

Report on

**Demonstration of
Enzymatic Prebleaching
in A Pulp & Paper Mill**

**Submitted
to
CESS Grant Authority**

By



**Central Pulp & Paper Research Institute
Saharanpur , U. P – 247 001**

PROFILE

Project	Demonstration of Enzymatic Prebleaching In a Pulp & Paper Mill
Objective	The Institute would like to demonstrate the biobleaching process on mill scale for improving the quality optical properties of pulp and environment in terms of reduced AOX level in bleach effluent.
Duration	April , 2002 – March , 2006
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Acknowledgments

“ Science should shape in to technology ” - Once again the Moto of CPPRI is fulfilled on successful completion of this project.

Central Pulp & Paper Research Institute is indebted to members of CESS grant commission for sanctioning the project and the financial support.

Special gratitude goes to our Industrial partner **Seshasayee Paper & Boards Ltd., Erode** for their valuable input:

- Shri N. Gopalratnam , M.D – for making the SPB as the Industrial partner for the project
- Shri K.S. Kasi Viswanathan, Director (Operations) – for his intellectual, technical, Hospitality every support needed by CPPRI & SPB for the enzymatic prebleaching trial.
- Dr.S.K.Paul & Mr. Rajendran (R & D) – For their support during lab scale experiments and also in data creation during mill trials.
- Dr.C.S.Sudarsan & Mr. Subrahmanyam (Chemical mill) – for their incredible support for the process modifications & technical discussions in the mill in critical conditions also as and when required by CPPRI for enzyme bleaching trial.
- CPPRI would also like to thank all the members of process lab, Chemical mill, R & D lab, Projects for their support during the enzyme trial at SPB ltd.

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Chapter – 1

Executive Summary

CPPRI has carried out extensive lab and pilot scale research on enzymatic prebleaching with various imported and indigenous xylanase enzymes on industrial grade pulps derived from different kind of raw materials. Studies carried out at the Institute and at mill showed that there is a need to demonstrate the technology in a mill using proper equipment and conditions required for efficient implementation of the process. Demonstration of the process should help in creating the much needed confidence among the paper mills.

In this context, CESS Committee awarded a project entitled **“Demonstration of Enzymatic Prebleaching in a Pulp & Paper Mill”**.

Main objective of the project is to demonstrate the biobleaching process on mill scale for reduction of elemental chlorine and chlorine bleach chemicals and at the same time improvement in environment in terms of reduced AOX level in bleach effluent.

Seshasayee Paper & Boards Ltd., Erode was identified by IPMA for the demonstration of the trial. As per the requirement of the process at SPB mill, equipment for the enzyme trial i.e MC Pump, Enzyme dosing pump etc. were procured and installed. Xylanase enzyme for mill trial has been identified on the basis of both performance and cost effectiveness.

Mill Trials

Two trials using different xylanase enzymes were conducted by CPPRI at SPB using the installed equipment. With the first enzyme, Sebritte BB from M/s Advanced Biochemicals Ltd., Mumbai, the trials were conducted during the month of June 2006 and with the second enzyme, i.e Pulpzyme HC of Novozymes, supplied by M/s Value Addition Papers Ltd., New Delhi, mill trials were conducted during November, 2005. It has been concluded that installation of required equipments, i.e MC Pump, stand Pipe and enzyme dosing pump etc. by CPPRI at the pulp mill and proper enzyme dosing has helped in proper mixing of the enzyme with the pulp. This has resulted in achieving the targeted results in terms of savings of chlorine to the tune of 15% and above in both stages as well as reduction in AOX level of around 22-25%.

Observations of the mill trial are drafted in the form of record note at the end of each trial. Record notes are cited as Annexure – 1 & 2. Preliminary economics of the technology also has been calculated on the basis of mill data.

The Enzyme trial has been quite successful and it is very helpful to Paper Industry –

- MC Pump has well taken care of mixing of enzyme.
- Reduction of total chlorine consumption by 15% , Significant drop in PC Number and good reduction in AOX levels.

Mr. K.S. Kasi Viswanathan
Director (Operations)
SPB Ltd. Erode

Chapter – II

The existing technology for the production of paper uses a chemical bleaching step, which is required to obtain the desired brightness and quality of paper. However the bleaching step produces substantial amount of chlorinated by-products such as AOX, which are toxic. These chlorination degradation products are carcinogenic and teratogenic. Disposal of such toxic effluents is a major problem, as these compounds are recalcitrant to degradation by electrochemical or biochemical treatments. Hence there is an urgent need to develop alternate eco-friendly technologies. It has been demonstrated that pretreatment with xylanase lowers the consumption of molecular chlorine or chlorine dioxide, thereby reducing absorbable halogen (AOX) levels in the effluents.

Studies on enzymatic prebleaching of pulps have been carried out in the developed countries using their own xylanase preparations on the pulps produced from softwood and certain species of hardwood. The technology has been tried on commercial scale in few of mills based on softwood and hardwood, however studies on application of the biobleaching technology using xylanase on nonwood pulps like Bagasse and straw is scarce.

Efforts are under way for adoption of the enzymatic prebleaching technology in India. However in view of the prevalent conditions in the Indian Paper Industry like wide variation in pH of pulps, temperature and nature of fibrous raw material, the technology could not be adopted successfully. Some of the laboratory scale works on production of xylanases have been carried out at NCL, Pune, IIT, Delhi and other laboratories like Punjab University etc. However no successful attempts on development of Xylanase prebleaching technology have been made.

1. Background of The Project :

In the global context of switch over to biotechnology, the other alternative being used for pulp bleaching using chlorine is viewed with positive disfavour. Therefore, it becomes essential that paper industry in India too should opt for the 'enzyme alternative' at the earliest. The day may not be far off when paper products manufactured with chlorine compound-based technology are prohibited for wrapping food products and other consumer items so that our export markets do not suffer.

If we have to develop indigenous enzyme technology to suit the indigenously available raw materials for paper manufacture, we have to evolve strategies that generate viable technologies for xylanases' production, based on original discoveries. Programmes involving microbiologists, biochemists, process engineers have to be coordinated; and in collaboration with the paper industry the problem must be looked at in total perspective to create effective and functional networks which are progress- and result-oriented.

CPPRI has been engaged for last four years in the area of enzymatic prebleaching of pulps using the globally available enzymes where in several commercial xylanase preparations have been evaluated for their response on wood kraft pulps. Studies carried out at the Institute and at the mill site on wood kraft pulp have shown wide variation in the pH of the pulps and other conditions making it difficult to achieve desired enzyme efficiency. When applied under optimum conditions of pH, Temperature, enzyme dosing with proper mixing of the enzyme with the pulp, enzymatic prebleaching can be successfully employed to achieve 2% gain in final bleached pulp brightness, 15% reduction on chlorine demand and equivalent reduction in AOX generation.

Biobleaching technology is in the developmental stage as far as Indian Paper Industry is concerned. Xylanase enzymes have been developed and available in the market but these enzyme preparations are highly sensitive to pH and temperature which varies considerably from mill to mill depending upon the fibrous raw materials employed

and also the process conditions. Under the conditions prevalent in the Indian industry it is required to select enzymes which could tolerate the extreme conditions existing in the mills so that enzymes could work effectively. Efforts have been made in the past at CPPRI, to evaluate the performance of various prebleaching enzymes available on various pulps. Based on the studies it was found that these enzymes have different pH & temperature optima. The effectivity of the enzymes also varied from enzyme to enzyme.

Biobleaching employing enzymatic bleaching techniques is now one of the preferred routes, primarily because of number of advantages offered over conventional chemical routes. The major advantage of the process is the reduction in AOX levels in the discharge effluents by reducing the requirement of elemental chlorine during bleaching. During the 9th five year plan, Institute has created required infrastructure & facilities for carrying out the research in the area of Biotechnology, as they were not existing earlier. Institute has also made significant progress in establishing the analytical procedures & screening of enzymes / microorganisms for biobleaching. Studies carried out at the Institute and at mill showed that there is a need to demonstrate the technology in a mill using proper equipment and conditions required for efficient implementation of the process. Demonstration of the process should help in creating the much needed confidence among the paper mills.

Central Pulp & paper Research Institute realized the need and specific requirement of Industry, demonstration of the enzymatic prebleaching process in a Pulp & Paper Mill will generate confidence and will encourage for adoption of the technology in Indian mills. In this context, CESS Committee awarded a project entitled **“Demonstration of Enzymatic Prebleaching in a Pulp & Paper Mill”**. The project basically aim towards the demonstration of the enzymatic prebleaching process on mill scale for improving the quality optical properties of pulp and environment in terms of reduced AOX level in bleach effluent.

2. Xylanase Enzymes in Pulp Bleaching

Xylanases such as endoxylanases are xylan specific enzymes. They catalyze the hydrolysis of xylose - xylose bonds within the xylan chain and only solubilize a fraction of the total xylan present. It catalyzes the hydrolysis of 1, 4, β -D-xylosidic linkages in xylan, a hemicellulose in pulp. Several industrial xylanase preparations are commercially available, with many other natural xylanases having been identified. Variations in xylanases can occur between size, structure, amino acids, position attached on the xylan, attachment to xylan, stability in the pulp slurry and also stability while acting on the pulp.

Effective xylanases should preferentially have the characteristics like:

- These should be stable on kraft pulps. Some xylanase preparations non-specifically adsorb to pulp fibers and are inactivated by degradation products from kraft pulping.
- These should have a neutral to alkaline pH optimum since residual alkali leaks out of the pulp during enzyme treatment, and the pH of even well washed pulp stocks can shift upwards dramatically.
- These should have good thermal stability.

Innovative approaches for the screening of novel xylanolytic microbial strains from mesophilic as well as extreme environments must be undertaken with full vigor to explore, isolate, and conserve in germplasm banks a diversity of microbial species on which basic microbiological studies as well as recombinant DNA explorations can be undertaken for obtaining xylanase enzymes with novel properties that can be eventually exploited commercially. Realistic cost estimates and improvement in process economics are the key factors in the commercial success of any technology and therefore it must be clearly understood that no enzyme-based process for bleaching can be as inexpensive as using chlorine or even organic chlorine compounds. Thus, the added expenses incurred by the use of enzymes must be

viewed in terms of their accrued indirect benefits like prevention of environmental derangement and reduced health hazards to mankind

Current efforts are aimed at process optimization, simplification, and cost reduction of enzyme application in pulp industry. Nisson *et al.* have pointed out that with the xylanases available commercially at present, a pH adjustment of the incoming pulp from pH 10–11 to 6–8 is necessary for its optimal activity. From an industrial point of view, it is simple to adjust the pH but difficult and expensive to control temperature due to the cost of cooling. The ideal solution therefore would be to use enzymes with higher pH and temperature stability, which will make the large-scale operations more simple and cost effective. It is thus obvious that the focus of future developments will be on identifying xylanases with higher thermostability at high alkaline pH, and developing process technologies for commercial-scale manufacture of such enzymes.

Enzyme application improves pulp fibrillation and water retention, reduction of beating times in virgin pulps, restoration of bonding and increased freeness in recycled fibres, and selective removal of xylan from dissolving pulps. Xylanases are also useful in dissolving pulps, yielding cellulose for rayon production, and biobleaching of wood pulps. Application of xylanases together with other bleach agents, such as oxygen and hydrogen peroxide in pulp industry has been extensively investigated and projections of a totally chlorine-free pulp technology have been put forward. Pulpzyme HA, introduced by Novo Nordisk A/S, was the first commercially available xylanase for use in biobleaching of wood pulps. It was extracted from a strain of *Trichoderma reesei* and was used in the first bleaching stage to reduce the dosage of active chlorine. Several multinational biotech companies are marketing various xylanase preparations, such as Irgazyme (Genencor International), Cartazyme (Sandoz), Ecopulp (Alko), VAI xylanase (Voest Alpine), Biopulp (Biocon), and Pulpzyme HC (Novozymes). Enzymatic pre-bleaching has been successfully demonstrated on mill scale wherein a pulp with 88% ISO brightness was achieved when used together with chlorine dioxide and hydrogen peroxide. The following data published from Finland of a large-scale mill trial would enable a better appreciation of enzymatic pre-bleaching with xylanase: 35 tons of Albazyme 10 was used to treat

and produce 35,000 tons of fully bleached pulp from hard wood as well as soft wood. The enzyme was added to a kraft-cooked pulp, after suitably adjusting the pH and temperature to suit the optimum conditions for enzyme activity. An overall reduction of 12% chlorine use could be achieved. Lundgren *et al.* have reported mill trials on soft-wood pulp using a xylanase optimally active at pH 9.0 and 65° C. They observed that in the pulping sequence where chlorine was totally eliminated, the pulp bleaching and brightness were satisfactory and also consumed lesser quantities of H₂O₂. This is an example of applying an alkali-stable and heat-stable xylanase for large-scale pulp biotechnology.

How xylanases act and improves bleachability?

Xylanase treatment can improve lignin extraction, alter carbohydrates and lignin association or cleave reabsorbed xylan.

Mechanism:

One possible mechanism is that xylan reabsorbed on the fiber surface during kraft pulping and creates a barrier against lignin removal during bleaching with bleach chemicals. The xylanase treatment may remove the xylan barrier allowing better access by bleaching chemicals to residual lignin covered up by the xylan barrier.

The other possible mechanism suggests that readsorbed xylan may be chemically bonded to residual lignin in pulp. These lignin carbohydrate complexes are thought to be difficult to be removed due to diffusion limitations. According to this hypothesis, by cleaving xylan chains involved in these complexes, smaller residual structures of increased mobility are formed. The increased mobility allows for easier removal of these structures from the fiber.

Xylanase enzyme cleaves the xylan that allows the removed of such groups. This may result in savings as chemicals that would be needed to bleach these residues. The removal of the hexenuronic acid by xylanase treatment also help in respect of brightness reversion of the treated kraft pulp.

It has also been suggested that one UV absorbing material, hexenuronic acid is formed during kraft pulping from methylglucuronic acid residues present on xylan. The cleavage of the xylan allows the removal of such groups, resulting in savings of bleach chemicals, that would be needed to bleach those residues.

Chapter – III

Mill Scale Application of Xylanases In Bleaching

Xylanase enzyme aided bleaching is the most widely used example of biotechnology in actual mill operations. About 20 bleached kraft mills in Canada, Finland, the US, and other countries use xylanase on a continuous basis. The mills use xylanase to decrease costs, decrease chlorine dioxide usage, increase pulp brightness, decrease effluent discharges or obtain any of several other benefits. All of these benefits are obtained with low capital costs.

Xylanase is added to the pulp prior to bleaching. The enzyme does not bleach, brighten or delignify the pulp. Rather, the removal of a portion of the xylan increases the efficiency of the subsequent bleaching chemicals. More specifically, xylanase treatment increases the alkaline extractability of the lignin, which decreases the amount of conventional bleaching chemicals required to bleach the pulp.

Xylanase is added to brown stock, typically just prior to the high density brownstock storage tower as shown in fig – 1. The enzyme is a solution of active protein in water and is added to the brown stock Decker, to the repulper or to the suction head of the stock pump. The key issues in choice of location include corrosion of equipment, safety of acid use, and mixing of the enzyme in to the pulp. The enzyme is usually added with acid and dilution water, to ensure the control of the pH and adequate dispersion of the enzyme in to the pulp, respectively. After enzyme addition, the pulp is pumped to the brown stock storage tower, where it traverses the tower while xylanase acts on the pulp. When the pulp emerges from the brown stock storage tower, it is ready to be bleached.

The treated pulp requires 10% to 20% less bleaching chemicals to reach a given brightness than the untreated pulp. Alternatively, the treated pulp can be bleached to a

higher brightness than an untreated pulp. In some mills, a combination of higher brightness and lower chemical usage is obtained.

The enhance bleachability by Xylanase offers mills several options. The decreased use of chemicals results in a decrease in overall bleaching costs. This is the most common and most important benefit from using Xylanase. The enzyme treatment can also extend the capacity of a ClO_2 generator, in mills that are limited by ClO_2 production. The increase in brightness ceiling can enable a mill to make a new grade of pulp. The decrease in ClO_2 usage corresponds to a decrease in effluent discharges, including dioxin and AOX.

Characteristics of mills with xylanase treatment

Property	Mills running xylanase
Chip furnish	Hardwood, Softwood, Eucalyptus
Digester	Batch , Continuous, MCC
Additives	Surfactant, AQ
Brownstock	Conventional, O2 - delignified
Bleaching	Chlorine, ECF,TCF
Extraction stages	E,Eo,Ep,Eop
Pulp	Semi bleached, fully bleached

Other mill specific benefits are often observed with xylanase -

- A primary benefit of xylanase is the low capital cost required for implementation of the technology.
- A second important feature is the versatility of the technology

1. Process conditions of xylanase treatment in a bleach plant :

The following parameters are necessary for the implementation of xylanase enzyme technology in a mill

- 1. Pulp Susceptibility**
- 2. Enzyme selection**
- 3. Mixing & Dispersion**
- 4. Reaction conditions**
- 5. Bleach Plant control**

1. Pulp Susceptibility

Pulp susceptibility, the amount of xylose released from the pulp for a given dosage and time of xylanase enzyme treatment. The susceptibility of hardwood is higher than other agrobased & softwood pulps.

A second property of the system is the selectivity, which relates the bleaching benefit to the amount of xylose released during xylanase treatment. The bleaching benefit is expressed as the decrease in the amount of bleaching chemical required to achieve a given level of brightness. A system with a high selectivity has a relatively high bleaching chemical savings per unit of xylose released.

The selectivity does not correlate with pulp susceptibility, brightness gain, brightness target or other factors. Selectivity clearly depends on the pulp used. Different selectivities are also reported with different xylanases as well. Therefore, the selectivity depends on both the pulp and the enzyme used. An understanding of selectivity will point the way toward improving the enzymes and the value of the xylanase treatments.

Evaluation of the response of Rice straw pulps towards xylanases

Pulps : Rice straw + Jute (85:15) (Madhya Bharat Paper Mills Ltd, Champa.)

Kraft Pulp (Rice straw) prepared in Laboratory with 17% Sulfidity as Na₂O 11%.

Enzyme : Xylanase received from IIT, New Delhi

Enzyme Activity, IU/ml : 700.0

Table- 1 Enzyme Pretreatment Conditions				
Particulars	Control	Enzyme Treated pulps	Control	Enzyme Treated pulps
	Sulfite pulps		Kraft Pulps	
Enzyme dose, IU/g (OD pulp)	-	10	-	10
Treatment Time , (hrs)	2.0	2.0	2.0	2.0
Temperature , ° C	50	50	50	50
Consistency of the pulp , %	8.0	8.0	8.0	8.0
pH	5.8	5.8	8.4	8.46

Table- 2 Effect of Enzyme Treatment on Unbleached Pulp				
Particulars	Control	Enzyme Treated pulp	Control	Enzyme Treated pulp
	Sulfite pulps		Kraft Pulps	
Brightness, % ISO	35.6	35.5	39.3	40.0
Kappa number	19.15	18.94	10.0	9.59

Table : 3 Effect f Enzyme Treatment on Sulfite & Kraft Pulps Bleached With CE(P)H Sequence				
Particulars	Control	Enzyme treated pulp	Control	Enzyme treated pulp
	Sulfite pulp		Kraft pulp	
Chlorination stage				
Kappa Factor	0.25	0.25	0.25	0.25
Applied chlorine,%	4.8	4.8	2.5	2.5
Consumption , %	91.5	92.5	85.5	86.6

E (p) - Alkali Extraction stage				
Applied NaOH,%	2.8	2.8	2.8	2.8
Peroxide ,%	0.04	0.04	0.04	0.04
Consumption,%	51.79	40.54	34.61	42.5
CE brightness,%	58.9	60.3	58.9	58.0
Hypo stage				
Applied Hypo,%	3.0	3.0	1.5+1.5	1.5+1.5
Consumption , %	95.9	94.0	95.1	94.01
Final brightness of the pulp, % ISO	79.5	78.2	63.6	61.5
Brightness improvement% ISO	-	-	-	-

- Enzymatic prebleaching experiments were carried out on rice straw sulfite pulps (collected from mill) to study the xylanase enzyme response on these pulps.
- The pulps were treated with two xylanase enzymes , enzyme-1 & 2 (Commercial and R & D lab enzymes) and bleached with CE(O)H sequence.
- The results showed that xylanase enzyme treatment did not show any improvement in brightness of the pulp.

As, therefore the response of rice straw sulfite pulps towards xylanase is not significant as it was evaluated by two enzymes.

2. Enzyme Selection :

The important attributes of a Xylanase enzyme for bleaching applications are :

- 1. pH range**
- 2. Temperature range**
- 3. Reaction time**
- 4. Bleaching benefit**

Much research has focused on the properties of the enzyme, especially the pH and temperature ranges. Commercial xylanases encompass an effective span of 1 to 2 pH units with in pH 5 to 8.5 and 5 to 10 °C with in 45 to 65°C. The variation among xylanase in rate of reaction on pulp and bleaching benefit was reported. Xylanases can require 15 minutes to two hours to achieve positive benefit. The amount of bleaching chemical saved varies several fold among xylanases, as does the selectivity, which is often expressed as yield loss.

Effect of Reaction Time on Enzyme Pre bleaching:

- During the mill scale studies on enzymatic prebleaching at various mills , it was observed that the Pulp retention time in High density tower varies drastically in the range of 1- 4 hr.
- To study the effect of enzyme retention time on pulp, experiments were carried out on hard wood pulps collected from a hard wood based mill using different retention periods, 1.5, 3.0 & 6.0 hr.

Pulp : Hard wood pulp (Star Paper Mills Ltd.)

Enzyme : Sebrite 'BB'

Enzyme Activity, IU/g : 6,600 :

Table- 4 Enzyme Pretreatment Conditions						
Retention time (hrs)	1.5		3.0		6.0	
Particulars	Contr ol	ET	Control	ET	Control	ET
Enzyme dose, % (OD pulp)	--	0.04	--	0.04	--	0.04
Treatment Time , (hrs)	1.5	1.5	3.0	3.0	6.0	6.0
Temperature , ° C	50	50	50	50	50	50
Consistency of the pulp , %	10	10	10	10	10	10
pH	8.0	8.1	8.0	8.15	8.34	8.3

Table- 5 Effect of Enzyme Treatment on Unbleached Pulp						
Retention Time (hrs)	1.5		3.0		6.0	
Particulars	Control	ET	Control	ET	Control	ET
Brightness, % ISO	27.3	27.9	27.4	27.9	27.4	27.6
Kappa number	19.5	19.9	20.9	21.4	20.3	22.2
Characterisation of Water Extracts Before And After Enzyme Treatment						
Colour., kg/tp	546	1062	616	949	758	981.4
Lignin , kg/tp	58	113.5	75.8	112.5	80.4	101.6
sugars as xylose, g/l	0	162	0	246	0	405

Table : 6 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CEH Sequence						
Retention Time, hrs)	1.5		3.0		6.0	
Chlorination stage						
Particulars	Control	ET	Control	ET	Control	ET
Applied chlorine, %	3.9	3.35	3.9	3.35	3.9	3.35
Consumption , %	99	99	99	99	99	99
Savings in chlorine,%	-	14.1	-	14.1	-	14.1
Alkali Extraction stage						
Applied NaOH, %	1.8	1.8	1.8	1.8	1.8	1.8
Consumption, %	69	58	69	64	57	67
Hypo stage						
Applied Hypo, %	2.5	2.5	2.5	2.5	2.5	2.5
Consumption , %	92	92	90	92	93	93
Final brightness of the pulp, % ISO	78.2	78.1	78.5	79.0	78.4	76.9

ET- Enzyme treated

Table - 7 Strength & Optical Properties of Wood Pulp Before & After Enzyme Treatment				
Retention Time, (hrs)	1.5		6.0	
Strength Properties				
Particulars	Control	ET	Control	ET
Revolution, PFI	2000	2000	2000	2000
Freeness, CSF	190	200	220	285
Apparent density, g/m ³	0.85	0.87	0.82	0.87
Burst Index, Kpa.m ² /g	3.50	4.00	3.60	3.80
Tensile Index, Nm/g	56.5	60.0	70.0	56.0
Tear Index Mnm ² /g	4.5	4.55	5.80	5.70

ET- Enzyme treated

- The results showed that there is brightness improvement upto 3.0 hr of enzyme treatment of pulps and after that in the next 3.0 hrs enzyme treated pulps showed loss in brightness without much change in strength properties when compared with untreated pulps.

3. Mixing and dispersion of the enzyme into the pulp

The importance of uniform dispersion of enzyme into the pulp can not be overlooked. Many trials have failed for this reason. The uniformity of dispersion can be quantified by mixing tests. Typically MC pumps do a fine job of mixing the enzyme in to the pulp. With a proper configuration, thick stock pumps can match this performance.

4. Maintaining reaction conditions :

The primary reaction conditions are pH, temperature and reaction time. Most xylanase treatments take place in the brown stock high-density storage tower, prior to the bleach plant. The short, squat nature of brown stock storage towers leads to channeling of the pulp, which decreases the time that xylanase can act on the pulp. The organics present in the pulp because of poor washing may effect the enzyme efficiency.

Enzymatic Prebleaching studies on different carryover (COD) in the pulp samples

Pulp Sample Collected : Hard wood based mill

Enzyme : Sebritte BB

Table – 8 Results of enzyme treated & untreated hardwood pulp with different COD loads						
Enzyme pretreatment conditions						
Treatment Time , (hrs) : 1.5						
Temperature , ° C : 50						
Consistency of the pulp , % : 10						
pH : 8.2						
Particulars	COD, 5.8 kg/tp		COD, 14.5 kg/tp		COD, 28.9 kg/tp	
	Control	Enzyme treated pulp	Control	Enzyme treated pulp	Control	Enzyme treated pulp
	C1	ET1	C2	ET2	C3	ET3
Enzyme dose, % /g OD pulp	--	0.05	--	0.05	--	0.05
Unbleached pulp characteristics						
Brightness , %	26.3	26.9	26.2	26.5	25.8	26.0
Characterisation of water extracts						
Colour , kg/tp	4.72	7.19	13.46	15.37	33.76	28.89
Lignin, kg/tp	0.96	1.35	1.37	2.59	9.05	7.61

Table – 9 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CEH Sequence						
Particulars	COD, 5.8 kg/tp		COD, 14.5 kg/tp		COD, 28.9 kg/tp	
	Control	Enzyme treated pulp	Control	Enzyme treated pulp	Control	Enzyme treated pulp
Chlorination stage						
Applied chlorine, %	4.7	4.7	4.7	4.7	4.9	4.9
Consumption , %	95	83	93	93	98.5	98
Alkali Extraction stage						
Applied NaOH, %	2.0	2.0	2.0	2.0	2.0	2.0
Consumption , %	61	70	68	79	82	79
CE brightness, %	32.5	34.9	32.4	34.3	31.0	32.5
Hypo stage -1						
Applied Hypo, %	2.5	2.5	2.5	2.5	2.5	2.5
Consumption , %	98	94	98	94	98	94
Final brightness of the pulp, % ISO	78.0	81.1	75.5	78.8	76.0	76.5

Brightness Improvement , %	--	3.1	---	3.3	-	0.5
Hypo stage - 2						
Applied Hypo, %	0.5	0.5	0.5	0.5	0.5	0.5
Final brightness of the pulp, % ISO	79.7	82.5	78.5	81.2	81.5	81.1
Brightness Improvement , %	-	2.8	-	2.7	-	-

Observations:

- From the results showed in table - 8 and 9 showed that higher COD effects the enzymatic prebleaching efficiency.
- Brightness improvement with enzyme obtained upto the 15kg/ton COD after that the enzyme efficiency was affected.

5. Control of the bleach plant:

The amount of chemicals saved depends on where the chemicals are cut in the bleach plant. The optimum areas to cut can be determined based on the control strategy the mill uses.

Xylanase aided bleaching is the most widely used example of biotechnology in actual mill operations. About 20 bleached kraft mills in Canada, Finland, the US, and other countries use Xylanase on a continuous basis. The mills use Xylanase to decrease costs, decrease chlorine dioxide usage, increase pulp brightness, decrease effluent discharges ,or obtain any of several other benefits. All of these benefits are obtained with low capital costs.

Increasing our understanding of selectivity and other aspects of the mechanisms of xylanase in bleaching offers the potential for improving benefits in a mill.

2. Identification of a Pulp & Paper Mill for demonstration of Enzymatic Prebleaching :

On the basis of extensive research in the area of enzymatic prebleaching at lab and bench scale levels, Institute had planned for mill scale trials. Lot of correspondence has been made for taking up demonstration trials in 8 Indian integrated Pulp & Paper Mills and received positive response from some of the mills.

- Correspondence has been made for taking up demonstration trials in 8 Indian Paper Mills and received positive response from some of the mills.

Mills with which correspondence has been made

1. M/s Seshasayee Paper & Boards Ltd., Tamilnadu
2. M/s West Coast Paper Mills Ltd. Karnataka
3. M/s Cachar Paper Mills, Assam
4. M/s Nagaon Paper Mills, Assam
5. M/s Mysore Paper Mills, Karnataka
6. M/s Tamilnadu Newsprint Ltd., Tamilnadu
7. M/s JK Paper Mills Ltd., Orissa
8. M/s Star Paper Mills Ltd. Saharanpur

Mills from which positive response received

1. M/s Cachar Paper Mills, Assam
2. M/s Nagaon Paper Mills, Assam
3. M/s Mysore Paper Mills, Karnataka
4. M/s Tamilnadu Newsprint Ltd., Tamilnadu
5. M/s Seshasayee Paper & Boards Ltd., Tamilnadu
6. M/s West Coast Paper Mills Ltd., Karnataka

Selected mills:

- For the selection of mill in the meeting held with IPMA, M/s Seshasayee Paper Boards Ltd., Erode, Tamilnadu has been identified by Indian Paper Manufacturers Association (IPMA) and CPPRI has received the acceptance from the mill. Further it was also decided that M/s West Coast Paper Mills Ltd., Karnataka will be the second mill for taking up demonstration of the enzymatic prebleaching on plant scale.

3. Selected Mill For Mill Trial - Process Conditions & Studies on Enzymatic Prebleaching Studies At SPB Ltd.

Two Scientists of CPPRI visited the identified mill -- M/s Seshasayee Paper & Boards Ltd., to see the existing mill processes, type of raw materials used, pulping, washing & bleaching conditions and also to discuss the other mill facilities including civil, electrical and mechanical installations for application of enzymatic prebleaching process. The conditions were evaluated specially for the enzyme dosing point which is very important for the efficiency of the process. The details of the visit and the results of enzymatic prebleaching studies conducted at R & D lab, mill site are detailed in ANNEXURE - 3. The same detailed report has been submitted to mill authorities.

ANNEXURE - 3

REPORT ON

**ENZYMATIC PREBLEACHING OF PULPS
STUDIES CARRIED OUT**

AT

**R & D LABORATORY
M/S SESHASAYEE PAPER & BOARDS LTD.
ERODE, TAMILNADU**



**CENTRAL PULP & PAPER RESEARCH INSTITUTE
P. O. Box. No. 174, SAHARANPUR,
U. P – 247 001**

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- 1. Back ground**
- 2. Visit to Mill & Data collection**
- 3. Material & Methods**
- 4. Experiments - Bench Scale enzymatic pre
bleaching studies**
 - I. At R & D lab at mill site**
 - II. At CPPRI**
- 5. Observations**
- 6. Further Action Plan**

1. Back ground

As the global trend & Customers preference are switching towards cleaner and greener products, pulp and paper industry in India is required to modify its technology for elemental chlorine free (ECF) bleaching so as to reduce to adopt technologies which are eco friendly & there is a reduced generation of AOX in bleaching operation. Although the enzymatic prebleaching technology is widely adopted in developed countries, it is in very primitive stage as far as Indian Paper Industry is concerned.

CPPRI has been engaged in the area of enzymatic prebleaching of pulps using the globally available enzymes wherein several commercial xylanase preparations have been evaluated for their response on different kinds of pulps. Xylanase enzymes have been developed and are available in the market but these enzyme preparations are highly sensitive to pH and temperature which varies considerably from mill to mill depending upon the fibrous raw materials and also the process conditions.

In view of wider potential and urgent need of adoption of enzymatic prebleaching technology in Indian Paper Mills, CESS Grant Authority sponsored project to CPPRI entitled **“Demonstration of enzymatic Prebleaching in a Pulp & Paper Mill”** Demonstration of the process on mill scale should help in creating the confidence among the paper mills.

2. Visit to Mill & Data collection

CPPRI selected Seshasayee Paper & Boards Ltd. as the first mill for the mill scale trial of enzymatic prebleaching. Two scientists of CPPRI visited the mill to see the existing mill processes, type of raw materials used, pulping, washing & bleaching conditions and also to discuss the other mill facilities for application of enzymatic prebleaching. The process conditions were evaluated specially for the enzyme dosing point and enzyme mixing equipment which is very important for the efficiency of the process.

2.1 Selection of the dosing Point of the enzyme:

From the process lay out of the mill, two points were taken in to consideration for the enzyme addition.

1. HD Chest after brown stock 4th Washing : Unscreened Pulp
2. After screening : Screened pulp

Pulp Characteristics:

Unscreened pulp	Screened pulp
Consistency : 6%	Consistency : 6%
Temperature : 50° C	Temperature : 30 – 35 ° C
Retention time : 60 - 90 min.	Retention time : 45- 60 min.
pH : 10 -11	pH : 8.0-9.0
COD Carry over : 15-20 kg/tp	COD Carry over : 7-10 kg/tp
Kappa number : 21- 25	Kappa number : 21- 25
For Unscreened pulp <ol style="list-style-type: none">1. Because of unscreened nature of pulp enzyme demand will be high which increases the cost.2. There should be pH adjustment with acid to 8.0-9.0 which is the optimum pH range for most of the commercial xylanases , which may lead to corrosion of pipes and also adds to the cost.	Screened Pulp <ol style="list-style-type: none">1. Enzyme demand will be low which decreases the cost.2. No pH adjustment. Hot water washing is required to raise up the temperature.
On the basis of above characters, Decker was selected for the enzyme addition and for retention is Screened chest.	

Enzyme Dosing :

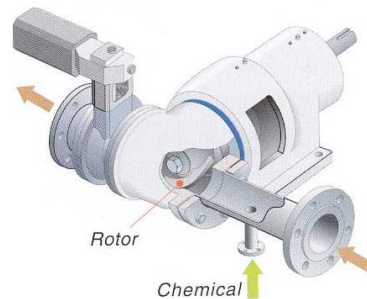
Dosing pump for enzyme will be supplied by the mill

Mixing of the enzyme with the pulp :

The main bottleneck for the application of this technology is mixing of the enzyme with the pulp. For proper mixing of enzyme with the pulp a mixing device should be provided after the enzyme addition. An efficient & cost effective device has to be selected suggested equipment are as follows.

1. AHLMIX Chemical mixer : (App.16 Lacs)

1. AHLMIX is a medium consistency chemical mixer designed for mixing both gaseous and liquid bleaching chemicals in to paper stock.
2. Variously used for oxygen, peroxide, chlorine dioxide & chlorine mixing.



2. Peg mixer :

Mixing in medium consistency pulp suspensions has conventionally been achieved by peg mixers. These are tubular vessels having one or two shafts with pegs that rotate between stationary elements attached to the mixer casing. As pulp is conveyed through the mixer, the rotating bars shear the suspension against the stationary elements.

Used to mix bleaching chemicals in to high consistency pulp.

3. Material & Methods

Pulp samples:

Unbleached screened pulp samples of mixed hardwood (Casuriana + Eucalyptus hybrid) pulp from the Decker washer and OD thickner were collected and mixed in a ratio of 70: 30 to make a Composite samples which were used for enzymatic prebleaching studies conducted both at R & D lab at SPB and at CPPRI.

3.2 Enzyme source:

Two of the identified xylanase enzymes, xylanase-1 & xylanase-2 procured from reputed enzyme manufacturing companies has been used for these studies.

Enzyme	Enzyme activity, IU/ ml
Xylanase -1	5,000 IU/g
Xylanase –2	7,000 IU/ml

2.3 Enzyme pretreatment of pulps:

Xylanase pretreatment of pulp was carried out. Enzymes were added to the pulp after sufficient dilution & mixed properly by kneading mechanism. The conditions for enzyme treatment of pulp are shown in corresponding Tables. Control was run paralally with maintaining all the conditions except the enzyme.

Process conditions used during bleaching of Pulps :

Both enzyme treated and untreated pulps were followed by conventional bleaching without washing the pulps after enzyme treatment stage. Process conditions employed for bleaching of Pulps CE (P) HH sequence both with & with out enzyme preparations according to the present mill conditions are shown in table-1.

Table-1 Process conditions used during bleaching of pulps				
Particulars	Chlorination stage	Alkali Extraction Stage (p)	Hypo stage - 1	Hypo stage - 2
Temperature, °C	Ambient	70	2.0	2.0
Pulp Consistency, %	3.0	10.0	10.0	10.0
Retention Time, min	45	60	120	120
Final pH	1.8-2.0	>10.5	>9.0	>9.0

4. Experiments

I. Bench Scale enzymatic pre bleaching studies at R & D lab at mill :

Lab scale enzymatic prebleaching studies were conducted at R & D laboratory of the mill to evaluate the response of enzymes on the pulp produced by the mill and also using the mill prevalent conditions.

Evaluation of the enzyme response on the pulp as prebleaching agent in order to

- Explore potential of savings of elemental / total chlorine so that possibility of reduction of AOX could be explored.
- Assessing gain in brightness of the pulp after final bleach stage without losing strength properties

Table – 2 Characteristics of the unbleached screened pulp		
Particulars	Decker	OD Thickner
pH of the pulp	8.8	8.9
Consistency of the Pulp	7.34	6.84
Brightness , % ISO	22.0	21.0
Soda loss, %	13.4	13.4
Kappa number	19.5	19.5
Both pulps mixed in a ratio of 70: 30		

Table - 3 Enzyme Pretreatment Conditions			
Particulars	Control	Enzyme treated pulp	
		Xylanase-1	Xylanase-2
Enzyme dose, % (OD pulp)	0	0.04	0.06
Treatment time , (hrs)	1.0	1.0	1.0
Temperature , ° C	50	50	50
Consistency of the pulp , %	7.0	7.0	7.0
pH	8.7	8.7	8.7

Results & discussion

Effect of enzyme treatment on pulp:

a) Kappa number :

From the results shown in Table - 4, it is clearly evident that there is reduction in kappa no. of unbleached Kraft wood pulp after enzyme treatment which has been decreased by 1.0 point with gain in pulp brightness of 1.0 point i.e. from 22.0 to 23% ISO.

Table- 4 Effect of Enzyme Treatment on Unbleached Pulp			
Particulars	Control	Enzyme treated pulp	
		Xylanase-1	Xylanase-2
Brightness, % ISO	22.0	23.0	21.0
Kappa number	19.3	18.3	18.5

Effect of enzyme on brightness of the Pulp:

Results of the enzymatic prebleaching followed by CEHH bleaching are shown in the table –5. The enzyme treated pulps could be bleached to higher brightness with a gain in brightness level of 1.5-2.0 % ISO in both sets while using similar chlorine dosages as in case of control pulp sample.

Effect of xylanase treatment on bleach chemical requirement:

Xylanase treated pulps differ in response to bleach chemical when compared to untreated pulps. Bleaching of the control and enzyme treated pulps using

conventional CEHH bleach sequence showed remarkable reduction in the requirement of total chlorine 16.5% i.e from 8.5 % to 7.1% whereas elemental chlorine which is reduced from 4.5 (45.00 kg/tp) to 3.8% (38.0 kg/tp) and hypo demand is reduced from 4.0% to 3.3% in case of enzyme treated pulps while maintaining the targeted brightness of 83.

Table : 5 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CEHH Sequence					
Particulars	Control	Xylanase-1		Xylanase-2	
		ET1	ET2	ET1	ET2
Chlorination stage					
Applied chlorine, %	4.5	4.5	3.8	4.5	3.8
Cl ₂ Consumption , %	98.9	98.9	99.2	98.5	98.9
Savings in chlorine , %	--	--	15.5	-	15.5
Permanganate number	6.0	5.6	5.8	5.5	5.7
Alkali Extraction stage					
Applied NaOH, %	1.7	1.7	1.7	1.7	1.7
Peroxide , %	1.0	1.0	1.0	1.0	1.0
CE brightness, %	53.0	56.0	52.0	57.0	54.0
Permanganate number	3.5	3.2	3.8	3.6	3.8
Hypo stage – H1					
Applied Hypo, %	2.0	2.0	1.5	2.0	1.5
Consumption , %	96.1	95.4	96.7	95.4	97.6
Brightness of the pulp, % ISO	77.0	78.0	75.0	77.0	75.0
Hypo stage – H1I					
Applied Hypo, %	2.0	2.0	1.8	2.0	1.8
Final brightness of the pulp, % ISO					
Evaluated at SPB	83.0	84.5	83.5	84.0	83.0
Evaluated at CPPRI	82.0	83.31	81.0	85.33	82.60
CED Viscosity , Cm ³ /g	402	342	450	302	441

ET1: Enzyme treated pulps bleached with same Cl₂ dose as of control

ET2: Enzyme treated pulps bleached with less Cl₂ dose as of control

4. Observations:

Studies on evaluation of the identified xylanases at R & D lab on wood kraft pulp of SPB has been found to be encouraging in terms of

- Reducing the requirement of Cl_2 to the tune of 15-18% in wood kraft & kraft bagasse pulps
- Brightness gain of the bleached pulp is estimated to more than 2% ISO

II. Bench Scale enzymatic pre bleaching studies conducted at CPPRI

Unbleached pulp from the mill was brought to CPPRI to confirm the results of enzymatic prebleaching studies conducted at R & D lab and also for detailed characterization of the enzyme treated pulps.

Table – 6 Characteristics of the unbleached screened pulp	
Particulars	Pulp collected from Decker & OD Thickner
pH of the pulp	8.0
Consistency of the Pulp	9.67
Brightness , % ISO	22.00
Soda loss, Kg/tp	14. 25
COD Carryover of the Pulp , kg/tp	43.26
Kappa number (unwashed)	24.73
(Washed)	19.56

A. Determination of the Kappa no. of pulp:

Kappa no. of the pulp procured from the mills, control pulp & enzyme treated pulp i.e just after xylanase treatment, after chlorination & extraction stage (CE) were determined following standard TAPPI procedure T-236-0S-76.

B. Evaluation of bleached pulp characteristics:

a) Determination of pulp brightness :

Brightness of the pulp samples were measured on a pulp by following the procedure given in ISO DIS 3688.

b) Determination of strength properties :

Strength properties of both enzyme treated & untreated pulp samples were determined by beating the pulp in PFI mill to various degree of freeness under standard conditions as per ISO DP 5264 i.e :

- Beating pressure -- 17.7 N/Cm.
- Relative speed -- 6.0 m/s
- Beating consistency -- 10% on weight basis.
- CSF measurement -- ISO DP 5267.

The temperature of the stock is recorded before and immediately after beating. Handsheets are made as per ISO DP 5269 & dried on plates in stack under the standard conditions for tropical countries (27°C , 65% RH).Physical testing of the handsheets are made according to the following standards:

- Tensile Index -- ISO 1924
- Tear index -- ISO 1974
- Burst Index -- ISO 2758

c) Determination of Intrinsic viscosity :

Viscosity of the pulp was measured as per standard procedure SCAN C – 15:62

Characterisation of the unbleached pulp:

Unbleached pulp samples both control & enzyme treated were characterised for Kappa No., Brightness % ISO & Viscosity and other parameters of interest.

Table- 7 Enzyme Pretreatment Conditions			
Particulars	Control	Enzyme treated pulp	
		Xylanase-1	Xylanase-2
Enzyme dose, % (OD pulp)	0	0.04	0.06
Treatment Time , (hrs)	1.0	1.0	1.0
Temperature , ° C	50	50	50
Consistency of the pulp , %	8.0	8.0	8.0
pH	8.0	8.0	8.0

Results & discussion

Table- 8 Effect of Enzyme Treatment on Unbleached Pulp			
Particulars	Control	Enzyme treated pulp	
		Xylanase-1	Xylanase-2
Brightness, % ISO	23.0	23.64	23.58
Kappa number	19.94	19.57	19.16

Effect of enzyme on brightness of the Pulp:

The results of the enzymatic prebleaching studies with SPB pulp conducted at CPPRI are similar to the results of the enzymatic studies conducted at R & D lab of SPB.

Data shown in the table-9 indicated that the enzyme treated pulps could be bleached to higher brightness with a gain in brightness level of 1.5-2.0 % ISO in both sets while using similar chlorine dosages as in case of control pulp sample.

Effect of xylanase treatment on bleach chemical requirement:

Xylanase treated pulps differ in response to bleach chemical when compared to untreated pulps. Bleaching of the control and enzyme treated pulps using conventional CEHH bleach sequence showed remarkable reduction 16.5% in the requirement of total chlorine i.e from 7.3 % to 6.1% whereas elemental chlorine which is reduced from 4.8 (48.00 kg/tp) to 4.1% (41.0 kg/tp) and hypo demand is reduced from 2.5% to 2.0 % (20%) in case of enzyme treated pulps while maintaining the targeted brightness level of 83.5. Results are shown in the table – 9. Overall impact of enzymatic bleaching is shown in table-10. There was considerable decrease in yellowness of enzyme treated pulps compared to the untreated pulp.

Table : 9 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CEH Sequence					
Particulars	Control	Xylanase-1		Xylanase-2	
		ET1	ET2	ET1	ET2
Chlorination stage					
Applied chlorine, %	4.8	4.8	4.1	4.8	4.1
Consumption , %	96.3	90.4	97.5	94.4	96.1
Savings in chlorine , %	--	--	14.6	--	14.6
Kappa number	6.4	6.1	6.4	6.5	6.5
Brightness, %	36.0	36.7	36.7	37.07	36.5
Alkali Extraction stage					
Applied NaOH, %	1.8	1.8	1.8	1.8	1.8
Peroxide , %	0.5	0.5	0.5	0.5	0.5
Alkali Consumption, %	84.4	84.8	83.6	86.39	88.39
CE brightness, %	51.44	50.81	50.89	52.55	52.53
CE kappa number	2.87	2.87	2.87	3.08	2.87
Hypo stage					
Applied Hypo, %	2.5	2.5	2.0	2.5	2.0
Savings in Hypo , %	--	--	20	--	20
Consumption , %	99.5	99.7	99.4	98.5	98.7

Final brightness of the pulp, % ISO	83.7	84.7	83.6	85.4	84.7
Post Colour Number	2.75	1.17	1.66	2.01	1.16
Yellowness , %	8.41	8.08	8.4	7.40	7.50

TABLE – 10 OVERALL IMPACT OF ENZYMATIC PREBLEACHING ON HARD WOOD PULPS

Parameters	Xylanase-1	Xylanase-2
Total Cl₂ reduction , %	16.5	16.5
Elemental Cl ₂ reduction , %	14.6	14.6
Hypo Reduction, %	20	20
Final Brightness improvement , % ISO (with same 16.5 % less Cl₂ dose of control)	--	1.0
Final Brightness improvement , % ISO (with same Cl₂ dose as of control)	1.0	1.8

Effect of xylanase treatment on strength & optical properties of the pulp :

Strength properties of both enzyme treated pulps showed improvement when compared with the untreated pulps. Results are shown in table – 11. Determination of the strength properties of both enzyme treated pulps bleached with same Cl₂ dose, showed that burst, tensile, and tear index could be slightly improved which were found to be 3.0 and 3.1 Kpa .m²/g against 3.2 Kpa .m²/g of control and 52.0 and 56.5 N.m/g against 52.5 N.m/g and 3.7 & 3.6 mN.m²/g against 3.6 mN.m²/g of control in case of enzyme treated pulps respectively with Xyl – 1 and Xyl – 2 respectively.

There is a significant improvement in strength properties could be noticed in enzyme treated pulp bleached with less Cl_2 dose. The burst index was increased from 3.2 Kpa .m²/g of control to 3.6 & 3.5 Kpa .m²/g of both the enzyme treated pulps. Tensile & tear indexes of the enzyme treated pulps were improved considerably when compared to the control. i.e from 52.5 N.m/g to 60.0 & 58.0 N.m/g and 3.6 mN.m²/g to 4.2 & 4.1 mN.m²/g.

Table - 11 Strength & Optical Properties of Wood Pulp Before & After Enzyme Treatment

Particulars	Control	Xylanase -1		Xylanase -2	
		ET1	ET2	ET1	ET2
Strength Properties					
Revolution , PFI	2000	2000	2000	2000	2000
Freeness, CSF	205	185	225	200	225
Apparent density, g/m ³	0.88	0.88	0.87	0.91	0.84
Burst Index, Kpa.m ² /g	3.20	3.00	3.60	3.10	3.50
Tensile Index , Nm/g	52.5	52.0	60.0	56.5	58.0
Tear Index , Mnm ² /g	3.60	3.70	4.20	3.60	4.10
Optical properties					
Opacity, %	79.6	78.2	80.1	77.9	78.9

Environmental effect of enzyme treatment :

Reduction in total chlorine demand of around 16% during CE(P)HH bleach sequence resulted in lowering the toxicity of the bleach plant effluent in enzyme treated pulps remarkably. From the results shown in table-12 it is evident that the AOX level in the bleach effluent generated from enzyme treated pulps was reduced to 20%. i.e from 2.88 to 2.27 Kg/tp.

Table - 12 Characterization of Bleach Effluent (C+E +H Stage) Before & After Enzyme Treatment of Pulp					
Particulars	Control Pulp	Xylanase -1		Xylanase-2	
		ET1	ET2	ET1	ET2
AOX , Kg / tp	2.88	2.80	2.33	2.89	2.27
AOX Reduction , %	--	--	19.1	--	21.18
COD , Kg / tp	52.5	55.5	58.82	57.34	56.75
BOD, Kg / tp	25.18	28.32	28.74	29.17	29.1
COD: BOD Ratio	1:2.1	1:1.96	1: 2.05	1: 1.97	1:1.95

Optimisation of Enzyme doses:

To see the effect of low doses of the enzyme on SPB hardwood pulp, experiments were conducted with the enzyme which showed better results, Xyl-2. The process conditions and results are shown in the tables – 13 to 15. Results indicated that there was no brightness improvement in enzyme treated pulps with 0.02 & 0.04% doses when compared to the control but there was total chlorine reduction of 14.5 % in enzyme treated pulps maintaining the untreated pulp brightness level.

Table - 13 Enzyme (Xylanase -2) Pretreatment Conditions			
Particulars	Control	Enzyme treated pulp	
Enzyme dose, % (OD pulp)	0	Enzyme dose 0.02 %	Enzyme dose 0.04 %
Treatment Time , (hrs)	1.0	1.0	1.0
Temperature ,° C	50	50	50
Consistency of the pulp , %	8.0	8.0	8.0
pH	8.0	8.0	8.0

Table – 14 Effect of Enzyme (Xylanase -2) Treatment on Unbleached Pulp			
Particulars	Control	Enzyme treated pulp	
		Enzyme dose 0.02 %	Enzyme dose 0.04 %
Brightness, % ISO	22.93	23.02	22.96
Kappa number	20.79	20.2	20.12

Table : 15 Effect of Enzyme (Xylanase –2) Treatment on Hard Wood Pulps Bleached With CEHH Sequence					
Particulars	Control	Enzyme dose 0.02 %		Enzyme dose 0.04 %	
		ET1	ET2	ET1	ET2
Chlorination stage					
Applied chlorine, %	4.8	4.8	4.1	4.8	4.1
Consumption , %	98.09	97.13	98.88	98.09	98.32
Savings in chlorine , %	--	--	14.6	--	14.6
Alkali Extraction stage					
Applied NaOH, %	2.5	2.5	2.5	2.5	2.5
Peroxide , %	0.5	0.5	0.5	0.5	0.5
Alkali Consumption, %	92.52	87.3	85.3	86.76	85.04
Hypo stage -1					
Applied Hypo, %	2.5	2.5	2.0	2.5	2.0
Savings in Hypo , %	--	--	20	--	20
Consumption , %	94.64	93.88	94.55	94.36	96.49
Final brightness of the pulp, % ISO	76.51	79.47	78.36	78.87	74.27
Hypo stage -2					
Applied Hypo, %	1.0	1.0	1.0	1.0	1.0
Consumption , %	89.8	80.2	80.8	85.9	80.8
Final brightness of the pulp, % ISO	82.7	82.6	82.5	83.6	82.4

5. Observations

- * **Bench scale experiments on enzymatic prebleaching were conducted with two identified xylanase enzymes to see the response of the pulp produced at Seshasayee Paper & Boards Ltd., Tamilnadu.**
- * **The results are encouraging .The final pulp brightness improvement is up to 1.52. in enzyme treated pulps with same chlorine dose as that of control pulps .**
- * **The Savings in total Cl_2 demand is 16.5% & in elemental chlorine is 16% while targeting the brightness 83.0 % ISO.**
- * **Discussions are made with mill officials regarding the process conditions Selection of equipment for the mixing of enzyme at HD tower will be selected after the techno economical evaluation by CPPRI Scientists & mill technocrats.**
- * **Mill personnel suggested for one-month trial for the assessment of techno economical viability of the process in mill scale.**

6. Further Action Plan

- ♠ **CPPRI needs large amount of commercial enzyme for one month's trial & and to tie up with suppliers for the supply of enzymes and finalise the terms & conditions etc.**
- ♠ **Selection and procurement of equipment needed for the implementation of this technology in the existing SPB's Process lay out.**

Chapter – IV

Selection & Procurement of Equipment And Enzyme For Mill Trial at SPB Ltd.

1. About the mill

Seshasayee Paper and Boards Limited (SPB), the flagship company belonging to 'ESVIN GROUP', operates an integrated pulp, paper and paper board Mill at Pallipalayam, Erode - 638 007, District Namakkal, Tamilnadu, India with 1,15,000 tonnes per annum installed capacity. It is a 'ISO 9001' & 'ISO14001' certified company, markets almost all varieties of paper boards & speciality grades and is a significant exporter in the Indian Paper Industry. Under the expansion /modernization project of SPB, the company showed very keen interest for the demonstration of enzymatic prebleaching technology.

The mill uses wood, bagasse, waste paper & purchased pulp for making of various grades of specialty papers, writing & printing grade, packing grade etc. A considerable amount of pulp was made from wood i.e Eucalyptus and Casuarina. Brown stock pulp is washed through vacuum washers and screened prior to thickness in high density storage and bleaching. The bleach sequence used was CE(p)1E(p)2H. The total chlorine used for Wood Street is high so mill wants to reduce the amount of total chlorine through enzymatic bleaching route. So it was decided to implement enzyme prebleaching technology in Wood Street. The process layout of Wood Street of SPB is shown in fig .2 Flow sheet of process layout of incorporation of equipment of enzyme technology is shown in fig. 3.

For the successful mill trials, there are two activities which play important role. As per the recommendations of the first visit, discussions are made with mill officials

regarding the process conditions and selection of equipment for the mixing of enzyme at HD tower were selected after the techno-economical evaluation by CPPRI Scientists & SPB Technocrats.

a. Selection of equipment : The most appropriate equipment for thorough mixing of enzyme with the pulp.

b. Selection of Enzyme : Selection of cost effective indigenous enzyme with high bleach efficiency.

2. Selection & Procurement of Equipment for mill trial

The equipment needed for mill trial of enzymatic prebleaching were selected and has been procured.

The following equipment were procured as per specifications given by SPB.

1. Medium consistency Pump
2. Motor for MC pump
3. Enzyme dosing pump
4. Stand pipe
5. Vacuum pump
6. Accessories like Cables & Switches etc.

1. Medium consistency Pump

Specifications: (Given by SPB)

Production rate	: 150 ADMT/day
Pressure	: 5-6 kg/cm ²
Speed	: 1500
Consistency	: 10-12%

Material of Construction

- All Wetted Parts : CF8M
- Bearing Housing : Cl Gr 25
- Sleeve : AISI 316
- Shaft : AISI 410

Medium Consistency Pump : 1Piece

S. No	Description of the items
1.	Medium Consistency Pump having following specification : Liquid handled : pulp Temperature in ° C : Ambient(Assumed) Specific gravity : 1.0 (Assumed) Suction condition (Assumed) : Flooded Capacity in M ³ /Hr : 60 Pump Type / Model : TPS+H100/430 Size in mm : 200x100 Motor power in KW : 75 Speed in RPM : 1550 Material of Construction Casing : ASTM A743 CF8M (SS-316) Impeller : ASTM A743 CF8M (SS-316) Wear Ring : ASTM A743 CF8M (SS-316) Pump Shaft/Shaft Sleeve : AISI 410/AISI 316 Pump Shaft/Shaft Sleeve: AISI 410/AISI 316 Shaft Sealing : Mechanical Seal Brg. Bed& other Part : S.G. Iron & STD.MOC
2.	Mechanical Seal

2. Specifications for Motor for MC pump

S. No	Particulars	Specifications
1	Manufacturers	Alstom Ltd, Bharat Bijli, Kirloskar Cummins, Crompton creaves
2	Type	Horizontal centrifugal
3	Motor type	Squirrel Cage
4	Frame size & Type designation	D250M
5	Applicable standard to which motor confirms	IS 325
6	Rated out put	100HP/75KW

7	Type of enclosure & method of cooling	TEFC/1C411
8	Degree of protection	IP 55
9	Rate voltage & Frequency	415 V / 50HZ
10	Rated speed	1470 RPM
11	Power factor at designed duty point (at 100%/75%/50% load)	0.86 / 0.85 / 0.78
12	Type of mounting	Foot / IMBB
13	Bearing type	DE - NU217 NDE - 6313C3



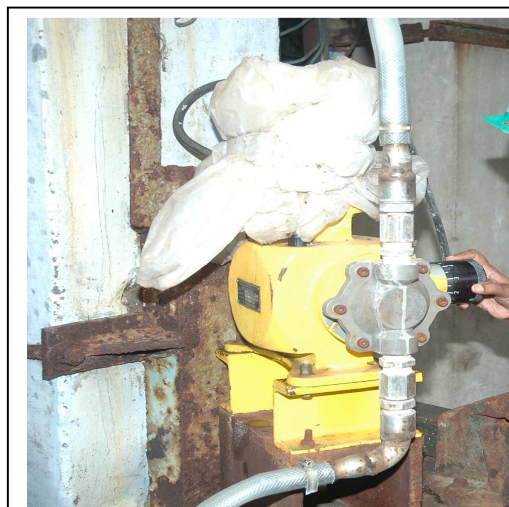
MC Pump



Motor for MC Pump

3. Enzyme dosing pump:

Enzyme dosing pump was purchased.
It is used to add enzyme in desired amounts
maintaining different flow levels.



Enzyme dosing pump

4. Stand Pipe:

Stand pipe was purchased according to the specifications given by SPB Ltd. and installed after MC pump for pulp passage after addition of enzyme.



Stand pipe

5. List of accessories for Mill trial - SPB:

Some spares and accessories needed for the implementation and running of the procured equipment are listed below.

S.No	Item Description	Quantity
1	Motor for Vacuum pump (5HP/1400RPM)	1NO.
2	Power Guard Sfu 250 Amps In Steel Enclosure 3 Kl-25-37-1 Yaaa	1 No.
3	L & T Make M10 Star delta Starter With Copper Busbar With Mniz Relay 60-100 Amps With Fn 250 A Switch Fuse Unit With Ammeter And Voltmeter With Steel Enclosure. 41Svac Suitable For 100 Hp Motor.	1 No.
4	Pvc Armored Aluminium Cable 3 Core X 70 Sqmm 1100v Grade.	100 Mr
5	Pvc Armored Copper Cable 3 Core X 1.5 Sqmm 1100v Grade	100 Mr
6	Flange Type Cable Gland 70 Sqmm.	8 No.
7	Flange Type Cable Gland No.4 Size 3/4"	4 No.

8	Pushbutton Actuator With Plastic Ring And Holder Code 3 Sb 1200-Oae 01 Green Colour.	2 No.
9	Push Button Actuator With Plastic Ring And Holder Code 3 Sb 1200-Oac01 Red Colour.	2 No.
10	Push Button Contact Element 1 No + I Nc Rear Type 3 Sb 1400-Oa.	4 No.
11	Led Type Indication Fitting 230 V Ac 22.5mm Dia. Red Colour.	1 No.
12	Led Type Indication Fitting 230 V Ac 22.5mm Dia. Green Colour.	1 No.
13	Concard Make Lockable Stop Button Station With Strovd.	1 No.
14	Pvc Armovred copper Cable 3 Core X 1.5 Sqmm 1100v Grade.	175mt.
15	Pvc Armovred copper Cable 3 Core X 2.5 Sqmm 1100v Grade.	100mts.
16	FN 400 A Switch with enclosure – L& T make	1No.
17	70 Sqmm. PVC insulated 3core armoured aluminum cable	100mts.
18	120 SQ.mm PVC insulated 3 core armoured aluminum cable	150mts.

All the equipment were received at SPB & installation has been done at mill site.

Installation of the procured equipment

Two Scientists of CPPRI visited the identified mill -- M/s Seshasayee Paper & Boards Ltd., to see the equipment procured and for the installation of the same in the mill.

3. Selection of Enzyme For Mill Trial

Procurement of Enzymes And Evaluation of Efficiency of Enzymes on Hard Wood Pulp

- During the visit, mill personnel suggested for one month trial for the assessment of techno economical viability of the process in mill scale.

- CPPRI needs large amount of commercial enzyme for one month's trial & wants to tie up with suppliers for the supply of enzymes and finalise the terms & conditions etc. for long term association.
- CPPRI published a advertisement in Hindu Newspaper to extend the wide publicity among nationwide enzyme suppliers for the selection of a better supplier on the basis of quality & cost of the enzyme.

Advertisement



CENTRAL PULP & PAPER RESEARCH INSTITUTE

POST BOX NO. 174, PAPER MILL ROAD, HIMMAT NAGAR, SAHARANPUR – 247001 (U.P.) INDIA

Tel. EPABX (0132) 2725317, 2722756, Direct 2727227, 2726834, 2726794

Fax (0132) 2727387, 2721367 website: www.cppri.org e-mail: director@cppri.org

Central Pulp & Paper Research Institute is the premier R & D organisation in pulp & paper sector and caters to the need of entire paper industry in India and South East Asia. The Institute has ventured into frontier technologies and Biotechnology is essentially one of them. Institute promotes Biotechnological applications in Pulp & Paper Industry in order to improve the product quality as well as for environmental protection. Enzymatic prebleaching is the thrust area and Institute plans to demonstrate enzymatic prebleaching in some leading mills for extended periods upto one month to create greater degree of confidence in the industry and also to evaluate the benefits accrued over a period of time. CPPRI invites proposals from enzyme manufactures and suppliers for tie up with CPPRI for supply of enzymes for studies on mill scale and for future cooperation to promote enzymatic prebleaching on mutually agreeable terms. For any details required please contact Director CPPRI on the above address. Interested parties may send the details relating to complete company profile, production facilities at supplier's end, terms and conditions of supply, willingness for long-term association and any other relevant

information within two weeks of publication of the advertisement, addressed to Director CPPRI.

Interaction with Enzyme Suppliers:

Further, Correspondence also has been made with the reputed commercial enzyme manufacturers, for supply of xylanase enzyme suitable for prebleaching of pulps on plant scale along with the enzyme specifications and cost of the enzyme.

- CPPRI received good response and proposals with detailed information about quality & cost of the enzymes from various enzyme manufacturers.

Comparative statement of the responses received for the advertisement regarding enzyme

S. No	Company & Address	Response	CPPRI Feed Back
1.	R. Ramamurthy 1254,Nadar Colony Trichy Road, Coimbatore-641018	Interested in development of enzymatic prebleaching of pulp and to produce and market the enzyme, requests for details on the enzyme used and guidance in this venture.	---
2.	P.N. Balaji Colour - Chem. Ltd. Delhi.	Interested in participating in development of enzyme prebleaching process. They will send the details on our request.	Considered
3.	U.V. Satyanarayana Dy. General Manager (Tech) APPM	Requested to provide the procedure for estimation the enzyme activity.	---
4.	Arvind Kumar Sharma ANMOL POLYMERS Pvt. Ltd.	Authorized distributors of Biocon (I) Ltd. They are leading manufacturer and suppliers of paper chemicals in India. So if CPPRI is interested then they can	Considered

		depute somebody to visit Saharanpur to discuss further in this regard.	
5.	C. Ravindran Partner Brewers nest 13/2,chandrabagnggh avenue , Mylapore Chennai-600004	The supplier have enquired <ul style="list-style-type: none"> • Brief note on the process of manufacture • CPPRI terms and conditions • Can they over there, prepare samples and take up trials and based on the feedback from various mills can they start commercializing it 	---
6.	Surajit Ray General manager Sharda@jkmail.com	They wish to know weather there is any contact made with JK Pharmachem in the regard of supply of enzyme. Received Jkzyme from JK Pharmachem.	---
7.	M.K. Chatterji Advanced Biochemicals Ltd.	Details about their enzyme - <ul style="list-style-type: none"> • Company`s capability for supply of xylanase enzyme : any quantity up to 5MT at a time • Minimum time required for supply : 2 weeks • Enzyme activity : 6000U/g • Enzyme stability : 6 months • Dosage per ton of pulp : 300-500g/tn • Optimal condition : • pH : 7.0-8.0 • Temp.: 60 °C • Retention time: 2.5-3.0 hrs. 	Considered

		<ul style="list-style-type: none"> Cost of enzyme: Rs.200/-per kg. 	
8.	Alok Kumar Gupta Managing Director Value addition Papers Private Limited (representative of Novozymes)	Regarding plant scale trail in one of the Indian paper mills (Capacity 150MT/Day) for one month, they recommend their Pulpzyme HC. The total quantity required for this trail will be around 2250 kg. They will supply this quantity at one week notice.	Considered
9	C.V. Seshadri DGM – Marketing J.K. Pharma chem Limited Khivraj Complex – II , 5 th Floor, 480, anna salai, Nandanam , Chennai – 600 035	Keen to associate with CPPRI and to the details of the bleaching enzyme JKZYME had been enclosed.	Considered
10	T.S. Venkatraman Managing Director Esvin Advanced technologies limited , Esvin house, Porungudi, Chennai	Requested to register their company as the supplier of the enzyme and they will send the detailed proposal within a week's time.	Considered

- Final short listing of the enzyme suppliers was made on the basis of effectivity and cost of the enzyme.

Xylanase Enzyme details from various selected enzyme manufacturers

Particulars	Enzyme Manufacturer / Supplier					
	Colour Chem Ltd. , Delhi	Anmol Polymers pt. Ltd. (Biocon)	JK Pharma chem. Ltd.	Value addition Papers Pt. Ltd, (Novozyme)	Advanced Biochem. Ltd. (Sebritte BB)	Esvin Advanced technologies limited
Evaluated by CPPRI	Yes	Yes	No	Yes	Yes	Yes
CPPRI Experience	Good	Good	--	Good	Good 1.0 b% & 15% Cl ₂ reduction	Not good
Enzyme Description	Will send the details soon	No Product specifications	Aqueous solution	Dr. R. K. Jain has got all the technical information	Powder form	Will send the details the details with in a week time
Enzyme Activity , IU/g or IU/ml			1000IU/ml		6000 IU/g	
Solubility			Completely soluble in water		Partially soluble in water	
Colour			Yellow brown liquid		Cream	
Material safety data sheet			included		Not included	
Storing conditions			Less than 25 °C		Not mentioned	

Enzyme stability	Will send the details soon	No Product specifications	90% of enzyme activity at pH – 10.0 More than 60% activity is retained when exposed to temp.70 ° c		6 months	
pH optimum pH active range			5.5 5.0-9.0		7-8	
Temperature , °C			50-70 °C		60 °C	
Retention time , h			1.0		2.5-3.0	
Enzyme dosage IU/g or %			2 IU/g		0.03 – 0.05 %	
Enzyme response on different pulps			Softwood , Hardwood & agro pulps		Hard wood kraft pulp	
Enzyme dose required for ton pulp			2kg		300 – 500 g	
Enzyme requirement per day (140t/day) , kg			280kg		42-70 kg	
Enzyme requirement for one month trial			8680 kg		1302 - 2170	

Minimum time requirement for supply of the enzyme	Will send the details soon	No Product specifications	NM		Two weeks	
Enzyme cost per Kg, Rs.					200 /-	
Enzyme cost per one month trial					2.6 - 4.34 lacs	
Bleach cost per ton of pulp without enzyme						
Bleach cost per ton of pulp with enzyme						
Other information	Interested to tie up	Will depute somebody to CPPRI for detailed discussions	Interested to work out the details and enter in to a MOU.	Seeking a suitable date for detailed discussions.	Interested to tie up with CPPRI	Requested to register their company's name as the supplier of enzyme

Evaluation of Selected Enzymes - Enzymatic Prebleaching Studies of Hard Wood Pulps

Xylanase enzymes received from various enzyme suppliers were evaluated on hardwood pulps obtained from Star Paper Mills Ltd., Saharanpur and SPB Ltd., Erode.

Case study – 1

Pulp Sample Collected	: Hard wood: Bamboo based mill
Enzyme - A	: Jkzyme (J. K. Pharmaceuticals Ltd.)
Enzyme - B	: Alkaline Xylanase (Esvin technologies Ltd)

Table –1 Enzyme Pretreatment Conditions			
Particulars	Control	Enzyme treated pulp	
		JKZYME Enzyme - A	ESWIN Enzyme - B
Enzyme dose , kg/t	--	2.0	0.3
Treatment time (min.)	60	60	60
Temperature , °C	50	50	50
Consistency , %	8.0	8.0	8.0
pH	8.4 - 8.6	8.4 - 8.6	8.4 - 8.6

Enzyme treated and untreated pulps were evaluated for brightness and kappa number. Effluents were analysed for colour, lignin and xylose sugars. After enzyme treatment pulps were subjected to conventional bleaching.

Table –2 Unbleached pulp Characteristics			
Particulars	Control	Enzyme treated pulp	
		Enzyme - A	Enzyme - B
Kappa Number	16.7	16.1	15.0
Characteristics of water extracts of enzyme treated pulps			
Colour, Kg/tp	20.75	23.08	25.33
Lignin, kg/tp	1.58	1.99	2.02
Reducing sugars as xylose , kg/tp	2.53	2.58	2.67

Table – 3 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CE(P)H Sequence					
Particulars	Control	Enzyme treated pulp			
		JKZYME Enzyme - A		ESWIN Enzyme - B	
		ET1	ET2	ET1	ET2
Chlorination stage					
Applied chlorine, %	3.5	3.5	3.0	3.5	3.0
Consumption , %	96.7	99.4	97.7	98.0	98.5
Elemental Chlorine Savings, %	--	--	14.3	--	14.3
C- Kappa Number	5.6	4.0	6.0	4.0	4.25
Alkali Extraction stage E(p)					
Applied NaOH, %	2.3	2.3	2.3	2.3	2.3
Peroxide applied, %	0.5	0.5	0.5	0.5	0.5
Consumption , %	61.0	56.3	49.4	31.0	54.0
CE brightness , %					
CE Kappa number	2.76	2.8	2.76	2.8	3.0
Hypo stage					
Applied Hypo, %	2.0	2.0	2.0	2.0	2.0
Consumption , %	70.6	63.3	57.1	61.6	61.2
Final brightness of the pulp, % ISO	84.37	84.09	82.63	84.96	83.56
Brightness Improvement , % ISO	--	--	--	0.59	--
Total chlorine applied , %	5.5	5.5	5.0	5.0	5.0
Ele. chlorine savings, %	--	--	14	--	14

ET1: Enzyme treated pulps bleached with same Cl₂ dose as of control

ET2: Enzyme treated pulps bleached with less Cl₂ dose as of control

Observations:

The results of the enzymatic prebleaching of kraft hard wood pulps procured from star paper mills ltd. are

- Gain in final brightness only 0.6.
- Savings of elemental chlorine to the tune of 14%.

Both the evaluated enzymes did not show any brightness improvement, but they reduce the chlorine demand by 14%.

Case study – 2

Pulp Sample Collected : Hard wood based mill

Enzyme : Alkaline xylanase (Esvin technologies Ltd.)

Table – 4 Enzyme Pretreatment Conditions		
Particulars	Control	ESVIN enzyme treated pulp
Enzyme dose , l/t	--	0.5
Treatment time (min.)	60	60
Temperature , °C	65	65
Consistency , %	8.0	8.0
pH	8.6	8.6

Table – 5 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CE(P)H Sequence				
Particulars	Control	Enzyme treated pulp		
		ET1	ET2	
Chlorination stage				
Applied chlorine, %	3.5	3.5	3.0	
Consumption , %	96	96	99	
Elemental Chlorine savings , %	--	--	14	
Alkali Extraction stage E (p)				
Applied NaOH, %	2.3	2.3	2.3	
Peroxide applied, %	0.5	0.5	0.5	
Consumption , %	64	60	64	
Hypo stage				
Applied Hypo, %	2.0	2.0	2.0	1.5
Consumption , %	53	49	44	48
Final brightness of the pulp, % ISO	81.1	84.3	84.4	83.4
Brightness Improvement , % ISO	--	3.2	3.28	2.28
Total chlorine applied , %	5.5	5.5	5.0	4.5
Total chlorine savings , %	--	--	10	18
Ele. chlorine savings	--	--	14	14

ET1: Enzyme treated pulps bleached with same Cl₂ dose as of control

ET2: Enzyme treated pulps bleached with less Cl₂ dose as of control

Evaluated xylanase showed good response both in terms of brightness improvement and reduction in chlorine demand.

Case study – 3

Pulp Sample Collected : Hard wood: Bamboo based mill
Enzyme - A : Jkzyme (J. K. Pharmaceuticals Ltd.)
Enzyme - B : Pulpzyme HC (NovoZyme)

Table – 6 Enzyme Pretreatment Conditions			
Particulars	Control	Enzyme treated pulp	
		JKZYME Enzyme - A	PULPZYME Enzyme - B
Enzyme dose , kg/t	--	2.5	0.7
Treatment time (min.)	120	120	120
Temperature , °C	50	50	50
Consistency , %	8.0	8.0	8.0
pH	9.1	8.9	8.9
Unbleached pulp			
Brightness , % ISO	30.5	30.8	31.6

Table –7 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CE (P)H Sequence					
Particulars	Control	Enzyme treated pulp			
		Enzyme - A		Enzyme - B	
		ET1	ET2	ET1	ET2
Chlorination stage					
Applied chlorine, %	3.5	3.5	3.0	3.5	3.0
Consumption , %	99.0	98.7	99.2	99.01	96.2
Elemental Chlorine Savings , %	--	--	14.3	--	14.3
Alkali Extraction stage E (p)					
Applied NaOH, %	2.3	2.3	2.3	2.3	2.3
Peroxide applied, %	0.5	0.5	0.5	0.5	0.5
CE brightness , %	54.1	55.3	52.0	56.1	53.2
Hypo stage					
Applied Hypo, %	2.0	2.0	2.0	2.0	2.0

Consumption , %	70.2	68.6	70.2	67.4	64.1
Final brightness of the pulp, % ISO	79.4	80.2	78.3	80.9	79.4
Brightness Improvement , % ISO	--	0.8	-1.0	1.5	--
Total chlorine applied , %	5.5	5.5	5.0	5.0	5.0
Ele. chlorine savings	--	--	14	--	14

ET1: Enzyme treated pulps bleached with same Cl₂ dose as of control

ET2: Enzyme treated pulps bleached with less Cl₂ dose as of control

Case study – 4

Pulp Sample Collected Hard wood and Bamboo (90:10) — Star Paper Mill

Kappa No. – 16.0

pH– 7.8

Enzyme – A - JKZyme-1

Enzyme – B - JKZyme-2

Enzyme – C - Pulpzyme

Table – 8 Enzyme Pretreatment Conditions of Hard wood pulp pH –7.8				
Particulars	Control pH (7.8)	Enzyme Treated pulp		
		Enzyme A	Enzyme B	Enzyme C
Enzyme Dose g/t	---	2.5	2.5	2.5
Treatment time (min.)	120	120	120	120
Temperature, C	50	50	50	50
Consistency, %	10	10	10	10
Unbleached Pulp				
Brightness, % ISO	29.0	29.4	29.9	29.7
Kappa No.	13.4	12.3	12.9	12.8
Water Extracts				
Colour (kg/ton)	13.4	22.6	15.6	16.5
Lignin (kg/ton)	2.01	1.75	1.70	1.80
Reducing Sugars, Xylose (kg/ton)	0.34	0.44	0.38	0.45

Table – 9 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CE (P) HH Sequence (7.8 pH)				
Particulars	Contr ol	Enzyme Treated pulp		
		Enzyme-A	Enzyme-B	Enzyme -C
Chlorination				
Applied Chlorine%	3.5	3.2	3.2	3.2
Consumption%	91.4	91.4	93.5	92.8
Alkali Extraction Stage E (p)				
Applied NaOH, %	3.0	3.0	3.0	3.0
Peroxide applied	0.5	0.5	0.5	0.5
CE brightness	58.1	59.3	59.3	59.5
CE μ Kappa No.	4.2	4.04	3.9	4.0
Hypo stage 1				
Applied Hypo, %	1.5	1.5	1.5	1.5
Consumption, %	96.6	93.6	93.5	94.5
Brightness, % ISO	76.9	79.9	81.3	81.7
Hypo stage 2				
App. Hypo, %	0.6	0.6	0.6	0.6
Consumption, %	84	73.3	69.2	75.5
Final brightness,%	82.5	83.7	84.7	84.9
Brightness improvement, %	--	1.2	2.2	2.4
Viscosity (cm/gm)	269	241	232	244

Enzyme B & C were further evaluated at pulp pH 9.0 showed good brightness improvement.

Table –10 Enzyme Pretreatment Conditions of Hard wood pulp pH-9.0			
Particulars	Control pH (9.0)	Enzyme Treated pulp	
		Enzyme B	Enzyme C
Enzyme Dose, kg/t	---	2.5	2.5
Treatment time, (min.)	120	120	120
Temperature, C	50	50	50
Consistency, %	10	10	10
Unbleached Pulp			
Brightness, %ISO	30.48	30.43	30.47
Kappa No.	13.4	12.8	12.8
Water Extracts			

Colour (kg/ton)	20.8	23.68	29.07
Lignin (kg/ton)	2.33	2.26	2.43
Reducing Sugar, (kg/ton)	0.51	0.59	1.22

Table – 11 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CE (P) HH Sequence (9.0 pH)			
Particulars	Control (9.0pH)	Enzyme Treated Pulp	
		Enzyme - B	Enzyme - C
Chlorination			
Applied Chlorine%	1.75	1.75	1.75
Consumption%	90.2	89.5	90.8
Alkali Extraction Stage E (p)			
Applied NaOH, %	3.0	3.0	3.0
Peroxide applied	0.5	0.5	0.5
CE brightness	56.7	60.0	59.8
CE μKappa No.	4.0	3.9	3.9
Hypo stage 1			
Applied Hypo, %	1.5	1.5	1.5
Consumption, %	96.0	94.5	93.5
Brightness, % ISO	77.8	81.1	81.9
Hypo stage 2			
App. Hypo,%	0.6	0.6	0.6
Consumption, %	82.6	84.7	84.7
Final brightness,%	82.5	84.7	84.7
Brightness improvement, %	--	2.0	2.0
Viscosity (cm/gm)	239	248	236

At pH 9.0, Enzyme B and C showed brightness improvement i.e 2.0 unit.

Case study – 5

Effect of two xylanase treatments before chlorination and after alkali extraction stage were evaluated on hardwood pulp with the pulpzyme enzyme.

Pulp Sample Collected Hard wood and Bamboo (90:10) — Star Paper Mill

Kappa No. – 16.0

pH– 7.8

Enzyme – Pulpzyme

Table – 12 Enzyme Pretreatment Conditions of Hard wood pulp pH –7.8(1st stage)				
Particulars	Control pH (7.8)	Enzyme Treated pulp		
		XCEXH	XCEXEH	XCEH (Less Chlorine)
Enzyme Dose, kg/t	---	1.5	1.5	2.5
Treatment time , (min.)	120	120	120	120
Temperature, C	50	50	50	50
Consistency, %	10	10	10	10
Unbleached Pulp				
Brightness, %ISO	29.2	29.5	29.5	29.7
Kappa No.	13.6	13.1	13.4	13.3
Water Extracts				
Colour, (kg/ton)	7.2	9.7	8.2	8.5
Lignin, (kg/ton)	1.0	1.3	1.1	1.1
Reducing Sugar, (kg/ton)	0.3	0.4	0.3	0.3

Table –13 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CEH Sequence (7.8 pH)				
Particulars	Control	Enzyme Treated pulp		
		XCEXH	XCEXEH	XCEH (Less Chlorine)
Chlorination				
Applied Chlorine%	3.0	3.0	3.0	3.0
Consumption%	90.0	92.0	90.6	92.0
Alkali Extraction Stage E ₁				
Applied NaOH, %	2.0	2.0	1.5	2.0
CE brightness, %	44.3	45.6	45.2	43.7
CE μ Kappa No.	4.6	4.8	5.0	4.8
Enzyme Pretreatment 2 nd stage				
Particulars	Control pH (7.8)	Enzyme Treated pulp		
		XCEXH	XCEXEH	XCEH (Less Chlorine)

Enzyme Dose, kg/t	--	1.0	1.0	--
Treatment time, (min)	--	120	120	--
Temperature, C	--	50	50	--
Consistency, %	--	10	10	--
Water Extracts				
Colour, (Kg/ton)	--	4.3	4.4	--
Reducing Sugar , (Kg/ton)	--	0.2	0.3	--
Alkali Extraction Stage E₂				
Applied NaOH, %	--	--	0.5	--
Brightness, %	--	--	49.55	--
Hypo stage				
Applied Hypo, %	2.0	2.0	2.0	2.0
Consumption, %	84.5	77.5	72.0	76.0
Final Brightness, % ISO	73.1	74.7	74.5	70.7
Brightness improvement, %	--	1.6	1.4	2.4

Xylanase treatment after alkali extraction stage did not show any effect/improvement on final brightness of pulp.

Case study – 6

Pulp Sample Collected Hard wood and Bamboo (90:10)—Star Paper Mill

Kappa No. – 16.0

pH– 7.8

Enzyme – Eswinzyme from Eswin Technologies, Chennai

Table –14 Enzyme Pretreatment Conditions of Hard wood pulp pH –7.8			
Particulars	Control pH (7.8)	Enzyme Treated pulp	
		Same Chlorine	Less Chlorine
Treatment time, (min.)	60	60	60
Temperature, C	70	70	70
Consistency, %	10	10	10
Unbleached Pulp			
Brightness, % ISO	29.4	29.8	30.5
Kappa No.	13.7	13.2	13.1

Water Extracts			
Colour, (kg/ton)	8.34	16.0	18.5
Lignin, (kg/ton)	0.8	1.2	1.2
Reducing Sugar , (kg/ton)	0.3	0.4	0.4

Table – 15 Effect of Enzyme Treatment on Hard Wood Pulps Bleached With CE (P) HH Sequence (7.8 pH)			
Particulars	Control	Enzyme Treated pulp	
		Same Chlorine	Less Chlorine
Chlorination			
Applied Chlorine, %	3.0	3.0	2.5
Consumption%	90.6	91.7	93.0
Alkali Extraction Stage E			
Applied NaOH,%	2.0	2.0	2.0
CE brightness	40.1	47.2	45.1
CE μ Kappa No.	4.8	4.2	4.4
Hypo stage 1			
Applied Hypo, %	1.5	1.5	1.5
Consumption, %	95.4	96.6	97.1
Brightness, % ISO	75.8	77.5	75.4
Hypo stage 2			
App. Hypo, %	0.5	0.5	0.5
Final brightness,%	80.5	81.5	80.6
Brightness improvement, %	--	1.0	0.1

Case study – 7

Pulp sample collected – Hardwood pulp from Seshashayee Paper & Boards ltd.,

Erode

Kappa no. –19.0

pH – 8.5

Enzyme A – PULPZYME

Enzyme B – SEBRITE BB

Enzyme C – BIOPULP

Enzyme D – ESWINZYME

Enzyme E – J K ZYME

Table –16 Enzyme Pretreatment Conditions of hardwood Pulp						
Particulars	Contr ol	Enzyme A	Enzyme B	Enzyme C	Enzyme D	Enzyme E
Enzyme activity , (IU/ml)	--	8129	5889	7889	23422	300
Enzyme Dose, %	--	0.06	0.05	0.05	0.05	0.025
Treatment time, (min.)	90	90	90	90	90	90
Temperature, °C	50	50	50	50	50	50
pH	8.5	8.4	8.5	8.5	8.4	8.5
Water Extracts						
Colour, (Kg/ton)	52.0	64.1	54.8	61.7	57.0	53.3
Lignin, (Kg/ton)	6.1	7.5	6.2	7.5	7.1	6.8
Xylose sugars, (Kg/ton)	7.7	12.9	6.7	11.4	11.2	9.5

**Table –17 Effect of Enzyme Treatment on Hard Wood Pulp
Bleached With CE(P) HH Sequence**

Particulars	Control	Enzyme treated pulp				
		Enzyme A	Enzyme B	Enzyme C	Enzyme D	Enzyme E
Chorination						
Applied Chlorine, %	4.1	4.1	4.1	4.1	4.1	4.1
Consumption ,%	97.6	96.8	97.6	97.6	97.6	97.6
Alkali Extraction Stage E						
Applied NaOH, %	2.0	2.0	2.0	2.0	2.0	2.0
Applied Peroxide, %	0.5	0.5	0.5	0.5	0.5	0.5
Hypo Stage						
App. Hypo, %	1.5	1.5	1.5	1.5	1.5	1.5
Consumption ,%	89.3	82.7	85.2	84.9	77.0	87.5
Brightness, %	85.8	86.6	86.9	87.4	86.3	86.8
Brightness Improvement ,%	--	+0.8	+1.1	+1.6	+0.5	+1.0

Case study – 8

Pulp sample collected – Hardwood from Seshashayee Paper & Boards ltd., Erode
(Unbleached and Screened at CPPRI)

Kappa no. –19.0

pH – 8.4

Enzyme A – PULPZYME

Enzyme B – ESWINZYME - 50⁰C

Enzyme C – ESWINZYME - 70⁰C

Table – 18 Enzyme Pretreatment Conditions of hardwood Pulp (screened)				
Particulars	Control	Enzyme A	Enzyme B	Enzyme C
Enzyme activity, IU/ml)	--	8129	7889	7889
Enzyme Dose, %	--	0.06	0.05	0.05
Treatment time, (min.)	90	90	90	90
Temperature, ⁰ C	50	50	50	70
Initial pH	8.5	8.4	8.5	8.5

Table – 19 Effect of Enzyme Treatment on Hard Wood Pulp Bleached With CE(P)HH Sequence				
Particulars	Control	Enzyme A	Enzyme B	Enzyme C
Chlorination				
Applied Chlorine %	3.7	3.7	3.7	3.7
Consumption ,%	97.4	97.4	98.3	98.3
Alkali extraction				
Applied NaOH, %	2.0	2.0	2.0	2.0
Alkali Peroxide, %	0.5	0.5	0.5	0.5
Hypo stage				
App. Hypo, %	1.0	1.0	1.0	1.0
Consumption, %	94.3	94.9	94.9	94.9
Brightness ,%ISO	84.0	86.1	85.3	85.2
Brightness Improvement, %	--	+2.12	+1.37	+1.27

The brightness improvement of the pulp treated with Eswinzyme at 70 °C showed that the enzyme is active at 70 °C also.

Case study – 9

Pulp sample collected – Hardwood from Seshashayee Paper & Boards ltd., Erode
(Screening after enzyme treatment)

Kappa no. – 14.5

pH – 8.4

Enzyme A – ESWINZYME

Enzyme B – PULPZYME

Table –20 Enzyme pretreatment conditions of hard wood pulp			
Particulars	Control	Enzyme A	Enzyme B
Enzyme activity , (IU/ml)	--	7889	8129
Enzyme Dose , %	--	0.05	0.06
Treatment time, (min.)	90	90	90
Temperature, °C	50	50	50
Water Extracts			
Colour, (Kg/ton)	25.3	28.7	23.3
Lignin, (Kg/ton)	2.75	3.20	2.85
Reducing sugars , (kg/ton)	2.99	5.20	3.12

Table –21 Effect of Enzyme Treatment on Hard Wood Pulp Bleached With CE(P)HH Sequence

Particulars	Control	Enzyme A	Enzyme B
Applied Chlorine, %	3.0	3.0	3.0
Consumption %	99.0	99.3	99.3
Alkali Extraction			
Applied NaOH%	2.0	2.0	2.0
Applied Peroxide %	0.5	0.5	0.5
Hypo Stage - 1			
App. Hypo %	1.0	1.0	1.0
Brightness % , ISO	77.7	82.10	80.88
Brightness Improvement %	--	+ 4.3	+3.2
Hypo Stage - 2			
App. Hypo, %	0.5	0.5	0.5
Brightness , % ISO	81.6	84.16	82.98
Brightness Improvement %	--	+2.56	+1.4

Case study – 10

Pulp sample collected – Hard wood from star paper mill , Saharanpur

pH – 8.0

Enzyme A – Texzyme 0.025%

Enzyme B – Texzyme 0.05 %

Enzyme C – Texzyme 0.10%

Enzyme D – Texzyme 0.15%

Enzyme E – Texzyme 0.20%

Table – 22 Enzyme Pretreatment Conditions						
Particulars	Control	Enzyme A	Enzyme B	Enzyme C	Enzyme D	Enzyme E
Enzyme activity (IU/ml)	--	2000	2000	2000	2000	2000
Enzyme Dose, %	--	0.025	0.05	0.10	0.15	0.20
Treatment time, (min.)	120	120	120	120	120	120
Temperature, °C	50	50	50	50	50	50
Unbleached pulp						
Particulars	Control	Enzyme A	Enzyme B	Enzyme C	Enzyme D	Enzyme E
Kappa no.	13.7	12.0	12.7	12.5	11.9	11.9

Table –23 Effect of Enzyme Treatment on Hard Wood Pulp Bleached With CE(P)HH Sequence						
Particulars	Cont rol	Enzyme Treated Pulp				
		Enzyme A	Enzyme B	Enzyme C	Enzyme D	Enzyme E
Chlorination						
Applied Chlorine, %	2.2	2.2	2.2	2.2	2.2	2.2
Consumption %	98.0	92.0	88.5	88.5	90.5	90.1
Alkali extraction stage E						
Applied NaOH, %	2.0	2.0	2.0	2.0	2.0	2.0
Applied Peroxide%	0.5	0.5	0.5	0.5	0.5	0.5
Hypo stage						
App. Hypo, %	1.5	1.5	1.5	1.5	1.5	1.5
Brightness, %	84.2	84.5	84.4	85.6	84.8	83.6
Brightness Improvement %	--	+0.3	+0.2	+1.4	+0.63	-0.6

Pulp treated with 0.1% Texzyme enzyme dose showed 1.4 unit brightness improvement.

Case study – 11

Pulp sample collected – Hard wood from star paper mill, Saharanpur

pH – 8.0

Enzyme A – Texzyme 0.10%

Enzyme B – Texzyme 0.10% (less Cl₂ dose)

Table – 24 Enzyme Pretreatment Conditions			
Particulars	Control	Enzyme A	Enzyme B
Enzyme activity , IU/ml)	--	2000	2000
Enzyme Dose , %	--	0.1	0.15
Treatment time , (min.)	120	120	120
Temperature , °C	50	50	50
Water Extracts			
Colour (Kg/ton)	4.7	6.3	6.3
Lignin (Kg/ton)	0.5	0.8	0.9
Reducing sugars , (Kg/ton)	0	0	0
Unbleached pulp			
Kappa no.	13.9	14.0	14.3
Brightness, % ISO	34.8	35.4	35.3
Brightness Improvement %	--	+0.6	+0.5

Table – 25 Effect of Enzyme Treatment on Hard Wood Pulp Bleached With CE(P)HH Sequence					
Particulars	Control	Enzyme Treated Pulp			
		Enzyme A	Enzyme B	Enzyme C	Enzyme D
Chlorination					
Applied Chlorine %	2.2	2.2	2.2	2.2	2.2
Consumption %	98.0	94.0	9.7	95.2	98.9
Kappa no.	5.2	4.1	3.9	3.1	3.0

Alkali extraction stage E					
Applied NaOH%	2.0	2.0	2.0	2.0	2.0
Alkali Consumption %	63.3	65.4	62.2	62.9	59.7
Applied Peroxide %	0.5	0.5	0.5	0.5	0.5
Brightness %	64.9	64.0	64.9	65.3	64.6
Hypo stage					
App. Hypo %	1.2	1.2	1.2	1.2	1.2
Hypo Consumption %	83.3	85.8	80.8	80.0	89.2
Brightness, %	85.2	85.7	85.3	86.6	86.0
Brightness Improvement %	--	+0.5	+0.1	+1.4	+0.8
Yield , %	93.6	95.1	89.6	91.4	94.3

Selection of Enzyme for mill trial

- Final short listing of the enzyme suppliers was made on the basis of effectivity and cost of the enzyme.
- Based on the performance of the enzyme and the cost of the enzyme for 15 days trial, following enzymes were identified for mill trial.

1. Sebritte BB (Advanced Biochemicals ltd., Mumbai)
2. Pulpzyme HC (Value addition Pvt.Ltd., New Delhi)

Chapter – V

Xylanase mill trial at Seshasayee Paper & Boards Ltd., Erode

1. Mill Trial – 1 with Sebritte BB

After extensive interaction with SPB, mill trial of enzyme bleaching with identified xylanase sebritte BB was planned. Before going for mill scale it was decided to evaluate the response of consignment sample of purchased enzyme on SPB hardwood pulp at R & D lab, SPB, Erode.

The objective of the trial was to identify the potential to reduce the applied charges of chlorine using identified xylanase enzyme on kraft pulp with simultaneous reduction in release of chloro organic compounds (AOX) without adversely effecting the quality of the pulp.

Enzymatic prebleaching experiments with the consignment sample:

Some experiments on xylanase prebleaching on SPB unbleached unscreened pulp using identified xylanase Sebritte BB using process conditions of SPB Ltd. Experiments were conducted with the association of the members of R & D lab, SPB Ltd. Kappa number & brightness were analysed by mill lab.

Exp.: 1

Enzymes : Sebritte BB, Pulpzyme HC

Pulp : Unscreened & Unbleached hardwood pulp from SPB,
(Seshasayee Paper & Boards Ltd., Erode)

Bleaching Sequence: CE_(P) E_(P) H (Mill bleach Sequence)

Table –1 Enzyme treatment conditions			
Particulars	Control	Sebritte BB	Pulpzyme HC
Enzyme Dose, %	-	0.05	0.05
Consistency, %	8.0	8.0	8.0
Temperature, ° C	55	55	55
Treatment time, hr	2.0	2.0	2.0
pH	8.3	8.3	8.3

Table – 2 Results of bleaching of enzyme treated and untreated hardwood pulp										
Particulars	Control		Sebritte BB				Pulpzyme HC			
Bleaching (CE _(P) E _(P) H)										
	Control		Same Cl ₂		Less Cl ₂		Same Cl ₂		Less Cl ₂	
Appl. Cl ₂ , %	3.9		3.9		3.4		3.9		3.4	
Consumption, %	87		96		96		90		96	
Savings, Cl ₂ , %	-		-		13/15		-		13/15	
E (P) –1										
Applied NaOH, %	1.0		1.0		1.0		1.0		1.0	
Applied H ₂ O ₂ , %	1.25		1.25		1.25		1.25		1.25	
Brightness, %	57.6		61.7		61.6		61.8		58.5	
E (P) –2										
Applied NaOH, %	0.5		0.5		0.5		0.5		0.5	
Applied H ₂ O ₂ , %	0.75		0.75		0.75		0.75		0.75	
Brightness, %	65.3		71.7		69.0		70.2		65.7	
Hypo										
Applied Hypo, %	1.5	2.0	1.5	2.0	1.5	2.0	1.5	2.0	1.5	2.0
Applied NaOH, %	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
		5			5		5		5	
Brightness, %	81.0	81.2	83.1	83.0	82.8	82.7	82.8	82.6	80.7	80.9
Imp. in Brightness	-	-	2.1	1.7	1.9	1.5	1.8	1.4	-	-

Table – 3 Results of bleaching of enzyme treated and untreated hardwood pulp			
Particulars	Control	Sebritte BB	Pulpzyme HC
Bleaching (CE_(P) E_(P) H)			
		Less Cl₂	Less Cl₂
Appl. Cl ₂ , %	4.0	3.2	3.2
Cl ₂ Consumption, %	76	77	78
Savings, Cl ₂ , %	-	20	20

E(P) –1						
Applied NaOH, %	1.0	1.0	1.0	1.0	1.0	1.0
Applied H ₂ O ₂ , %	1.25	1.25	1.25	1.25	1.25	1.25
Brightness, %	59	61.0	61.0	61.0	65.0	65.0
E(P) –2						
Applied NaOH, %	0.5	0.5	0.5	0.5	0.5	0.5
Applied H ₂ O ₂ , %	0.75	0.75	0.75	0.75	0.75	0.75
Brightness, %	62.8	68.0	68.0	68.0	72.0	72.0
Hypo						
Applied Hypo, %	1.5	2.0	1.5	2.0	1.5	2.0
Applied NaOH, %	0.25	0.25	0.25	0.25	0.25	0.25
Brightness, %	86.3	86.8	85.8	87.7	87.2	88.1
Imp. in Brightness	-	-	-	1.0	1.0	1.3

Optimisation of enzyme dose

Exp. : 2

Enzymes : Sebritte BB

Pulp : Unscreened & Unbleached hardwood pulp from SPB,
(Seshasayee Paper & Boards Ltd., Erode)

Bleaching Sequence: CE_(P) E_(P) H (Mill bleach Sequence)

Table – 4 Enzyme Treatment Conditions				
Particulars	Control	Sebritte BB (0.025%)	Sebritte BB (0.05%)	Sebritte BB (0.10%)
Enzyme Dose, %	-	0.025	0.05	0.10
Consistency, %	8.0	8.0	8.0	8.0
Temperature, ° C	55	55	55	55
Treatment time, hr	2.0	2.0	2.0	2.0
pH	8.3	8.2	8.3	8.4

Table – 5 Results of bleaching of enzyme treated and untreated hardwood pulp				
Particulars	Control	Sebritte BB (0.025%)	Sebritte BB (0.05%)	Sebritte BB (0.10%)
Unbleached Pulp Characteristics				
Kappa no.	22.2	20.8	21.0	21.3
Brightness	25	26	26	27
Bleaching (CE_(P) E_(P) H)				
Appl. Cl ₂ , %	4.4	4.4	4.4	4.4

Cl ₂ Consumption, %	4.15	4.0	4.16	4.16
E(P) –1				
Applied NaOH, %	1.0	1.0	1.0	1.0
Applied H ₂ O ₂ , %	1.25	1.25	1.25	1.25
Brightness, %	57	61	64	64
E(P) –2				
Applied NaOH, %	0.5	0.5	0.5	0.5
Applied H ₂ O ₂ , %	0.75	0.75	0.75	0.75
Brightness, %	62	64	67	67
Hypo				
Applied Hypo, %	1.75	1.75	1.75	1.75
Applied NaOH, %	0.25	0.25	0.25	0.25
Brightness, %	78	80	81	81
Imp. in Brightness	--	+2.0	+3.0	+3.0

Exp : 3

Enzymes : Sebritte BB

Pulp : Unscreened & Unbleached hardwood pulp from SPB,
(Seshasayee Paper & Boards Ltd., Erode)

Bleaching Sequence: CE_(P) E_(P) H (Mill bleach Sequence)

Table – 6 Enzyme Treatment Conditions			
Particulars	Control	Sebritte BB (0.05%)	Sebritte BB (0.10%)
Enzyme Dose, %	-	0.05	0.10
Consistency, %	8.0	8.0	8.0
Temperature, ° C	55	55	55
Treatment time, hr	2.0	2.0	2.0
pH	8.3	8.3	8.3

Table – 7 Results of bleaching of enzyme treated and untreated hardwood pulp			
Particulars	Control	Sebritte BB (0.05%)	Sebritte BB (0.10%)
Unbleached Pulp Characteristics			
Kappa no.	22.2	21.0	21.3
Brightness	25	26	27
Bleaching (CE_(P) E_(P) H)			
Appl. Cl ₂ , %	4.4	4.4	4.4
Cl ₂ Consumption, %	4.2	4.2	4.12
E(P) –1			
Applied NaOH, %	1.0	1.0	1.0

Applied H ₂ O ₂ , %	1.25	1.25	1.25
Brightness, %	59	62	63
E(P) –2			
Applied NaOH, %	0.5	0.5	0.5
Applied H ₂ O ₂ , %	0.75	0.75	0.75
Brightness, %	63	67	68
Hypo			
Applied Hypo, %	2.0	2.0	2.0
Applied NaOH, %	0.25	0.25	0.25
Brightness, %	79	83	83
Imp. in Brightness	--	+ 4.0	+ 4.0

Exp : 4

Enzymes : Sebritte BB

Pulp : Unscreened & Unbleached hardwood pulp from SPB,
(Seshasayee Paper & Boards Ltd., Erode)

Bleaching Sequence : CE_(P) E_(P) H (Mill bleach Sequence)

Table – 8 Enzyme Treatment Conditions			
Particulars	Control	Sebritte BB (0.05%)	Sebritte BB (0.10%)
Enzyme Dose, %	-	0.05	0.10
Consistency, %	8.0	8.0	8.0
Temperature, ° C	55	55	55
Treatment time, hr	2.0	2.0	2.0
pH	8.3	8.3	8.3

Table – 9 Results of bleaching of enzyme treated and untreated hardwood pulp			
Particulars	Control	Sebritte BB (0.05%)	Sebritte BB (0.10%)
Unbleached Pulp Characteristics			
Kappa no.	22.2	21.0	21.3
Brightness	25	26	27
Bleaching (CE_(P) E_(P) H)			
Appl. Cl ₂ , %	5.0	5.0	5.0
Cl ₂ Consumption, %	4.7	4.8	4.8
E(P) –1			
Applied NaOH, %	1.0	1.0	1.0
Applied H ₂ O ₂ , %	1.25	1.25	1.25

Brightness, %	66	68	69
E(P) –2			
Applied NaOH, %	0.5	0.5	0.5
Applied H ₂ O ₂ , %	0.75	0.75	0.75
Brightness, %	68	69	70
Hypo stage			
Applied Hypo, %	1.5	1.5	1.5
Applied NaOH, %	0.25	0.25	0.25
Brightness, %	82	83	84
Imp. in Brightness	--	+1.0	+2.0

Results of the enzymatic prebleaching experiments conducted with the consignment sample of Sebritte BB are encouraging and the mill management was satisfied with the performance of the enzyme in lab and gave permission for mill trial.

Xylanase Prebleaching Mill trial At Seshasayee Paper Boards, Erode employing Sebritte BB from Advanced Biochemicals Ltd.

Mill trail at SPB was conducted with Sebritte BB with commissioned equipment in June, 2005. After commissioning of additional required equipments like MC Pump,StandPipe,Enzyme dosing pump,a sparger at the pulp conveyer and all required facilities Civil, Mechanical and Electrical connections enzyme trial was started. Five member team of CPPRI was actively engaged at SPB ltd during mill trial. Before starting the enzyme trail general process data was collected and was presented in tables - 11&12.

Enzyme addition:

Xylanase enzyme Sebritte BB is in powder form .Enzyme was dissolved in water in 1M3 enzyme tank and pumped through enzyme dosing pump installed at SPB according to the desired flow rates. Enzyme was added to the pulp through a sparger pipe arranged at screw conveyor. Enzyme added pulp passed through standpipe and vigorous mixing of the enzyme with the pulp has been taken place with MC Pump. After mixing the pulp is subjected to usual screening process of the mill.

Efficiency of MC Pump installed at SPB

In order to ascertain the enzyme dispersion efficiency of MC Pump an experiment was conducted at SPB where in enzyme was dosed through dosing pump and the sample of the pulp was drawn from the pulp mill after the pulp was passed through MC Pump and fed to HD tower where in the retention time of 1.5 hr is given. Similarly in another sample similar dose of enzyme was mixed manually and given a retention time of 1.5 hr.

Table – 10 Enzymatic prebleaching of hardwood pulp drawn from MC Pump			
Particulars	Control – Pulp drawn from pulp mill after MC Pump	Pulp drawn after MC Pump – Enzyme mixed through MC pump	Pulp drawn from pulp mill after MC Pump – Enzyme mixed manually
Unbleached pulp pH – 9.8; Temp , 52 C; Consistency ,% - 6-7			
Enzyme Pretreatment conditions			
Enzyme dose, %	nil	0.075	0.075
Retention time , hr	1.5	1.5	1.5
pH	9.8	9.8	9.8
Temp, C	55	55	55
Consistency, %	6.5	6.5	6.5
Unbleached pulp after enzyme treatment			
Kappa No.	19.0	17.0	17.0
Brightness, %	30	31	30
Chlorination stage			
Cl ₂ applied Dose, %	4.2	3.6	3.6
Consumption, %	98	99	99
Cl₂ Savings , %	-	15	15
Brightness , %	40	41	40
Extraction stage - 1			
NaOH applied Dose, %	1.5	1.5	1.5
H ₂ O ₂ applied, %	0.5	0.5	0.5
Brightness , %	50	47	47
Extraction stage - 2			
NaOH applied Dose, %	1.0	1.0	1.0
H ₂ O ₂ applied, %	0.5	0.5	0.5
Brightness , %	58	58	58
Hypo stage			
Hypo applied Dose, %	2.0	2.0	2.0
NaOH applied, %	0.4	0.4	0.4
Brightness ,% ISO	83	84	83

Observations:

From the above results it is evident that the dispersion efficiency of the enzyme in the pulp when passed through MC Pump is at par with the manual mixing as in both the cases the savings in Cl_2 (around 15%) is similar while maintaining a brightness level of 83.0% . However the samples are preserved for evaluation of desired parameters at CPPRI.

Data of Mill Trial - 1

Table – 11 General data of mill		
Particulars	Without Enzyme 13.6.05-16.6.05	With Enzyme 17.6.05-26.6.05
Pulp Production, ton/Day	142-152	140-152
Enzyme Dose , Kg/Ton	-	0.66
PH of outlet of MC Pump	9.8-10.0	9.4-9.7
Temperature, ° C	57-60	52-58
Consistency %	6-7	6-7
Retention in HD chest , hrs (Enzyme treatment time)	2.0-2.5	2.0-2.5

Table – 12 Process data of mill trial								
Date	Eimco washer			M. C. Pump			Thickner	
	Temp p	pH	Cy	Temp	pH	Cy	Temp p	pH
2.6.05	56	10.0	-	55	9.4	6.0	-	-
3.6.05	52	10.3	-	56	9.3	6.0	-	-
4.6.05	69	10.4	-	56	10.2	8.0	-	-
5.6.05	64	10.0	-	53	9.8	8.0	-	-
6.6.05	65	10.2	-	55	10.0	8.0	-	-
7.6.05	63	10.1	--	54	9.5	7.2	-	-
8.6.05	56	10.2	-	52	9.9	4.4	-	-
9.6.05	60	9.9	-	55	9.7	-	-	-
10.6.05	58-61	9.9-10.3	13.6-14.3	50-52	9.5-9.7	5.8-6.0	39-41	8.0-8.2
11.6.05	70-74	9.5-10.0	13.8-14.2	49-54	9.4-9.5	6.5-7.0	38-40	7.9-8.3
12.6.05	68-70	9.4-9.7	13.6-14.0	57-60	9.4-9.6	7.5-8.0	37-40	7.7-8.1
13.6.05	70-72	9.5-9.9	14.0-14.2	56-60	9.8-10.0	6.4-7.2	37-38	7.2-7.5
14.6.05	68-	10.2-	14.0-	50-55	9.4-10.0	6.6-7.2	41-	9.5-9.7

5	70	10.6	14.4				43	
15.6.05	68-70	10.2-10.6	14.0-14.4	50-55	9.4-10.0	6.6-7.2	40-42	9.5-9.7
16.6.05	68-70	10.2-10.6	14.0-14.4	50-55	9.4-10.0	6.6-7.2	43-45	9.5-9.7
After Enzyme treatment								
17.6.05	57-68	9.8-9.9	10.0-12.5	56-60	9.4-9.6	7.0-8.0	42-44	9.5-9.7
18.6.05	68-70	10.5-10.6	14.2-14.6	48-50	10.0-10.2	6.3-7.0	45-47	9.5-9.8
19.6.05	70-72	10.0-10.5	14.0-15.2	48-50	9.3-9.5	6.6-6.9	44-46	9.2-9.3
20.6.05	65-68	10.1-10.6	13.8-14.4	48-50	9.4-9.6	6.6-6.8	45-48	9.2-9.5
21.6.05	65-70	10.5-10.8	13.8-14.5	50-52	9.6-9.8	6.5-8.0	44-48	9.5-9.7
22.6.05	60-70	10.5-10.9	13.8-14.5	50-51	9.2-9.6	6.3-8.0	45-48	9.0-9.7
23.6.05	60-61	10.2-10.7	14.2-14.7	50-53	9.4-9.7	6.4-7.8	46-48	9.2-9.6
24.6.05	65-70	10.3-10.9	14.0-14.4	49-52	9.0-9.4	7.0-7.6	45-48	8.7-8.9

Table – 13 Data of Unbleached Pulp Characteristics				
Date	Wood Bleached pulp TPD	Final Washer Kappa No.	Thickner Kappa No.	Kappa No. Reduction (unit)
13.6.05	117	22.6-23.4	21.8-22.6	0.8-1.2
14.6.05	142	22.1-23.4	21.6-23.1	0.5-0.3
15.6.05	143	22.8	21.5	1.3
16.6.05	152	22.8-23.3	22.4-22.8	0.4-.05
After enzyme treatment				
17.6.05	143	24.1-25.6	22.0-23.1	2.1-2.5
18.6.05	141	21.6-23.3	20.0-22.5	1.6-0.8
19.6.05	126	22.0-27.6	21.7-24.7	0.3-2.9
20.6.05	151.6	20.9-23.6	20.4-22.0	0.5-1.6
21.6.05	136+5	20.8-24.1	19.0-22.0	1.8-2.1
22.6.05	134	22.8-24.5	21.0-22.5	1.8-2.0
23.6.05	143+8	21.6-23.6	20.5-22.5	1.1
24.6.05	137+8.5	22.5-24.0	20.5-22.1	2.0-1.9
25.6.05	143+7.9	20.8-22.5	20.0-21.8	0.8-0.7
26.6.05	-	20.4-23.7	19.8	-

Table – 14 Data of Enzyme trial – Chlorination stage					
Date	Cl₂ Pressure Kg/cm²	Cl₂ applied Kg/tpd	C stage Kappa No.	Kappa No. Reduction	C Brightness %
13.6.05	2.7	50.0	7.3	--	37-40
14.6.05	2.7	50.0	7.0	--	36-42
15.6.05	2.7	50.0	--	--	36-43
16.6.05	2.7	50.0	--	--	39-42
After enzyme treatment					
17.6.05	2.7	50.0	6.2-6.7	0.8-0.5	40-45
18.6.05	2.7	50.0	6.5-7.0	0.5-0.3	39-44
19.6.05	2.7	50.0	6.8	0.4	39-42
20.6.05	2.7	50.0	6.4	0.8	37-42
21.6.05	2.7	50.0	6.1-6.3	0.9-1.0	39-43
22.6.05	2.2-2.4	48.0	5.6-6.3	1.4-1.0	39-45
23.6.05	2.2-2.4	48.0	5.8-6.6	1.2-0.8	38-43
24.6.05	2.2-2.4	48.0	6.1-6.3	0.9-1.0	37-45
25.6.05	2.2-2.4	48.0	6.2-6.4	0.8-0.9	41-44
26.6.05	2.1-2.3	48.0	5.8	1.4	38-46

Table – 15 Data of Enzyme trial -Alkali Extraction Stage				
Date	CEpIst stage Kappa No.	CEpIst Brightness %	CEpIInd stage Kappa No.	CEpIInd Brightness %
13.6.05	5.0	41-43	3.8	44-54
14.6.05	5.6	41-47	3.6	52-58
15.6.05	5.9	42-48	4.5	54-58
16.6.05	--	40-49	--	47-53
After enzyme treatment				
17.6.05	5.2-5.9	41-48	3.7-4.1	48-57
18.6.05	3.0-4.5	44-47	2.4-3.5	47-55
19.6.05	4.7	40-48	3.6	47-59
20.6.05	5.2	40-46	3.4	46-56
21.6.05	4.0-5.2	43-53	3.0-3.6	55-58
22.6.05	4.0-4.9	40-46	3.0-3.5	53-59
23.6.05	3.9-5.0	42-49	2.6-3.6	50-58
24.6.05	4.2-5.4	40-50	3.0	50-57
25.6.05	4.3-4.4	41-50	3.1-3.2	44-60
26.6.05	4.5	45-51	2.6	53-62

Table – 16 Data of Enzyme trial – Hypo stage			
Date	Hypo flow	Applied hypo%	Brightness %
13.6.05	75+	4.5	80-81
14.6.05	75+	.5	80-81
15.6.05	75+	4.5	80-81
16.6.05	75+	4.5	80-81
After enzyme treatment			
17.6.05	70+	4.3	82
18.6.05	62-65	4.0	80-81
19.6.05	62-66	3.82	80-81
20.6.05	65-75	4.68	81-82
21.6.05	65-70	4.08	80-81
22.6.05	65-68	4.02	80-81
23.6.05	66	4.02	81-82
24.6.05	66	3.8	81-82
25.6.05	66	3.9	81-82
26.6.05	62-66	3.82	81-82

Table –17 Residual chlorine in C & Hypo stage effluents		
Date	Residual Cl₂ in C stage Effluent ppm	Residual Cl₂ in H stage Effluent ppm
14.6.05	71	121
15.6.05	21	23
16.6.05	135	561
After enzyme treatment		
17.6.05	200-383	774-1200
18.6.05	56-234	405-1300
19.6.05	21-300	475-1235
20.6.05	256-480	338-1079
21.6.05	155-410	319-806
22.6.05	142-410	398-980
23.6.05	156-298	270-1036
24.6.05	92-185	213-624
25.6.05	185-454	213-1214
26.6.05	14-284	192-545

Table – 18 Strength Properties of enzyme treated and untreated pulps								
Date	Brightn ess%	P.C. No.	⁰SR	Bulk	Burst Index, kPa. m²/g	Tear Index, mN.m²/g	Tensile Index, N.m/g	Viscosity (cm³/ gm)
13.6. 05	82-83	3-4	43	1.32	4.3	4.5	71	--
14.6. 05	82-83	3-4	42	1.33	4.5	4.4	71	377
15.6. 05	82-83	3-4	42	1.35	4.4	4.9	70	239
16.6. 05	82-83	3-4	42	1.30	4.4	4.9	70	--
After enzyme treatment								
17.6 .05	82-83	2-2.5	42	1.35	4.3	5.0	71	--
18.6 .05	82-83	2-2.5	41	1.34	4.4	4.9	69	--
19.6 .05	82-83	2-2.5	41	1.35	4.5	4.7	68	--
20.6 .05	82-83	2-2.5	40	1.33	4.7	5.1	73	--
21.6 .05	82-83	2-2.5	42	1.35	4.5	5.3	71	--
22.6 .05	82-83	2-2.5	--	--	--	--	--	--
23.6 .05	82-83	2-2.5	--	--	--	--	--	--
24.6 .05	82-83	2-2.5	--	--	--	--	--	401
25.6 .05	82-83	--	--	--	--	--	--	--
26.6 .05	82-83	--	--	--	--	--	--	299

Some glimpses of Xylanase trail at SPB ltd.



1. Final washer pulp at SPB



3. Addition of enzyme in enzyme tank



4. Enzyme addition to pulp at screw conveyor through sparger pipe



5. MC Pump & Standpipe installed at SPB to enhance enzyme mixing



6. Analysis of pulps & Effluents at R & D lab. SPB during enzyme trial



Table – 19 Consolidated Enzymatic Prebleaching results of Sebritte BB- Mill Trial –1		
Particulars	Without Enzyme 13.6.05-16.6.05	With Enzyme 17.6.05-26.6.05
BSW pulp kappa No.	23-25	23-25
Screened Pulp Kappa No. - Thickner	22-24	21-22
Kappa No. Reduction	1.0 –1.5	2.0-3.0
Thickner pulp Brightness % ISO	27-30	29-32
CED Viscosity, cm ³ /g	727-836	779-892
Chlorination Stage		
Appl. Cl ₂ Dose %	5.0	4.8
Appl. Cl ₂ Dose , Kg/Tp	50	48
Cl ₂ Savings, Kg/Tp	-	2.0
Cl ₂ Savings, %	-	4%
Residual Cl ₂ in Filtrate (ppm)	50-135	100-450
Residual Cl ₂ in pulp , ppm	20-36	30-70
Micro Kappa No.	7.0-8.0	6.0-7.0
Brightness , % ISO	36-38	40-43
Brightness Improvement , %	-	3-4
Ep-1st Stage		
Applied NaOH, %	1.5	1.5
Applied H ₂ O ₂ %	0.5	0.5
Micro kappa No.	5.0-5.9	4.0-5.0
Brightness, %	42-47	43-50
Brightness Improvement%	-	2-3
Ep - 2nd Stage		
Applied NaOH, %	0.5	0.5
Applied H ₂ O ₂ , %	0.5	0.5
Micro kappa No.	3.6-3.8	3.0-3.5
Brightness , % ISO	53-54	54-59
Brightness Improvement, % ISO	-	3-4
Hypo Stage		
Hypo Flow , M ³ /Hr	75	62-65
Applied Hypo, kg/ton	45	38-40
Applied Hypo, %	4.5	3.8-4.0
Hypo Savings, %	-	11-15
Residual Cl ₂ in Filtrate (ppm)	460-560	400-1000
Residual Cl ₂ in pulp , ppm	25-85	50-300
Brightness, %	82-83	82-83
PC Number	3.0-4.0	2.0-2.5
CED Viscosity, cm ³ /g	239-377	299-401

Table – 20 Strength Properties of enzyme treated pulps tread with SEBRITTE BB during enzyme trial		
Particulars	Without Enzyme 13.6.05-16.6.05	With Enzyme 17.6.05-26.6.05
Strength properties - Unbleached pulp		
Bulk	1.36-1.40	1.38-1.41
Burst Index, kPa. m ² /g	5.1-5.3	5.1-5.6
Tensile Index, N.m/g	79-81	79-82
Tear Index, mN.m ² /g	6.9-7.1	7.0-7.4
Strength properties – Hypo bleached pulp		
Bulk	1.30-1.35	1.33-1.35
Burst Index, kPa. m ² /g	4.3-4.5	4.3-4.7
Tensile Index, N.m/g	70-71	68-73
Tear Index, mN.m ² /g	4.4-4.9	4.7-5.3

Strength properties evaluated at SPB (Factors converted into indexes at CPPRI).

Observations:

Observations on mill scale trial on enzyme prebleaching of pulp at SPB Ltd, Erode. conducted by Central Pulp and Paper Research Institute, Saharanpur (12/06/05 to 27/06/05)

Outcome of the mill trial on xylanase prebleaching was summarized as record note. (Record note was enclosed in the summary)

- I. Having commissioned the equipments required for enzyme prebleaching of pulp i.e. MC Pump, standpipe, dosing pump etc., the enzyme prebleaching trials were started at SPB employing xylanase enzyme procedure by CPPRI at the mill .
- II. Before starting the trial , the pulping and bleaching conditions prevail in the mill were constantly monitored for a period of 10 days in respect of temperature , pH , kappa number , carry over of the unbleached pulp and bleach chemical requirement as well as the residual chlorine and other parameters in the filtrates and pulp mats at various washers .

- III. Before starting the trial the consignment sample of the enzyme was evaluated under the existing pulping conditions in respect of the activity and to optimize under the existing mill conditions.
- IV. The dosing of enzyme at the above mentioned dose was started on 17/06/05. over a period of 24 hrs the effect of enzyme started reflection in the pulping and bleaching streams . The following observations were made.
- The reduction in kappa of unbleached pulp was noticed which was dropped from an average of 23.0-25.0 to 21.0-22.0
 - The residual chlorine in the filtrate at “C” stage and “H” stage increased from a level of around 50ppm to more than 300 ppm (C stage) and from around 300 ppm to more than 1000ppm (H stage) indicates a scope for chlorine reduction
 - There was a drop in the kappa number at all the stages during bleaching i.e at the C stage it was dropped from 7.0 to 6.0, at EP1 stage from 5.0 to 4.0 and EP2 stage from 4.0 to 3.0 with gain in brightness i.e around 2-3 units in each stage respectively
- V. Having observed the positive indications of the enzyme treatment and the scope to reduce chlorine consumption, the mill management decided to reduce initially the hypo consumption due to certain limitations at C-stage. The hypo flow rate was reduced from 75% - 60-65% ($15\text{M}^3/\text{h}$ to $13\text{M}^3/\text{hr}$) and the hypo consumption was reduced from 45.0kg/tp to 38.0-40.0Kg/tp indicating a saving of 10-12% of chlorine while maintaining the target brightness of 82-83% ISO. Further the post colour number (PC Number) of final bleached pulp after enzyme treatment was improved as the PC number was reduced from an average value of 4.0 to less than 2.5.

Enzymatic Prebleaching trial at SPB, Erode

Economics

Particulars	Without enzyme	With Enzyme	
Enzyme dose , kg/tp	-	0.66*	
Enzyme Cost/tp	-	Rs.122/-	
Electricity consumption in running M.C.Pump (Kw/hr)	--	27Kw/hr	
Electricity consumption cost in running M.C.Pump Rs./ton	--	Rs.9.06/-	
Total Cost of enzyme usage		Rs.131.06	
Bleach chemical reduction – chlorine at diff. stages			
At C- Stage			
Cl2 applied ,kg/tp	50	48 *	
Cl2 savings , kg/tp	-	2	
% reduction	-	4%	
Cost Savings , Rs/tp	-	16/-	
At Hypo stage			
Hypo Applied , kg/tp	45	38-40	As per mill calculation
Hypo savings , kg/tp	-	7 -5	-
% reduction	-	15-11	-
Hypo savings , m ³ /day	-	-	48
Hypo savings , m ³ /ton	-	-	0.32 m ³ per ton
Cost savings , (Cl2+Lime)	-	56/-+32/- = 88/-	(70+40) = 110/-
Electricity consumption savings in hypo preparation (hypo+lime) (Kw/hr)	--	8.67+11.2 =19.9	8.67+11.2=19.9
Electricity consumption cost savings in hypo preparation (hypo+lime) Rs./ton	--	Rs. 6.37/-	Rs. 6.37/-
Total Cost savings (H+C stages) during enzyme usage , Rs./tp	-	110.4/-	(110+16+6.4) =132.4/-
Added Benefits of enzyme usage		1. Reduction in PC number (30-40%) 2. Slight improvement in strength properties 3. AOX reduction (under evaluation) 4. Uniform target brightness in final bleached pulp	

- * Enzyme dose was increased to 0.66 kg per ton from optimum dose 0.5kg/ton because of higher pH of unbleached unscreened pulp at SPB, which resulted in to slightly higher cost of enzyme.
- * Because of process limitations of the mill like static Cl₂ mixer and absence of flowmeters at C-stage, savings in Cl₂ stage were not accurately measured. There is possibility of further reduction of elemental Cl₂ after overcoming the said limitations.

Cost of chemicals:

Enzyme , kg – Rs. 185/-

Lime cost, ton – 3500/-

Chlorine cost, ton - Rs, 8000/-

Hypo cost = (Lime + Chlorine)

2. Mill Trial – 2 with Pulpzyme HC

During the end meet of the enzyme mill trial –1, CPPRI & SPB were proposed next enzyme trial with a alternate enzyme will be planned after installation of the chlorine mixer, flow meter at C stage being initiated by the mill and attending the limitations described in the record note to achieve maximum benefit of chemical reduction with xylanase treatment .

After incorporation of the chlorine mixer and flow meter second mill trial with another identified xylanase Pulpzyme HC from Value addition Pvt. Ltd., New Delhi was planned in November, 2005. Pulpzyme HC was ordered by CPPRI for mill trial.

Xylanase Prebleaching of hard wood pulp of SPB using consignment sample of Pulpzyme HC

Xylanase Prebleaching lab scale experiments with consignment sample were conducted in R & D lab, SPB with the help of the lab staff.

Table – 21 Results of Pulpzyme treated & untreated pulps			
Unbleached Unscreened Pulp – pH 9.8 ; Temp. ⁰ c -65 ; Brightness %- 29			
Enzyme treatment conditions			
Particulars	Control	Enzyme treatment pulp	
	1	2	
Enzyme dose , %	-	0.05	
Temperature , ⁰ c	50-60	50-60	
pH	9.4	9.2	
RT , hrs	2	2	
Consistency	8.0	8.0	
After enzyme treatment – pulp			
Kappa no.	18.5	18.0	
Brightness , %	31	31	
After enzyme treatment – pulp extract			
CE(p) E(p) H Bleaching			
Chlorination stage			
	1	2a	2b
Appl. Cl ₂ Dose,%	4.1	4.1	3.5
Consumption , %	99.0	99.0	99.0
Cl ₂ savings , %	-	-	15

Permanganate Number	8.0	8.0		8.0	
Brightness , %	43	45		42	
E(p) 1 st stage					
Applied NaOH , %	1.0	1.0		1.0	
Applied H ₂ O ₂ , %	0.75	0.75		0.75	
Permanganate Number	5.5	5.7		5.5	
Brightness , %	62	66		63	
E(p) 2 nd stage					
Applied NaOH , %	1.0	1.0		1.0	
Applied H ₂ O ₂ , %	0.5	0.5		0.5	
Permanganate Number	3.0	3.2		3.2	
Brightness , %	68	70		69	
Hypo stage					
Applied hypo,%	2.0	2.0	1.7	2.0	1.7
Consumption ,%	76	77	75	75	74
Applied NaOH,%	0.25	0.25	0.25	0.25	0.25
Hypo savings, %	--	--	15	--	15
Brightness , %	82	83	83	81	82

Table –22 Results of Pulpzyme treated & untreated pulps			
Unbleached Unscreened Pulp – pH ; Temp. ⁰ c -65 ; Brightness %			
Enzyme treatment conditions			
Particulars	Control	Enzyme treatment pulp	
	1	2	
Enzyme dose , %	-	0.065	
Temperature , ⁰ c	50-60	50-60	
pH	9.1	9.2	
Retention Time , min	90	90	
Consistency ,%	8.0	8.0	
After enzyme treatment – pulp			
Kappa no.	16.1	14.3	
Brightness , %	33	34	
After enzyme treatment – pulp extract			
CE(p) E(p) H			
Chlorination stage			
	1	2a	2b
Appl. Cl ₂ Dose,%	4.0	4.0	3.4
Consumption , %	92	92	97
Cl ₂ savings , %	-	-	15
Permanganate Number	8.3	7.9	7.9

Brightness , %	45	46	45		
E(p) 1 st stage					
Applied NaOH , %	1.0	1.0	1.0		
Applied H ₂ O ₂ , %	0.75	0.75	0.75		
Residual H ₂ O ₂ ,%	12	12	23		
P.No.	5.3	4.9	4.8		
Brightness , %	66	67	67		
E(p) 2 nd stage					
Applied NaOH , %	1.0	1.0	1.0		
Applied H ₂ O ₂ , %	0.5	0.5	0.5		
P.No.	3.5	3.2	3.2		
Brightness , %	70	73	73		
Hypo stage					
Applied hypo,%	2.0	2.0	1.7	2.0	1.7
Consumption ,%	71	70	69	71	70
Applied NaOH,%	0.25	0.25	0.25	0.25	0.25
Hypo savings, %	--	--	15	--	15
Brightness , %	82	83	82	83	82

Table – 23 Results of Pulpzyme treated & untreated pulps				
Unbleached Unscreened Pulp – pH - 9.2 ; Temp.⁰c -65 ; Brightness %				
Enzyme treatment conditions				
Particulars	Control	Enzyme treatment pulp		
	1	2	3	4
Enzyme dose%	--	0.05	0.065	0.075
Temperature ⁰ c	50-60	50-60	50-60	50-60
pH	8.9	8.9	8.9	8.9
RT , min	90	90	90	90
Consistency	8.0	8.0	8.0	8.0
After enzyme treatment – pulp				
Kappa no.	17.0	16.4	15.7	15.9
After enzyme treatment – pulp extract				
CE(p) E(p) H				
Chlorination stage				
Appl. Cl ₂ Dose, %	4.0	3.4	3.4	3.4
Consumption, %	97	99	99	99
Cl ₂ savings , %	-	15	15	15
Permanganate Number	8.5	8.3	8.2	8.2
Brightness , %	68	68	68	69
E(p) 1st stage				
App. NaOH %	1.0	1.0	1.0	1.0
App. H ₂ O ₂ , %	0.75	0.75	0.75	0.75
Residual H ₂ O ₂ , %	15	16	18	16

Permanganate Number	5.8	5.4	5.2	5.3
Brightness , %	68	68	68	69
E(p) 2nd stage				
App. NaOH , %	1.0	1.0	1.0	1.0
App. H ₂ O ₂ , %	0.5	0.5	0.5	0.5
Residual H ₂ O ₂ , %	--	--	--	--
Permanganate Number	4.5	4.3	4.9	4.3
Brightness , %	71	74	71	75
Hypo stage				
App. Hypo, %	2.0	1.7	1.7	1.7
Consumption, %	94	95	94	94
App. NaOH, %	0.25	0.25	0.25	0.25
Hypo savings, %	--	15	15	15
Brightness , %	82	82	83	83

Results with consignment sample of Pulpzyme HC are encouraging at different doses of enzyme and the enzyme was used for second mill trial.

**Xylanase Prebleaching Mill trial At Seshasayee Paper Boards,
Erode employing Pulpzyme HC from Value addition ltd.**

Table – 24 General data of mill		
Particulars	Without Enzyme 14.11.05-18.11.05	With Enzyme 19.11.05-24.11.05
Pulp Production, ton/Day	115.5 -142.3	111.6-151.3
Enzyme Dose , Kg/Ton	-	0.7-0.75
PH of outlet of MC Pump	9.4 -10.2	9.3-9.4
Temperature, ° C	48-60	48-52
Consistency %	6.4-8.0	6.3-8.0
Retention in HD chest , hrs (Enzyme treatment time)	2.0-2.5	2.0-2.5

Table – 25 Characteristics of unbleached pulp during trial					
Date	Wood Unbleached pulp TPD	Wood Bleached pulp TPD	Final Washer Kappa No.	Thickner Kappa No.	Kappa No. Reduction (unit)
14.11.05	136.8	120.6	24-25	23-24	1.0
15.11.05	141.3	127.2	26-27.5	25-26	1.0

16.11.05	142.3	128.0	23	23	--
17.11.05	137.8	124	22.6	20.6	2.0
18.11.05	115.5	--	23.7	22.7	1.0
After enzyme treatment					
19.11.05	138.9	123.9	24.3	22.3	2.0
20.11.05	151.3	136.2	23.0	20.0	3.0
21.11.05	147.6	132.5	24.0	22.0	2.0
22.11.05	122.7	110.4	24.0	22.0	2.0
23.11.05	111.6	--	23.5	22.5	1.0

Table – 26 Characteristics of chlorination stage pulps during trial					
Date	Cl₂ Pressure Kg/cm²	Cl₂ applied Kg/tpd(DC S)	Cl₂ applied Kg/tpd (Digital)	C stage Kappa No.	C Brightness %
14.11.05	2.9-3.5	47.8	54	6.5-7.5	42-44
15.11.05	2.8-3.5	41.2	47.1	6.0-7.0	39-43
16.11.05	3.0-3.2	40.0	45.9	5.5-6.0	42-45
17.11.05	3.3-3.7	42.9	49.1	5.6-7.0	35-40
18.11.05	2.7-3.3	48.6	55.9	5.6-5.8	38-42
After enzyme treatment					
19.11.05	3.1-3.5	44.3	50.3	5.5-5.8	42-44
20.11.05	3.1-3.3	38.0	43.6	5.0-5.5	44-46
21.11.05	3.0-3.2	37.5	43.2	5.2-5.5	43-45
22.11.05	2.8-3.0	45.3	51.9	5.1-5.5	44-47
23.11.05	-	-	-	5.8-6.0	44-48
24.11.05	--	-	-	5.5-5.8	-

- After enzyme treatment kappa number of C stage pulp is lowered and also improvement in C brightness was observed.

Table – 27 Characteristics of alkali extraction stage pulps during trial				
Date	CEpIst stage Kappa No.	CEpIst Brightness %	CEpIIInd stage Kappa No.	CEpIIInd Brightness %
14.11.05	4.7-6.1	44-50	3.5-6.9	45-60
15.11.05	4.5-6.5	41-46	4.1-5.8	42-55
16.11.05	4.4-5.5	43-49	3.0-5.1	47-58
17.11.05	5.2-5.5	42-45	3.1-4.9	46-56

18.11.05	4.4-4.9	42-49	2.9-4.2	53-57
After enzyme treatment				
19.11.05	3.4-5.4	46-50	2.7-4.1	52-57
20.11.05	4.4-5.7	44-48	3.7-5.3	48-53
21.11.05	4.0-6.0	44-46	3.4-5.4	44-58
22.11.05	4.8-6.1	43-49	3.5-5.7	51-57

- Enzyme treated pulps after extraction stage showed improvement in brightness.

Table – 28 Characteristics of Hypo stage pulps during trial			
Date	Hypo flow	Applied hypo, %	Brightness, %
14.11.05	83	5.74	82-83
15.11.05	84	5.65	82-83
16.11.05	84	5.53	82-83
17.11.05	84	5.77	82-83
18.11.05	81.8	6.7	82-83
After enzyme treatment			
19.11.05	77.9	5.3	82-83
20.11.05	78.3	4.8	82-83
21.11.05	76.8	4.90	82-83
22.11.05	73.4	5.76	82-83

- Maintaining the final brightness of the pulp 82-83, hypo amount was reduced in case of enzyme treated pulps when compared to pulp with out enzyme.

Table – 29 Residuals chlorine in C Stage & Hypo stage effluents during enzyme trial				
Date	Residual Cl₂ in C stage Effluent ppm		Residual Cl₂ in H stage Effluent ppm	
	Mat	Filtrate	Mat	Filtrate
14.11.05	7-227	14-106	7-43	7-28
15.11.05	57-440	170-658	7-71	7-43
16.11.05	43-185	170-665	7-28	7-71
17.11.05	57-355	192-220	7-14	7-21

18.11.05	71-341	185-554	28-57	36-57
After enzyme treatment				
19.11.05	56-355	163-788	7-57	14-142
20.11.05	42-365	142-1129	14-51	7-43
21.11.05	14-340	49-568	7-28	14-63
22.11.05	14-219	142-511	7-28	14-35

- As per the ISO norms of the mill, residual chlorine and hypo in respective effluents were maintained .

Table – 30 Strength Properties of pulps – Xylanase enzyme trial			
Details	Burst Index, kPa. m²/g	Tensile Index, N.m/g	Tear index (mN. m²/g)
Bleached pulps			
With out enzyme			
15.11.05	4.70	69.60	5.10
16.11.05	4.50	70.60	5.00
17.11.05	4.40	70.45	4.70
18.11.05	4.60	74.50	4.90
Enzyme			
20.11.05	4.70	75.50	4.80
Un bleached pulps			
With out enzyme			
15.11.05	5.8	87.3	6.8
16.11.05	5.7	85.3	6.7
17.11.05	5.6	93.1	6.6
18.11.05	5.8	95.1	6.8
Enzyme			
20.11.05	5.6	89.2	7.1

Table – 31 Characterization of effluent during enzyme trial				
Date	COD , mg/l	BOD , mg/l	AOX , Mg/l/kg/ton	AOX Reduction , %
Control				
14.11.05	784	-	-	-
15.11.05	864	-	75.0/5.25	-
16.11.05	624	246	-	-
17.11.05	688	238	-	-
18.11.05	656	248	-	-
Enzyme				
21.11.05	699	248	57.3/4.01	23.6
24.11.05		-	60.0/4.2	20

- Analysis of effluents showed no change in BOD & COD, but 20-24% of AOX reduction was observed during xylanase treatment.

CHEMICAL LOADS BEFORE & AFTER ENZYME TRIAL - NOV. 2005

Date	Pulp Production tpd		Chlorine (Gas)									Hypo						Savings, %			
	Unbl.	Ble.	Flow						Cl2 Cylinder (Gas)			Flow			Cyl.			Cl2 Gas		Hypo	
			DCS			Digital															
			Kg/D	Kg /tp	%	Kg/D	Kg /tp	%	No./Kg/ day	Kg/ tp	%	Ave.F low, %	Kg/ tp	%	No./Kg/ day	Kg/ tp	%	Flow (Dig)	Cyl .	Flo w	Cyl .
Before Enzyme Treatment																					
14/11/05	136.8	120.6	6542	47.8	4.78	7382	54	5.4	7/6300	46.1	4.61	83	57.4	5.74	10/9000	65.8	6.58				
15/11/05	141.3	127.2	5815	41.2	4.12	6655	47.1	4.71	8/7200	51.0	5.10	84	56.5	5.65	10/9000	63.7	6.37				
16/11/05	142.3	128	5691	40.0	4.0	6531	45.9	4.59	8/7200	50.6	5.60	84	55.3	5.53	10/900	63.2	6.32				
17/11/05	137.8	124	5923	42.9	4.29	6763	49.1	4.91	8/7200	52.2	5.22	84	57.7	5.77	10/9000	65.3	6.53				
18/11/05	115.5		5619	48.6	4.86	6459	55.9	5.59	7/6300	54.5	5.45	81.8	67	6.7	10/9000	77.9	7.79				
					4.41			5.40			5.09			5.88			6.72				
Enzyme Trial Started on 19/11/05 at 9.00 AM																					
19/11/05	138.9	123.9	6151	44.3	4.43	6991	50.3	5.03	8/7200	58.1	5.81	77.9	53.0	5.3	9/8100	58.3	5.83				
After Enzyme Treatment																					
20/11/05	151.3	136.2	5755	38.0	3.8	6595	43.6	4.36	7/6300	46.34	4.45	78.3	48.0	4.8	9/8100	53.5	5.35	8.3	6.7	11.9	11.3
21/11/05	147.6	132.5	5541	37.5	3.78	6381	43.2	4.34	7/6300	42.7	4.45	76.8	49.9	4.99	9/8100	54.9	5.42				
22/11/05	122.7	110.4	5790	45.3	4.53	6630	51.9	5.19	8/7200	58.7	5.64	73.4	57.6	5.76	10/9000	70.5	7.05				
Total					4.03			4.62			4.85			5.18			5.96				
23/11/05					3.98			4.67			4.84			-			-				

- Doses Calculation on the basis of unbleached pulps

Observations:

Observations on the second mill scale trial on enzyme prebleaching of pulp at Seshasayee Paper and Boards Ltd, Erode. (Record note was enclosed in the summary.)

1. The required infrastructure i.e MC Pump, stand pipe, dosing pump were installed in the wood street at Seshasayee Paper and Boards Ltd., and mill scale trial with an identified xylanase enzyme (Sebritte BB of M/s Advanced Biochem Ltd., Mumbai) was conducted over a period of two weeks during the month of June 2005 which was successfully completed with total chlorine savings potential of around 15% during bleaching along with AOX reduction of around 25%.
2. Subsequently, it was decided to carry out one more trial with alternate enzyme identified by CPPRI after overcoming certain limitations encountered during enzyme trial particularly in the C-stage of bleaching. These were overcome with the support of mill management by the installation of the chlorine mixer, flow meter for measuring the quantity of chlorine at C stage etc.
3. As per the request from the mill, the enzyme trial with alternate enzyme was started on 13.11.05. Before starting the trial, the pulping and bleaching conditions prevalent in the mill were constantly monitored for a period of one week in respect of temperature, pH, Kappa number, chemical with carry over the unbleached pulp, and bleaching chemical requirement as well as the residual chlorine and other parameters in the filtrates and pulp mats at various washers during pulp processing.
4. Prior to the trial, sample drawn from the consignment of the enzyme was evaluated under the existing pulping conditions in respect of activity and enzyme doses. A dose of 700-750 gm of enzyme per ton of pulp was found to be optimum under the existing mill conditions.

5. The dosing of enzyme at the above mentioned dose was started on 19/11/2005. The effect of enzyme start was reflected in the bleaching streams after a period of 6 hrs. The following observations were made.
- The reduction in kappa of unbleached pulp was noticed which was dropped from an average of 23.0-24.0 to 22.0-23.0
 - The residual chlorine in the filtrate at “C” stage and “H” stage increased from a level of 50ppm to a level of 150 ppm (C stage) and from around 200-300 ppm to 400-500 ppm (H stage) respectively.
 - There was a drop in the kappa number at all the stages during bleaching i.e at the C stage it was dropped from 6.0-7.0 to 5.0- 6.0, at EP1 stage from 5.0-6.0 to 4.0-5.0 and EP2 stage from 4.0 to 3.0 with gain in brightness i.e around 2 units at all three stages i.e C stage, EP1 and EP2.
6. Having observed the positive indications of the enzyme treatment and the scope to reduce chlorine consumption, the chlorine was reduced at C stage as well as Hypo stage. The hypo flow rate was reduced from an average flow rate of 84% to 77% (i.e 16.8 m³/hr to 15.4 m³/hr indicating reduction in hypo consumption from 58.8kg to 51.8 kg/t pulp. Similarly at the C stage, the chlorine charge was reduced from an average of 50.4 kg per ton of pulp to 46.2 kg per ton of pulp indicating the total savings in chlorine at both stage of bleaching i.e at “C” stage and “H” stage of around 15%.
7. Thus the results of the above said plant trial at SPB , indicated savings of around 15% of total chlorine with a potential to improve further after improved washing at BSW since the Soda / COD carryover during the plant trial was higher due to disturbance in the plant because of process disruption caused by heavy rains, and the same led to a higher pH (9.2 – 9.5) as against the required pH of less than 9.0+. Thus the enzyme trial was discontinued w.e.f 24.11.05 as decided by the mill management.

8. Further evaluation of the strength and optical properties of the unbleached and bleached pulp showed a drop in post colour number of the final bleached pulp after enzyme treatment indicating significant improvement (around 40%) which reduced from 1.5-2.2 to 0.7-1.2 without change in the strength properties.
9. From the results of the analysis of the effluent it has been observed that there was a reduction in AOX which dropped from 75 ppm to 57 ppm (24% reduction).

3. Economics of The Enzyme Prebleaching Technology *

(* based on trial data)

CPPRI has successfully demonstrated the xylanase prebleaching technology in a integrated hardwood based mill. Enzyme treatment has been done at pH 9-10 and temperature 50-60 °. The major benefits of this technology are two unit reduction in unbleached kappa number, 15% chlorine reduction and 20-25% AOX reduction in bleach effluents and 30-40% PC number reduction. The results in mill trial showed that the technology is viable in Indian paper Industry also. To assess the other important issue of the technology detailed economic calculations have been made with the help of mill process experts. The same are detailed below.

A. Cost Input (Expenditure)

1. Power cost for MC Pump

2. Enzyme cost

1. Power cost for MC Pump

MC Pump

Capacity – 100 HP –72-75 amperes

Power consumption – 45KWH per hour

Power cost = $45 \times 24 \times 4 = 4,320/-$ (@ I unit – Rs. 4.00/- grid Power)

Power cost per day = Rs.4, 320/-

2. Enzyme cost

Enzyme dose -- 650 g per ton of pulp

Enzyme cost -- per ton of pulp = Rs.195/- (enzyme cost kg - @ Rs.300/-)

Enzyme cost -- per ton of pulp = Rs.163/- (enzyme cost kg - @ Rs.250/-)

Enzyme cost -- per ton of pulp = Rs.130/- (enzyme cost kg - @ Rs.210/-)

Cost Per day -- per 150 tons = Rs.29, 250/- (Case 1)

Cost Per day -- per 150 tons = Rs.24, 450/- (Case 2)

Cost Per day -- per 150 tons = Rs.19, 500/- (Case 3)

Expenditure per day for enzyme technology for 150tpd mill

Power cost + Enzyme cost = Rs.4, 320/- + 29, 250/- = Rs.33, 570/- (Case 1)

Power cost + Enzyme cost = Rs.4, 320/- + 24, 450/- = Rs.28, 770/- (Case 2)

Power cost + Enzyme cost = Rs.4, 320/- + 19, 500/- = Rs.23, 820/- (Case 3)

B. Savings

1. Bleach Chemical Savings
2. Cost for Reduction of AOX 20-24% in effluents

1. Bleach Chemical Savings

a. Hypo

Hypo cost – (Commercial – strength 40gpl – Rs.1000/-)

Cost per M³ - Rs.500/-

(Includes all operational cost - lime+ Cl₂ +Power + Water)

During enzyme trial hypo savings – 2 M³ per hour

-- 48 M³ per day

Hypo savings per day – 48 X 500/- = Rs. 24,000/-

b. Elemental chlorine

Cl₂ Savings – 0.7 ton per day

(0.9 ton Cl₂ gas cylinder costs Rs. 8000/-)

Savings – Rs. 6222/-

Total bleach chemical savings = Gas + Hypo = Rs.6,222 + 24,000 = Rs.30,222/-

2. **Cost for Reduction of AOX 20-24% in effluents – Environmental economics of 24% AOX reduction is not considered for the present cost calculations.**

Cost of enzyme technology , per day = Savings – expenditure

Case 1 - Rs. 30,222/- - 33,570/- = - Rs.3,348/- (Not Viable)

Case 2 - Rs. 30,222/- - 28,770/- = + Rs.1,452/- (Viable)

(Annual Profit for mill =Rs.5,22,720/-)

Case 3 - Rs. 30,222/- - 19,500/- = + Rs.10,722/- (Viable)

(Annual Profit for mill =Rs.38,59,920/-)

Chapter – VI

Conclusion and Summary of Recommendations

Conclusion

Demonstration of the enzymatic prebleaching process in an integrated paper mill, SPB Ltd. has been proven to be successful.

- From the mill scale trials on enzymatic prebleaching of kraft pulp, conducted at SPB using the two identified xylanase enzymes, (the first enzyme, Sebritte BB from M/s Advanced Biochemicals Ltd., Mumbai, trials conducted during the month of June 2006 and the second enzyme, i.e Pulpzyme HC of Novozymes, supplied by m/s Value addition Papers Ltd., New Delhi, mill trials conducted during November, 2005) it has been concluded that installation of required equipments , i.e MC Pump, stand Pipe and enzyme dosing pump etc. by CPPRI at the pulp mill and proper enzyme dosing has helped in proper mixing of the enzyme with the pulp. This has resulted in achieving the targeted outputs in terms of savings of chlorine to the tune of more than 15% in both stages as well as reduction in AOX level of around 22-25%.
- Further, desired parameters like pH, Temperature & Consistency of the pulp etc. could also be achieved with the help of available perwater streams like paper machine back water etc.

Recommendations

1. Enzymatic prebleaching of chemical pulps in Indian Paper Industry using xylanase enzymes could prove to be an effective technology in reducing the chlorine demand by 15% while maintaining the targeted brightness level of +82% ISO.
2. Before introducing enzyme prebleaching technology in any mill it is important to evaluate a particular enzyme preparation for its response towards pulp for various parameters like temp., pH, enzyme activity and cellulose contamination.
3. Installation of enzyme mixing device for proper mixing of enzyme with the pulp will be highly effective and also helps in achieving the desired effect.
4. Good washing Practice of the mill will increase the efficiency of the enzyme and also lower the enzyme dose there by reducing the cost of the enzyme technology.
5. Use of xylanase in Paper industry will boost the enzyme production which may result in lowering of xylanase cost and enzyme may be available in market at lower price.
6. Incorporation of enzyme prebleaching technology in ClO_2 using mills is highly cost effective which decreases the amount of ClO_2 and generation cost of ClO_2 .
7. The technology needs to be boosted in Indian Paper Industry in order to explore the possibilities of achieving the desired standard norms in respect of the AOX levels.

Chapter – VII

Publications:

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3. R.K. Jain, R.M. Mathur, Vasanta V Thakur & A.G.Kulkarni “ **Mill Experiences With Enzyme Prebleaching - Opportunities In Indian Paper Industry** ” Proceedings of PAPEREX – 2003 held from 5 – 8th Dec at New Delhi.
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5. Vasanta V Thakur, R.M.Mathur , & A.G.Kulkarni, “**Enzyme processes for Pulp and Paper Industry**”, **BIOCONVERGENCE, 2004**, 18-20 November, 2004 at Patiyala, Punjab.
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7. R.K.Jain, R.M.Mathur, Vasanta.V.Thakur, Piyush Verma and A.G.Kulkarni
“ Implementation of Xylanase Prebleaching in Indian Paper Industry”,
IPPTA Convention Issue, 2006.