10,0	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
Fines, % in length		58,9	53,4	51,7	48,2	54,8	51,6	48,1	46,1	51,9	50,2	48,7	46,4
Fines, % in area		23,5	19,9	20,0	19,1	21,9	20,6	18,3	17,7	20,2	18,8	18,8	18,5

The morphological analysis did not show significant differences. The mean fibre lengths were comparable to a softwood mechanical pulp, however the fibre repartition by class indicated the presence of long fibres (classes 1 to 2 mm and 2 to 3 mm).Tthis was interesting for the tear index and consequently for a good paper machine runnability. It would not be necessary to use reinforcement pulps.

XX. BLEACHING

Bleaching conditions and results

As seen in the previous study, an enzyme (Xylanase) pretreatment before bleaching was not sufficiently efficient to improve the brightness. The bleaching trials were carried out with a single P stage after a chelation stage with DTPA.
The bleaching trials were carried out on the pulps refined at about 100 ml CSF.
The bleaching conditions and results are given in table n° 21.

		JUTE 1 115 ml CSF		JUTE 2 108 ml CSF		JUTE 3 98 ml CSF	
consistency 5% / 70 °C / 30 min,	DTPA %	0.5					
CHELATION DTPA	pH final	8.7		7.9		9	
	Brightness, % ISO	36.3		45.7		39.3	
consistency10% / 70 °C / 120 min	H_2O_2 %	3	4	2	3	2	3
	NaOH %	1.5	2	1	1.5	1	1.5
	Silicate %	4	4	4	4	4	4
	DTPA %	0.5	0.5	0.5	0.5	0.5	0.5
Hydrogen peroxide bleaching							
	Final pH	10.1	10.3	9.9	10.2	9.7	10
	consumed H_2O_2 %	1.74	2.43	1.24	1.76	1.1	1.58
	Brightness, % ISO	55.9	56.4	57.6	59.2	57.4	60.8
	Brightness at pH 5	59.3	59.9	61.7	64.3	59.6	62.5

Table n°21

Table 21 shows that the higher the sodium hydroxide charge in the impregnation, the higher the peroxide charge needed to bleach the pulp. The sodium hydroxide reacted on the lignin chromophorous groups, destroyed by the hydrogen peroxide oxidation.
Pulp n° 2 with 3% NaOH and 1% H_2O_2 in the impregnation stage led to better results.
It seems that the ratio NaOH/H_2O_2 had a significant effect.
The brightness target of 60-65 %ISO was achieved.

Mechanical properties

The mechanical properties were analyzed on the better brightness obtained on the 3 Impregnations. The results are given in table n°22

Table n°22

First step : Impregnation, defibering, refining												
	APMP 1				APMP 2				APMP3			
Impregnation	NaOH 4% - H_2O_2 1%				NaOH 3% - H_2O_2 1%				NaOH 2% - H_2O_2 0.5%			
Freeness, ml CSF	60	115	180	257	68	108	170	245	73	98	136	250
Bulk, cm^3/g	1.36	1.39	1.46	1.56	1.33	1.37	1.43	1.46	1.46	1.49	1.51	1.61
Tensile, Nm/g	71.2	66.3	65.3	62.9	78.6	72.7	66	62.9	67.8	64.4	64.7	57.7
Breaking length, km	7.26	6.76	6.66	6.4	8.01	7.41	6.73	6.41	6.91	6.56	6.6	5.88
Tear index, $mN.m^2/g$	4.7	5.3	5.9	6.3	4.7	5.7	6	6.5	5.9	6	6.3	6.6
Second step : Bleaching												
Bulk, cm^3/g		1.42				1.4				1.46		
Tensile, Nm/g		67.3				73.6				68.3		
Breaking length, km		6.86				7.5				6.96		
Tear index, $mN.m^2/g$		5.94				6.56				6.49		
Brightness at pH 5		59.9				64.3				62.5		
Opacity, %ISO		89.8				87.5				89.3		
Absoption coefficient K, m^2/kg		1.14				1.13				1.12		
Scattering coefficient S, m^2/kg		36.9				34				36.6		

The mechanical properties obtained on these bleached pulps showed an improvement of the tear index but no effect on the breaking length. However due to the obtained pulp quality, we could confirm a good suitability of these pulps for newsprint papermaking.
The optical properties were a bit low for news print paper, in term of opacity, some filler would have to be included in the paper production.
The K coefficient was normal with regard to the brightness level. The S coefficient was low compared to TMP softwood pulp usually used in news print paper (55 to 70, m^2/kg) but it was correlated with the opacity.
The brightness target was achieved.

Effluent analysis

The COD and the dry matter were analyzed in the pulp liquor after the first stage refining. The results are given in table n°23

Table n°23

	APMP1	APMP2	APMP3	Press (MSD)
NaOH, %	4	3	2	
H_2O_2, %	1	1	0,5	
pH	12,2	12,4	10,3	6,7
COD, kg/t	246,3	204,7	150,6	29,2
Liquor dry matter, kg/t	266,1	217,5	144,9	22,6

The COD of the pulp liquor after impregnation was well correlated with the sodium hydroxide charge applied as well as the liquor dry matter. The press effluent was also measured. The small COD charge was due to the small effluent flow at the MSD

pressafiner. The total COD obtained for APMP 2, which was the best compromise in term of mechanical and optical properties was about 235 kg/t of pulp. This polluting charge is usual for APMP process applied on hardwood.

Yields

The yields cannot be measured on the pilot plant but the liquor dry matter content can lead to a relatively precise estimation.
In case of APMP 2 the yield was 80% (total dissolved matters – mineral charge)and 85 % for APMP 3.

XXI. CONCLUSION

Whole jute is suitable for high yield pulping.
TMP pulp was obtained at pilot plant scale with weak mechanical properties. The pulp bleaching led to 70 %ISO brightness with 5% hydrogen peroxide charge. A chelation stage was necessary prior to the bleaching stage.
Three commercial enzymes were tested prior to the bleaching stage with minor effects
APMP process was tested successfully and pulps were obtained with different chemical charges leading to pulp mechanical properties suitable for news print papers. The brightness target of 60 – 65 %ISO was achieved.
The best chemical charge was 3 % sodium hydroxide and 1% hydrogen peroxide for the impregnation stage and 3 % hydrogen peroxide and 1.5 % sodium hydroxide applied for the bleaching stage. However these chemical charges can only be slightly reduced.
In order to obtain the desired opacity, some fillers would be introduced in the paper composition.
At about 100 ml CSF the total energy consumption was comparable for APMP with 3% NaOH in the impregnation and TMP (about 2000 kWh/t).

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