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Fiber Modification with Enzymes

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&

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Particulars of the Project

1.	Project No.	:	TCIRD/IPMA/0709-24/RCDP-100
2.	Project Title	:	Fiber Modification with Enzymes
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4.	Sponsoring Agency	:	Indian Paper Manufacturers Association
5.	Implementing Agency		Thapar Centre for Industrial Research and Development Central Pulp and Paper Research Institute
6.	Participating Agencies	:	Ballarpur Industries Ltd.
7.	Date of Start	:	Sept. 2010
8.	Duration of the Project	:	24 months

Contents

Nomenclature

CMCase	:	Carboxy Methyl Cellulase Activity
FPA	:	Filter Paper Activity
°SR	:	Schopper-Riegler
CSF	:	Canadian Standard Freeness
FPR	:	First Pass Retention
FPAR	:	First Pass Ash Retention
MD	:	Machine Direction
CD	:	Cross Direction
MOW	:	Mixed Office Waste
NDLKC	:	New Double Lined Kraft Cuttings
OCC	:	Old Corrugated Cartons
COD	:	Chemical Oxygen Demand
SEM	:	Scanning Electron Microscope
XRD	:	X Ray Diffraction

1. EXECUTIVE SUMMARY

Under this project the lab scale studies on mixed hardwood & mixed hardwood bamboo pulps were conducted at Thapar Centre for industrial Research and Development (TCIRD), Yamuna Nagar. Studies on recycled fibers were conducted at Central Pulp & Paper Research Institute (CPPRI), Saharanpur. Plant scale studies using screened enzyme based on the lab scale studies conducted at TCIRD were conducted at one of the unit of Ballarpur Industries Limited.

Enzyme-4, Enzyme-3 and Enzyme-2 were found to be more effective in reducing refining energy requirements of the wood based pulps targeting the same strength properties as that of control. Enzyme-3 and Enzyme-4 were capable to reduce refining energy by 29.6% whereas Enzyme-2 was able to reduce refining energy by 26.9% in the laboratory scale experiments carried out at TCIRD. Enzyme-3 was found most effective to improve drainage, as it improved the drainage of the pulp up to 17.1%.

Results of enzyme treatment of recycled fibers conducted at CPPRI showed improvement in strength properties along with marginal improvement in drainability of MOW pulp with enzyme 8 whereas improvement in drainability of pulp along with marginal improvement in strength properties with enzyme 9. Results of enzyme treatment of NDLKC and OCC varieties are promising and their use offers significant advantage over conventional chemical process viz. savings in energy during refining by 10-40%, reduction in pollution loads of colour by 80-90%, COD by 50-60% and iimprovement in yield by 2- 4%.

Plant scale trial conducted with screened enzymes on wood based paper mill showed reduction in refining energy by 20%, improvement in FPR and FPAR, improvement in freeness of the Head box and Machine chest pulps with reduced refining energy and improvement in breaking length.

2. BACKGROUND

Several enzymes having distinct nature are available in the market for fiber modification. The enzymes used in this application are from the cellulase and hemicellulase family. Enzymatic pretreatment of chemical pulps before refining helps in fibrillation; this can reduce the energy requirement in mechanical refiner. Therefore, the cutting of fibres or fines generation will reduce during refining. Due to lesser fines in the refined stock, the drainage rate at the wire will increase, requiring less or no drainage aid chemicals; machine speed can also be increased to produce more paper in the same machine. Due to better drainablity, the moisture content of the web before going to dryer section is expected to be lower, requiring less steam for sheet drying. The desired mechanical strength properties of paper can be obtained even with lesser degree of refining (at higher CSF), improving the bulk of the paper. The filler loading can be increased to some extent without sacrificing on the bulk.

Most of these enzymes are very sensitive towards raw material used and process parameters like pH, time and temperature. Indian paper mills do not have much exposure to the use of enzymes in their processes. Very little work has been carried out with indigenous raw material and literature on this subject is scanty.

On the above background the CESS Committee has sponsored this project to IPMA. Under this project the lab scale studies on mixed hardwood & mixed hardwood bamboo pulps were conducted at Thapar Centre for industrial Research and Development (TCIRD), Yamunanagar. Studies on recycled fibers were conducted at Central Pulp & paper Research Institute (CPPRI), Saharanpur. Plant scale studies using screened enzyme based on the lab scale studies conducted at TCIRD were conducted at one of the unit of Ballarpur Industries Limited.

3. OBJECTIVES

- To explore possibility of energy saving during refining of the pulp
- To study the effect of enzymatic refining on other pulp properties
- To overcome the bottleneck of refining capacity
- Demonstration of results with enzymes in the plant scale

4. EXPERIMENTAL

4a. Wood/ bamboo pulps *(Experiments carried out at TCIRD)*

i. Collection of enzyme samples

Different commercial enzymes were collected from international as well as national enzyme manufactures. In the present study four enzymes were collected from M/s Novozymes, Denmark, two enzymes from Dyadic International, U.S.A. and one from Tex Biosciences, Chennai. The enzyme samples received from the different manufacturers were randomly marked as Enzyme-1, Enzyme-2, Enzyme-3, Enzyme-4, Enzyme-5, Enzyme-6 and Enzyme-7.

ii. Collection of pulp samples

Bleached unrefined pulp was collected from bleached decker of an integrated pulp and paper mill in north India. The raw materials furnish used to produce the pulp in the mill was mixed hard wood along with bamboo. The bleaching sequence followed at the mill was $C_DE_{OP}D_1D_2$. In the later part of the report the mill was written as Mill-1.

For the study bleached unrefined pulp was also collected from HD tower of another integrated pulp and paper mill situated in southern part of the India. The raw material furnish used in the mill was mixed hard wood consisting of Eucalyptus Casuarinas and Subabul and the bleaching sequence followed was $OD_0E_{OP}D$. In the later part of the report the mill was written as Mill-2.

iii. Enzymatic treatment of pulp for reduction in refining energy

The bleached pulps collected from the mills were treated with different enzymes at $40^{O}C$ temperature for 60 minutes at pH 7.0 and 4% consistency in plastic beakers with mild stirring. The reference pulps were incubated at the same conditions as the enzyme treated pulps prior to refining. Refining of the pulps was carried out in the PFI mill at different CSF levels. Hand sheets of refined and unrefined pulps were prepared and tested for different physical strength and surface properties.

iv. Enzymatic treatment of pulp for drainage improvement

Enzymatic treatment of the refined pulp was done at different dosages after refining the pulp with and without using enzyme. During treatment similar conditions were maintained for all the pulps i.e. temperature- 40 ^{0}C, consistency-5% and pH 7.0±0.5 for 30 minutes.

4b. Recycled fiber pulps *(Experiments carried out at CPPRI)*

The following unbleached and bleached grades of recovered paper collected from paper mills were used in the study.

1. Mix office waste

2. New double lined Kraft cuttings (NDLKC)

3. Old corrugated cartoons (OCC)

Commercial enzymes used were procured from international and national reputed companies and are detailed in table 1.

Photomicrography of the untreated, chemical treated and enzyme treated pulps after refining was done with image analysis to see the effect of enzymes on fiber structure, swelling and fibrillation.

4c. Analytical techniques

Cellulase activity in enzymes: was determined by Ghose Method (Pure & Applied Chem., Vol.59, No.2, pp.257-268, 1987)

Drainability was determined by using DFR 04 (Dynamic drainage, freeness and retention tester).

Drainage of the pulp at TCIRD was determined as per method of Litchfield, E (1994). APPITA J 47: 62-65.

Fines analysis was performed with a 200 mesh Screen, according to TAPPI test method T261cm-90.

Fines content was processed through Britt's dynamic jar using -200 screen.

Freeness of pulp: was determined as per Tappi Test Method T 227 om-99 at TCIRD and as per ISO 5267-2 method at CPPRI.

Hand sheets of the pulp: were made according to Tappi Test Method T 205 sp-02 at TCIRD and as per ISO method 5364 at CPPRI.

Laboratory refining of pulp: was done as per Tappi Test Method T 248 sp-00.

Moisture content of the pulp: was determined as per Tappi Test Method T 210 cm-03.

Physical strength properties: were determined as per Tappi Test Method T 220 sp-01.

Reducing sugar content in effluent was measured by DNS as per the procedure followed by Miller as detailed in Miller G. L., "Use of Dinitrosalicylic acid (DNS) for determination of reducing sugars", Analytical Chemistry, 31, 1959.

The effluents obtained from enzymatic and chemical pulping processes were analyzed in terms of chemical oxygen demand (COD), colour and lignin accordingly to APHA (American Public Health Association) Standard Methods.

Xylanase activity in enzymes: were determined as per the method of Bailey et al., Journal of Biotechnology, 23 pp 257-270 (1992)

Enzymatic refining of wood/Bamboo pulp

5. RESULTS AND DISCUSSION - Enzymatic refining of wood/ bamboo pulp

5a. Activities of enzyme samples

Different activities of the enzyme samples were evaluated by the respective methods given in the experimental section of report. CMCase activity was evaluated at pH 6.0, 7.0 and 8.0. All the enzymes showed highest activity at pH 7.0. Enzyme 3 has the highest CMCase activity as compared to other enzymes. CMC activity of different enzymes at 50^o C and at various pH levels are given in Table 1.

Xylanase activity was evaluated at pH 6.0, 7.0 and 8.0. Enzyme-6 and Enzyme-7 were the Xylanase enzyme. Different enzymes showed highest activity at different pH. Enzyme 7 has the highest Xylanase activity as compared to other enzymes. Xylanase activity of different enzymes at 50^o C and at various pH levels are given in Table 2.

FPase (Filter Paper) activity was evaluated at pH 7.0 and 8.0. All the enzymes showed highest filter paper activity at pH 7.0. Enzyme 3 has the highest FPase activity as compared to other enzymes. FPase activity of different enzymes at 50^o C and at various pH levels are given in Table 3.

5b. Refining studies on mixed hardwood bamboo pulp (Mill-1)

The refining behavior of mixed hardwood bamboo pulp was determined at different CSF levels. The pulp required 750, 1400 and 1900 revolutions to get freeness level of 530, 490 and 460 ml CSF, respectively. Detailed results of refining behavior of control pulp collected from Mill-1 are given in the Table 4.

Physical strength properties of mixed hardwood bamboo pulp collected from Mill-1 were determined at different CSF levels without giving enzymatic treatment. The tensile, tear and burst index of the unrefined Mill-1 pulp was 41.5 Nm/g, 6.9 mNm^2/g and 2.6 kN/g, respectively. With the refining of pulp tensile and burst index improved to 67.9 Nm/g and 5.0 kN/g, respectively at 460 ml CSF. Physical strength properties of mixed hardwood bamboo pulp (Mill-1 pulp) at different CSF levels are given in Table 5.

The bleached mixed hardwood bamboo pulp collected from Mill-1 was refined in PFI mill by giving 1400 revolutions with and without enzymatic treatment using Enzyme-1. Enzymatic treatment of the pulp was given prior to refining with different dosages of Enzyme-1 i.e. 75, 100 and 150 175 g/TP. With the use of 75, 100 and 150 175 g/TP dose of enzyme prior to refining the refining energy was reduced by 8.9, 14.3 and 17.8% respectively. Detailed results of refining behavior of Mill-1 pulp with and without Enzyme-1 are given in Table 6.

Physical strength properties of pulp refined at ~500 ml CSF level with and without enzymatic treatment were determined. Tensile index and burst index of enzyme treated pulps were remained similar to that of control pulp, whereas tear index of the enzyme treated pulps marginally reduced. Detailed results of physical strength properties of Mill-1 pulp with the use of Enzyme-1 compared to control pulp are given in the Table 7.

The bleached mixed hardwood bamboo pulp collected from Mill-1 was refined in PFI mill by giving 1300 revolutions with and without enzymatic treatment using Enzyme-2. Enzymatic treatment of the pulp was given prior to refining with different dosages of Enzyme-2 i.e. 50, 100, 150 and 175 g/TP. With the use of 50,100, 150 and 175 g/t dose of enzyme prior to refining the refining energy was reduced by 15.3, 19.2, 19.2 and 26.9% respectively. Detailed results of refining behavior of Mill-1 pulp with and without Enzyme-2 are given in Table 8.

Physical strength properties of pulp refined at ~500 ml CSF level with and without the enzymatic treatment were determined. Tensile index of the Mill-1 pulp was improved by 1.9, 6.1, 6.8 and 5.0% after refining with 50, 100, 150 and 175 g/TP dosage of Enzyme-2 respectively, compared to control. Burst index of the Mill-1 pulp was improved by 2.3, 2.3, 7.0 and 9.3% after refining with 50, 100, 150 and 175 g/TP dosage of Enzyme-2 respectively, compared to control. The tear index of the pulp was reduced by 9.3% with increased dose of Enzyme-2. Smoothness of the paper was improved whereas Gurley porosity get reduced with the application of Enzyme-2 on Mill-1 pulp. Detailed results of physical strength properties of Mill-1 pulp with the use of Enzyme-2 compared to control pulp are given in the Table 9.

The bleached mixed hardwood bamboo pulp collected from Mill-1 was refined in PFI mill by giving 1350 revolutions with and without enzymatic treatment using Enzyme-3. With the use of 50,100, 150 and 175 g/t dose of enzyme prior to refining the refining energy was reduced by 14.8, 18.5, 22.2 and 29.6% respectively. Detailed results of refining behavior of Mill-1 pulp with and without Enzyme-3 are given in Table 10.

Physical strength properties of pulp refined at ~500 ml CSF level with and without the enzymatic treatment were determined. There was not much change in the tensile and burst index with the use of Enzyme-3 on Mill-1 pulp. The tear index of the pulp was reduced by 11.3 and 17% with 150 and 175 g/TP dose of Enzyme-3. Thre was improvement in double fold of the paper by 26% with the use of Enzyme-3. Smoothness of the paper get improved and Gurley porosity get reduced with the application of Enzyme-3 on Mill-1 pulp. Detailed results of physical strength properties of Mill-1 pulp with the use of Enzyme-3 compared to control pulp are given in the Table 11.

The bleached mixed hardwood bamboo pulp collected from Mill-1 was refined in PFI mill by giving 1350 revolutions with and without enzymatic treatment using Enzyme-4. With the use of 50,100, 150 and 175 g/t dose of enzyme prior to refining the refining energy was reduced by 14.8, 18.5, 22.2 and 29.6% respectively. Refining behavior of Enzyme-3 and Enzyme-4 was almost similar on the Mill-1 pulp. Detailed results of refining behavior of Mill-1 pulp with and without Enzyme-4 are given in Table 12.

Physical strength properties of pulp refined at ~500 ml CSF level with and without the treatment with Enzyme-4 were determined. There was not much change in the tensile and burst index with the use of Enzyme-4 on Mill-1 pulp. The tear index of the pulp was reduced by 2.0 and 9.4% with 150 and 175 g/TP dose of Enzyme-4. There was improvement in double fold of the paper by 23% with the use of Enzyme-4. Smoothness of the paper improved and Gurley porosity get reduced with the application of Enzyme-4 on Mill-1 pulp. Detailed results of physical strength properties of Mill-1 pulp with the use of Enzyme-4 compared to control pulp are given in the Table 13.

The bleached mixed hardwood bamboo pulp collected from Mill-1 was refined in PFI mill by giving 1200 revolutions with and without enzymatic treatment using Enzyme-5. With the use of 50, 75, 100, 150 and 175 g/t dose of enzyme prior to refining the refining energy was reduced by 6.0, 12.5, 12.5, 19.2 and 23.3% respectively. Detailed results of refining behavior of Mill-1 pulp with and without Enzyme-5 are given in Table 14.

Physical strength properties of pulp refined by giving 1200 PFI mill revolutions (450-480 ml CSF level) with and without the treatment with Enzyme-5 were determined. There was not much change in the tensile and burst index with the use of Enzyme-5 on Mill-1 pulp. The tear index of the pulp was reduced by ~5.0% with 150 and 175 g/TP dose of Enzyme-5. There was improvement in double fold of the paper by 58.5% with the use of Enzyme-5. Smoothness of the paper improved and Gurley porosity get reduced with the application of Enzyme-5 on Mill-1 pulp. Detailed results of physical strength properties of Mill-1 pulp with the use of Enzyme-5 compared to control pulp are given in the Table 15.

The bleached mixed hardwood bamboo pulp collected from Mill-1 was refined in PFI mill by giving 1350 revolutions with and without enzymatic treatment using Enzyme-6. With the use of 50, 100 and 150 g/TP of enzyme, no reduction in refining energy was obtained as compared to control. The reason behind non effectiveness of the enzyme to reduce refining energy may be due to the presence of only xylanase activity. Detailed results of refining behavior of Mill-1 pulp with and without Enzyme-6 are given in Table 16.

The bleached mixed hardwood bamboo pulp collected from Mill-1 was refined in PFI mill by giving 1350 revolutions with and without enzymatic treatment using Enzyme-7. With the use of 50, 100 and 150 g/TP dose of Enzyme-7 no reduction in refining energy was obtained as compared to control. The reason behind non effectiveness of the Enzyme-7 to reduce refining energy may be due to the absence of CMC and filter paper activity and presence of only xylanase activity. Detailed results of refining behavior of Mill-1 pulp with and without Enzyme-7 are given in Table 17.

5c. Refining studies on mixed hardwood pulp (Mill-2)

The refining behavior of Mill-2 pulp (mixed hardwood pulp) was determined at different CSF levels. The pulp required 750, 1250 and 1700 revolutions to get freeness level of 540, 500 and 480 ml CSF, respectively. Detailed results of refining behavior of control pulp collected from Mill-2 are given in the Table 18.

Physical strength properties of mixed hardwood bamboo pulp collected from Mill-2 were determined at different CSF levels without giving enzymatic treatment. The tensile, tear and burst index of the unrefined Mill-2 pulp was 39.8 Nm/g, 4.8 mNm^2/g and 3.7 kN/g, respectively. With the refining of pulp tensile and burst index improved to 64.6 Nm/g and 4.9 kN/g, respectively at 480 ml CSF. Physical strength properties of mixed hardwood pulp (Mill-2 pulp) at different CSF levels are given in Table 19.

The bleached mixed hardwood pulp collected from Mill-2 was refined in PFI mill by giving 1250 revolutions with and without enzymatic treatment using Enzyme-1. Enzymatic treatment of the pulp was given prior to refining with different dosages of Enzyme-1 i.e. 50, 100 and 150 g/TP. With the use of 50, 100 and 150 175 g/TP dose of enzyme prior to refining the refining energy was reduced by 8, 16 and 20% respectively. Detailed results of refining behavior of Mill-2 pulp with and without Enzyme-1 are given in Table 20.

Physical strength properties of pulp refined at ~500 ml CSF level with and without enzymatic treatment were determined. Tensile index and burst index of enzyme treated pulps were improved marginally compared to control pulp, whereas tear index of the enzyme treated pulps were marginally reduced. Detailed results of physical strength properties of Mill-2 pulp with the use of Enzyme-1 compared to control pulp are given in the Table 21.

The bleached mixed hardwood pulp collected from Mill-2 was refined in PFI mill by giving 1250 revolutions with and without enzymatic treatment using Enzyme-2. With the use of 75 and 150 g/t of enzyme, a reduction of 15-21 % in refining energy requirement was obtained as compared to control. Detailed results of refining behavior of Mill-2 pulp with and without Enzyme-2 are given in Table 22.

Physical strength properties of pulp refined at 500 ml CSF level with and without the enzymatic treatment were determined. Strength properties like tensile index, tear index, burst index and double fold were found comparable. Detailed results of physical strength properties of Mill-2 pulp with the use of Enzyme-2 compared to control pulp are given in the Table 23.

The bleached mixed hardwood pulp collected from Mill-2 was refined in PFI mill by giving 1250 revolutions with and without enzymatic treatment using Enzyme-3. With the use of 50,100 and 150 g/t of enzyme, a reduction of 14.8-25.5% in refining energy requirement was obtained as compared to control. Detailed results of refining behavior of Mill-2 pulp with and without Enzyme-3 are given in Table 24.

Physical strength properties of pulp refined at 500 ml CSF level with and without the enzymatic treatment were determined. Strength properties like tensile index, burst index and double fold were found comparable whereas tear index was slightly reduced (7%). Detailed results of physical strength properties of Mill-2 pulp with the use of Enzyme-3 compared to control pulp are given in the Table 25.

The bleached mixed hardwood pulp collected from Mill-2 was refined in PFI mill by giving 1250 revolutions with and without enzymatic treatment using Enzyme-4. With the use of 50,100 and 150 g/t of enzyme, a reduction of 12.1-22.2% in refining energy requirement was obtained as compared to control. Detailed results of refining behavior of Mill-2 pulp with and without Enzyme-4 are given in Table 26.

Physical strength properties of pulp refined at 500 ml CSF level with and without the enzymatic treatment were determined. Strength properties like tensile index, tear index, burst index and double fold were found comparable, whereas tear index was slightly reduced. Detailed results of physical strength properties of Mill-2 pulp with the use of Enzyme-4 compared to control pulp are given in the Table 27.

The bleached mixed hardwood pulp collected from Mill-2 was refined in PFI mill by giving 1250 revolutions with and without enzymatic treatment using Enzyme-5. With the use of

75 and 150 g/t of enzyme, a reduction of 20-25 % in refining energy requirement was obtained as compared to control. Detailed results of refining behavior of Mill-2 pulp with and without Enzyme-5 are given in Table 28.

Physical strength properties of pulp refined at 500 ml CSF level with and without the enzymatic treatment were determined. Strength properties like tensile index, tear index, burst index and double fold were found comparable. Detailed results of physical strength properties of Mill-2 pulp with the use of Enzyme-5 compared to control pulp are given in the Table 29.

5d. Observations and conclusions

The enzymatic products Enzyme-4, Enzyme-3 and Enzyme-2 were found to be more effective in reducing refining energy requirements targeting the same strength properties as that of control. Enzyme-3 and Enzyme-4 were capable to reduce refining energy by 29.6% whereas Enzyme-2 was able to reduce refining energy by 26.9% in the laboratory scale experiments carried out at TCIRD.

5e. Tables and figures

Table 1: CMC activity (IU/ml) in enzymes at various pH (at 50^{o} C)

pH	Enzyme-1	Enzyme-2	Enzyme-3	Enzyme-4	Enzyme-5
6.0	91.3	213.5	220.9	94.9	153.9
7.0	200.5	289.1	383.7	217.7	220.2
8.0	121.7	137.1	145.8	111.5	60.5

Table 2: Xylanase (IU/ml) activity in enzymes at various pH (at 50^{o}C)

pH	Enzyme-1	Enzyme-2	Enzyme-3	Enzyme-4	Enzyme-5	Enzyme-6	Enzyme-7
6.0	3347.7	13694	2156.6	183.3	804.6	10984	13347
7.0	3748	11406	1429.1	602.6	1324.3	10626	15156
8.0	683.3	3354.9	591.4	116.7	332.2	8108	10733

Table 3: FPase (IU/ml) activity in different enzymes at various pH (at 50^{o} C)

pH	Enzyme-1	Enzyme-2	Enzyme-3	Enzyme-4	Enzyme-5
7.0	9.7	12.2	22.9	7.3	7.0
8.0	6.7	7.4	12.0	5.3	3.0

Table 4: Refining behavior of control pulp collected from Mill-1

Revolution (no.)	CSF (ml)
0	585
750	530
1400	490
1900	460

Table 5: Physical strength properties of Mill-1 pulp at different CSF levels

Particular	Control			
CSF (ml)	585	530	490	460
Substance (g/m^2)	72.6	71.9	72.7	71.7
Bulk (cc/g)	1.57	1.49	1.31	1.25
Tensile index (N.m/g)	41.51	55.61	63.73	67.91
Breaking length (m)	4233	5671	6498	6925
Burst factor	26.5	39.8	44.9	51.0
Burst index (kN/g)	2.6	3.9	4.4	5.0
Tear factor	70.4	71.4	75.5	69.4
Tear index (mN.m^2/g)	6.9	7.0	7.4	6.8
Porosity (sec/100 ml)	3.48	6.89	14.33	22.18
Double fold (no.)	10	28	65	139
Smoothness (ml/min)	187	139	136	112

Table 6: Effect of Enzyme-1 on refining behavior of Mill-1 pulp

Particular	Results			
Enzyme dose (g/t)	0	75	100	150
PFI Revolutions	1400	1400	1400	1400
Freeness of pulp CSF (ml)	490	480	475	470
Difference in CSF	--	-10	-15	-20
PFI revolution to get similar CSF	1400	1275	1200	1150
Reduction in refining energy (%)	--	8.9	14.28	17.8

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 7: Effect of Enzyme-1 on physical strength properties of Mill-1 pulp

Particular	Control	Enzyme-1		
Enzyme dose g/t	--	75	100	150
Revolution (no.)	1400	1400	1400	1400
CSF(ml)	490	480	475	470
Difference in CSF	--	-10	-15	-20
Substance (g/m^2)	72.7	71.7	71.5	71.9
Bulk (cc/g)	1.31	1.33	1.34	1.32
Tensile index (Nm/g)	63.73	64.7	64.2	64.9
Breaking length (m)	6498	6597	6543	6618
Burst factor	44.9	46.1	45.9	45.3
Burst index (kN/g)	4.4	4.5	4.5	4.4
Tear factor	75.5	73.4	72.4	71.9
Tear index (mN m^2/g)	7.4	7.2	7.1	7.0
Gurley Porosity (sec/100 ml)	14.33	14.78	14.61	15.24
Double fold (no.)	65	82	95	87
Smoothness (ml/min)	136	135	129	126

Table 8: Effect of Enzyme-2 on refining behavior of Mill-1 pulp

Particular	Results				
Enzyme dose (g/t)	0	50	100	150	175
PFI Revolutions	1300	1300	1300	1300	1300
Freeness of pulp CSF (ml)	510	495	490	490	478
Difference in CSF	--	-15	-20	-20	-32
PFI revolution to get similar CSF	--	1100	1050	1050	950
Reduction in refining energy (%)		15.3	19.2	19.2	26.9

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 9: Effect of Enzyme-2 on physical strength properties of Mill-1 pulp

Particular	Control	Enzyme-2			
Enzyme dose g/t	--	50	100	150	175
CSF(ml)	510	495	490	490	478
Substance (g/m^2)	72.6	73.03	73.3	72.6	70.3
Bulk (cc/g)	1.32	1.33	1.34	1.34	1.32
Tensile index (Nm/g)	57.33	58.41	60.83	61.22	60.21
Breaking length (m)	5846	5956	6183	6242	6140
Burst factor	43.9	44.9	44.9	46.9	47.9
Burst index (kN/g)	4.3	4.4	4.4	4.6	4.7
Tear factor	55.1	53.0	51.0	50.0	51.0
Tear index (mN m^2/g)	5.4	5.2	5.0	4.9	5.0
Gurley Porosity (sec/100 ml)	11.11	15.83	16.50	18.43	19.04
Double fold (no.)	52	65	106	112	123
Smoothness (ml/min)	113	99	92	95	91

Table 10: Effect of Enzyme-3 on refining behavior of Mill-1 pulp

Particular	Results				
Enzyme dose (g/t)	0	50	100	150	175
PFI Revolutions	1350	1350	1350	1350	1350
Freeness of pulp CSF (ml)	495	485	480	470	464
Reduction in CSF	--	-10	-15	-25	-31
PFI revolution to get similar CSF	1350	1150	1100	1050	950
Reduction in refining energy (%)	--	14.8	18.5	22.2	29.6

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 11: Effect of Enzyme-3 on physical strength properties of Mill-1 pulp

Particulars	Control	Enzyme-3			
Enzyme dose (g/t)	--	50	100	150	175
CSF(ml)	495	485	480	470	464
Substance (g/m^2)	72.4	73.1	72.4	69.5	70.7
Bulk (cc/g)	1.25	1.24	1.24	1.24	1.26
Tensile index (Nm/g)	56.2	56.5	56.6	57.5	58.4
Breaking length (m)	5729	5758	5771	5866	5954
Burst factor	43.9	43.9	43.9	42.8	41.8
Burst index (kN/g)	4.3	4.3	4.3	4.2	4.1
Tear factor	54.1	51.0	50.0	47.9	44.9
Tear index (mN m^2/g)	5.3	5.0	4.9	4.7	4.4
Gurley Porosity (sec/100 ml)	14.5	16.6	17.8	18.6	18.9
Double fold (no.)	77	78	99	91	97
Smoothness (ml/min)	112	87	80	74	67

Table 12: Effect of Enzyme-4 on refining behavior of Mill-1 pulp

Particular	Results				
Enzyme dose (g/t)	0	50	100	150	175
PFI Revolutions	1350	1350	1350	1350	1350
Freeness of pulp CSF (ml)	495	485	480	472	462
Reduction in CSF	--	-10	-15	-23	-33
PFI revolution to get similar CSF	--	1150	1100	1050	950
Reduction in refining energy (%)	--	14.8	18.5	22.2	29.6

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 13: Effect of Enzyme-4 on physical strength properties of Mill-1 pulp

Particular	Control	Enzyme-4			
Enzyme dose g/t	--	50	100	150	175
CSF(ml)	495	485	480	472	462
Substance (g/m^2)	69.2	71.3	69.5	71.5	69.7
Bulk (cc/g)	1.27	1.26	1.26	1.26	1.28
Tensile index (Nm/g)	56.2	57.6	57.2	57.6	57.7
Breaking length (m)	5732	5873	5826	5875	5883
Burst factor	45.9	44.9	44.9	46.9	47.0
Burst index (kN/g)	4.5	4.4	4.4	4.6	4.61
Tear factor	54.1	52.0	52.0	53.0	49.0
Tear index (mN m^2/g)	5.3	5.1	5.1	5.2	4.8
Gurley Porosity (sec/100 ml)	15.90	18.6	19.83	20.73	20.91
Double fold (no.)	87	82	111	99	107
Smoothness (ml/min)	98	87	86	88	86

Table 14: Effect of Enzyme-5 enzyme on refining behavior of Mill-1 pulp

Particular	Results					
Enzyme dose (g/t)	0	50	75	100	150	175
PFI Revolutions	1200	1200	1200	1200	1200	1200
Freeness of pulp CSF (ml)	482	476	470	467	454	447
Reduction in CSF	--	-6	-12	-15	-28	-35
PFI revolution to get similar CSF	--	1140	--	1050	970	920
Reduction in refining energy (%)	--	6.0	--	12.5	19.2	23.3

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 15: Effect of Enzyme-5 on physical strength properties of Mill-1 pulp

Particular	Control	Enzyme-5				
Enzyme dose (g/t)	--	50	75	100	150	175
CSF (ml)	482	476	470	467	454	447
Substance (g/m^2)	70.4	72.6	71.9	71.9	71.7	70.7
Bulk (cc/g)	1.31	1.32	1.33	1.33	1.34	1.31
Tensile index (Nm/g)	61.34	61.47	58.68	59.79	58.83	59.17
Breaking Length (m)	6255	6268	5984	6097	5999	6033
Tear Index (mNm2/g)	4.60	4.55	4.50	4.73	4.84	4.82
Tear Factor	46.9	46.4	45.9	48.2	49.4	49.2
Burst Index (kN/g)	4.21	4.29	4.40	4.19	4.40	4.21
Burst Factor	42.9	43.8	44.9	42.7	44.9	42.9
Double fold (nos.)	53	63	76	84	80	78
Porosity (sec/100ml)	9.8	9.9	10.6	10.4	12.1	14.0
Smoothness (ml/min)	110	103	96	95	87	86

Table 16: Effect of Enzyme-6 on refining behavior of Mill-1 pulp

Particular	Results			
Enzyme dose (g/t)	0	50	100	150
PFI Revolutions	1350	1350	1350	1350
Freeness of pulp CSF (ml)	495	505	505	510
Reduction in CSF	--	+10	+10	+15
PFI revolution to get similar CSF	--	--	--	--
Reduction in refining energy (%)	--	--	--	--

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 17: Effect of Enzyme-7 on refining behavior of Mill-1 pulp

Particular	Results			
Enzyme dose (g/t)	0	50	100	150
PFI Revolutions	1350	1350	1350	1350
Freeness of pulp CSF (ml)	500	498	510	510
Reduction in CSF		-2	+10	+10
PFI revolution to get similar CSF	--	--	--	--
Reduction in refining energy (%)	--	--	--	--

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 18: Refining behavior of mixed hardwood pulp collected from Mill-2

PFI revolutions (no.)	CSF(ml)
0	570
750	540
1250	500
1700	480

Table 19: Physical strength properties of mixed hardwood pulp collected from Mill-2

Particular	Results			
CSF(ml)	570	540	500	480
Substance (g/m^2)	67.8	70.6	70.5	69.8
Bulk (cc/g)	1.36	1.24	1.20	1.15
Tensile index (Nm/g)	39.8	56.8	63.3	64.6
Breaking length (m)	4056	5791	6455	6586
Burst factor	37.7	42.8	47.9	50.0
Burst index (kN/g)	3.7	4.2	4.7	4.9
Tear factor	49.0	57.1	55.1	50.0
Tear index (mN m^2/g)	4.8	5.6	5.4	4.9
Gurley Porosity (sec/100 ml)	9.8	26.3	33.9	55.1
Double fold (no.)	13	59	143	135
Smoothness (ml/min)	109	93	77	67

Table 20: Effect of Enzyme-1 on refining behavior of Mill-2 pulp

Particular	Results			
Enzyme dose (g/t)	0	50	100	150
PFI Revolutions	1250	1250	1250	1250
Freeness of pulp CSF (ml)	498	490)	485)	478)
Reduction in CSF		-8	-13	-20
PFI revolution to get similar CSF	--	1150	1050	1000
Reduction in refining energy (%)		8%	16	20

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 21: Effect of Enzyme-1 on physical strength properties of Mill-2 pulp

Particular	Control	Enzyme 1		
Enzyme dose (g/t)	0	50	100	150
Revolution (no.)	1300	1300	1300	1300
CSF(ml)	498	490	485	478
Difference in CSF	--	-8	-13	-20
Substance (g/m^2)	72.4	73.2	72.7	73.7
Bulk (cc/g)	1.23	1.20	1.22	1.21
Tensile index (Nm/g)	63.3	60.81	64.0	65.8
Breaking length (m)	6455	6201	6527	6716
Burst factor	50.0	51.0	52.0	51.0
Burst index (kN/g)	4.9	5.0	5.1	5.0
Tear factor	58.1	58.1	56.1	55.1
Tear index (mN m^2/g)	5.7	5.7	5.5	5.4
Gurley Porosity (sec/100 ml)	43.1	45.5	42.8	45.9
Double fold (no.)	143	123	158	180
Smoothness (ml/min)	81	91	82	81

Table 22: Effect of Enzyme-2 on refining behavior of Mill-2 pulp

Particular	Results		
Enzyme dose (g/t)	0	75	150
PFI Revolutions	1400	1400	1400
Freeness of pulp CSF (ml)	480	465	460
Reduction in CSF		-15	-20
PFI revolution to get similar CSF	--	1200	1100
Reduction in refining energy (%)	--	15	21

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 23: Effect of Enzyme-2 on physical strength properties of Mill-2 pulp

Particular	Control	Enzyme-2	
Enzyme dose (g/t)	0	75	150
Revolution (no.)	1400	1400	1400
CSF(ml)	480	465	460
Difference in CSF	--	-15	-20
Substance (g/m^2)	70.6	70.5	71.3
Bulk (cc/g)	1.23	1.24	1.23
Tensile index (Nm/g)	58.9	62.1	63.4
Breaking length (m)	6006	6332	6465
Burst factor	45.9	48.9	47.9
Burst index (kN/g)	4.5	4.8	4.7
Tear factor	58.1	57.1	56.1
Tear index (mN m^2/g)	5.7	5.6	5.5
Gurley Porosity (sec/100 ml)	39.2	43.6	44.4
Double fold (no.)	38	52	50
Smoothness (ml/min)	76	66	75

Table 24: Effect of Enzyme-3 on refining behavior of Mill-2 pulp

Particular	Results			
Enzyme dose (g/t)	0	50	100	150
PFI Revolutions	1350	1350	1350	1350
Freeness of pulp CSF (ml)	515	505)	502)	495)
Reduction in CSF		-10	-13	-20
PFI revolution to get similar CSF	--	1150	1100	1050
Reduction in refining energy (%)		14.8	18.5	22.2

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 25: Effect of Enzyme-3 on physical strength properties of Mill-2 pulp

Particular	Control	Enzyme-3		
Enzyme dose (g/t)	--	50	100	150
Revolution (no.)	1350	1350	1350	1350
CSF(ml)	515	505	502	495
Difference in CSF	--	-10	-13	-20
Substance (g/m^2)	73.4	70.5	71.8	73.1
Bulk (cc/g)	1.26	1.27	1.26	1.26
Tensile index (Nm/g)	65.45	65.78	66.55	67.24
Breaking length (m)	6673	6707	6786	6856
Burst factor	46.9	45.9	46.9	45.9
Burst index (kN/g)	4.6	4.5	4.6	4.5
Tear factor	58.1	56.1	54.1	54.1
Tear index (mN m^2/g)	5.7	5.5	5.3	5.3
Gurley Porosity (sec/100 ml)	37.84	38.97	41.58	44.53
Double fold (no.)	45	54	67	74
Smoothness (ml/min)	69	64	51	44

Table 26: Effect of Enzyme-4 on refining behavior of Mill-2 pulp

Particular	Results			
Enzyme dose (g/t)	0	50	100	150
PFI Revolutions	1400	1400	1400	1400
Freeness of pulp CSF (ml)	495	487	482	477
Reduction in CSF	--	-8	-13	-18
PFI revolution to get similar CSF	1400	1150	1100	1050
Reduction in refining energy (%)	--	14.8	18.5	22.2

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 27: Effect of Enzyme-4 on physical strength properties of Mill-2 pulp

Particular	Control	Enzyme-4		
Enzyme dose g/t	--	50	100	150
CSF(ml)	495	487	482	477
Substance (g/m^2)	71.7	72.8	70.7	69.8
Bulk (cc/g)	1.27	1.27	1.28	1.28
Tensile index (Nm/g)	64.17	64.86	65.43	65.71
Breaking length (m)	6543	6613	6672	6701
Burst factor	48.0	49.0	49.0	49.0
Burst index (kN/g)	4.7	4.8	4.8	4.8
Tear factor	59.2	59.2	57.1	57.1
Tear index (mN m^2/g)	5.8	5.8	5.6	5.6
Gurley Porosity (sec/100 ml)	37.86	40.15	41.41	43.54
Double fold (no.)	78	84	99	117
Smoothness (ml/min)	65	59	52	42

Table 28: Effect of Enzyme-5 on refining behavior of pulp 2

Particular	Results		
Enzyme dose (g/t)	0	75	150
PFI Revolutions	1400	1400	1400
Freeness of pulp CSF (ml)	480	460	450
Reduction in CSF	--	-20	-30
PFI revolution to get 500 ml CSF	--	1100	1050
Reduction in refining energy (%)	--	21	25

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 29: Effect of Enzyme-5 on physical strength properties of Mill-2 pulp

Particular	Control	Enzyme-5	
Enzyme dose (g/t)	--	75	150
Revolution (no.)	1400	1400	1400
CSF(ml)	480	460	450
Difference in CSF	--	-20	-30
Substance (g/m^2)	70.6	70.2	72.1
Bulk (cc/g)	1.23	1.22	1.22
Tensile index (Nm/g)	58.9	60.2	62.2
Breaking length (m)	6006	6138	6343
Burst factor	45.9	46.9	46.9
Burst index (kN/g)	4.5	4.6	4.6
Tear factor	58.1	56.1	57.1
Tear index (mN m^2/g)	5.7	5.5	5.6
Gurley Porosity (sec/100 ml)	39.2	40.4	45.3
Double fold (no.)	38	63	46
Smoothness (ml/min)	76	66	65

Enzymatic drainage improvement

of

Wood/ Bamboo pulp

6. RESULTS AND DISCUSSION - Enzymatic drainage improvement of wood/ Bamboo pulp

6a. Drainage improvement studies on mixed hardwood bamboo pulp (Mill-1)

For the enzymatic drainage improvement study the enzyme treated (dosage 75 g/t) pre refined pulp with 1050 PFI revolutions was taken. Second stage enzymatic treatment was given to the pulp with 40, 60 and 80 g/TP dosage of enzyme-1. Slight improvement of 2.3-4.5 % in drainage was obtained as compared to control with the use of Enzyme-1 in post refining stage. Detailed results of two stage treatment of Enzyme-1 on drainage of Mill-1 pulp are given in Table 30.

Physical strength properties of pulp refined at similar CSF levels with (2 stage treatments) and without the enzymatic treatment were determined. Strength properties like tear index, burst index and double fold were found comparable whereas tensile index was slightly improved (5%). Detailed results of two stage treatment of Enzyme-1 on physical strength properties of Mill-1 pulp compared to control pulp are given in the Table 31

The mill pulp samples both treated with enzyme and untreated were refined at different PFI revolutions (reduced in case of enzyme treated) with a target of 500 ml CSF. With the use of 75 g/t dosage of enzyme in post refining stage i.e. 2nd enzyme treatment stage, an improvement of 6.1-10.7 % in drainage was obtained as compared to control. Detailed results of two stage treatment of Enzyme-2 on drainage of Mill-1 pulp are given in Table 32.

Physical strength properties of pulp refined at similar CSF levels with (2 stage treatments) and without using enzyme (Control) were determined. Strength properties like tensile index, tear index, burst index and double fold were found comparable. Detailed results of two stage treatment of Enzyme-2 on physical strength properties of Mill-1 pulp compared to control pulp are given in the Table 33.

The mill pulp samples both treated with enzyme (Dosage 50 &100 g/t) and untreated were refined at different PFI revolutions with a target of 500 ml CSF. With the use of 75 g/t dosage of enzyme in post refining stage i.e. 2nd enzyme treatment stage, an improvement of 8.4-17.1 % in drainage was obtained as compared to control. Detailed results of two stage treatment of Enzyme-3 on drainage of Mill-1 pulp are given in Table 34.

Physical strength properties of pulp refined at similar CSF levels with (2 stage treatments) and without the enzymatic treatment were determined. Strength properties like tensile index, burst index and double fold were found comparable, whereas tear index was reduced to certain extent (6%). Detailed results of two stage treatment of Enzyme-3 on physical strength properties of Mill-1 pulp compared to control pulp are given in the Table 35.

The mill pulp samples both treated with enzyme and untreated were refined at different PFI revolutions with a target of 500 ml CSF. With the use of 75 g/t dosage of enzyme in post refining stage i.e 2nd enzyme treatment stage, an improvement of 4.2-8.7 % in drainage was obtained as compared to control. Detailed results of two stage treatment of Enzyme-4 on drainage of Mill-1 pulp are given in Table 36.

Physical strength properties of pulp refined at similar CSF levels with (2 stage treatments) and without the enzymatic treatment were determined. Strength properties like tensile index, tear index, burst index and double fold were found almost comparable. Detailed results of two stage treatment of Enzyme-4 on physical strength properties of Mill-1 pulp compared to control pulp are given in the Table 37.

6b. Drainage improvement studies on mixed hardwood pulp (Mill-2)

The mill pulp samples both treated with enzyme and untreated were refined in PFI mill to get revolutions with a target of 500 ml CSF. With the use of 75 g/t dosage of enzyme in post refining stage i.e 2nd enzyme treatment stage, an improvement of 11-14% in drainage was obtained as compared to control. Detailed results of two stage treatment of Enzyme-2 on drainage of Mill-2 pulp are given in Table 38.

Physical strength properties of pulp refined at similar CSF levels with (2 stage treatments) and without the enzymatic treatment were determined. Strength properties like tensile index, tear index, burst index and double fold were found comparable. Detailed results of two stage treatment of Enzyme-2 on physical strength properties of Mill-2 pulp compared to control pulp are given in the Table 39.

The mill pulp samples both treated with enzyme (Dosage 50 &100 g/t) and untreated were refined at different PFI revolutions (reduced in case of enzyme treated) with a target of 500 ml CSF. With the use of 75g/t dosage of enzyme in post refining stage i.e. 2nd enzyme treatment stage, an improvement of 5.4-16.3 % in drainage was obtained as compared to control. Detailed results of two stage treatment of Enzyme-3 on drainage of Mill-2 pulp are given in Table 40.

Physical strength properties of pulp refined at similar CSF levels with (2 stage treatments) and without the enzymatic treatment were determined. Strength properties like tensile index, burst index and double fold were found comparable whereas a slight reduction in tear index was found (7%). Detailed results of two stage treatment of Enzyme-3 on physical strength properties of Mill-2 pulp compared to control pulp are given in the Table 41.

The mill pulp samples both treated with enzyme and untreated were refined at different PFI revolutions with a target of 500 ml CSF. With the use of 75 g/t dosage of enzyme in post refining stage i.e 2^{nd} enzyme treatment stage, a slight improvement of 0.9-2.8 % in drainage was obtained as compared to control. Detailed results of two stage treatment of Enzyme-4 on drainage of Mill-2 pulp are given in Table 42.

Physical strength properties of pulp refined at similar CSF levels with (2 stage treatments) and without the enzymatic treatment were determined. Strength properties like tensile index, tear index, burst index and double fold were found comparable. Detailed results of two stage treatment of Enzyme-4 on physical strength properties of Mill-2 pulp compared to control pulp are given in the Table 43.

6c. Observations and conclusion

- Enzyme 3 and Enzyme 2 were found effective in improving the drainage of pulp.
- Enzyme-3 was found most effective to improve drainage, as it improved the drainage of the pulp up to 17.1%.
- Tear index was a little reduced with treatment with Enzyme-3 at higher dose.

6d. Tables and figures

Table 30: Effect of two stage treatment of Enzyme-1 on drainage of Mill-1 pulp

Particular	Results			
1st Enzyme Treatment *(Enzyme dose - 75 g/t, PFI revolutions – 1050, CSF – 505)*				
2nd Enzyme Treatment (*Time - 60 min, temp. 40 ^{O}C, pH 7.0)*				
Enzyme dose (g/t)	0	40	60	80
CSF(ml)	500	505	495	500
Difference in CSF(ml)	--	+5	-5	0
Drainage time (sec) for 900 ml	47.18	46.11	45.23	45.08
Improvement in drainage (%)	--	2.3	4.1	4.5

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 31: Effect of two stage treatment of Enzyme-1 on physical strength properties of Mill-1 pulp

Particular	Results			
CSF(ml)	500	505	495	500
Substance (g/m^2)	73.2	71.1	72.3	72.6
Bulk (cc/g)	1.35	1.34	1.36	1.36
Tensile index (Nm/g)	55.2	57.7	58.1	58.3
Breaking length (m)	5631	5887	5921	5940
Burst factor	45.9	45.9	46.9	46.9
Burst index (kN/g)	4.5	4.5	4.6	4.6
Tear factor	55.1	52.0	53.0	53.0
Tear index ($mN\ m^2/g$)	5.4	5.1	5.2	5.2
Gurley Porosity (sec/100 ml)	12.4	11.4	12.1	11.2
Double fold (no.)	118	125	121	131
Smoothness (ml/min)	134	109	108	106

Table 32: Effect of two stage treatment of Enzyme-2 on drainage of Mill-1 pulp

Particular	Results			
1st Enzyme Treatment				
Enzyme dose (g/t)	--	--	50	100
PFI Revolution (no.)	1300	1300	1100	1050
CSF(ml)	500	496	503	500
2nd Enzyme Treatment				
Enzyme dose (g/t)	--	75	75	75
CSF(ml)	495	510	515	520
Difference in CSF(ml)	--	+15	+20	+25
Drainage time (sec) for 900 ml	48.31	45.38	44.18	43.17
Improvement in drainage (%)	--	6.1	8.5	10.7

Enzyme treatment conditions: time 60 min, temp. 40 ^{O}C, pH 7.0

Table 33: Effect of two stage treatment of Enzyme-1 on physical strength properties of Mill-1 pulp

Particular	Results			
CSF(ml)	495	510	515	520
Substance (g/m^2)	73.4	73.3	72.1	73.4
Bulk (cc/g)	1.32	1.32	1.33	1.34
Tensile index (Nm/g)	55.6	56.0	56.3	57.2
Breaking length (m)	5670	5710	5740	5832
Burst factor	46.9	49.0	50.0	47.9
Burst index (kN/g)	4.6	4.8	4.9	4.7
Tear factor	56.1	55.1	54.1	55.1
Tear index (mN m^2/g)	5.5	5.4	5.3	5.4
Gurley Porosity (sec/100 ml)	15.8	19.5	18.6	18.4
Double fold (no.)	78	87	105	118
Smoothness (ml/min)	133	101	111	106

Table 34: Effect of two stage treatment of Enzyme-3 on drainage of Mill-1 pulp

Particular	Results			
1st Enzyme Treatment				
Enzyme dose (g/t)	--	--	50	100
Revolution (no.)	1350	1350	1150	1100
CSF (ml)	495	500	505	502
2nd Enzyme Treatment				
Enzyme dose (g/t)	--	75	75	75
CSF (ml)	495	515	518	525
Difference in CSF (ml)	--	+20	+23	+30
Drainage time (sec) for 900 ml	44.28	40.58	39.52	36.71
Improvement in drainage %	--	8.4	10.7	17.1

Table 35: Effect of two stage treatment of Enzyme-3 on physical strength properties of Mill-1 pulp

Particular	Results			
CSF(ml)	495	515	518	525
Substance (g/m^2)	71.3	70.4	71.1	72.1
Bulk (cc/g)	1.26	1.25	1.25	1.27
Tensile index (Nm/g)	54.93	54.95	55.01	55.38
Breaking length (m)	5601	5603	5609	5647
Burst factor	41.8	41.8	41.8	42.8
Burst index (kN/g)	4.1	4.1	4.1	4.2
Tear factor	50.0	49.0	46.9	46.9
Tear index (mN m^2/g)	4.9	4.8	4.6	4.6
Gurley Porosity (sec/100 ml)	11.75	12.73	14.97	16.87
Double fold (no.)	77	85	97	109
Smoothness (ml/min)	110	100	97	82

Table 36: Effect of two stage treatment of Enzyme-4 on drainage of Mill-1 pulp

Particular	Results			
1st Enzyme Treatment				
Enzyme dose (g/t)	--	50	100	150
Revolutions (no.)	1350	1200	1150	1100
CSF(ml)	495	498	502	502
2nd Enzyme Treatment				
Enzyme dose (g/t)	--	75	75	75
CSF(ml)	495	500	500	505
Difference in CSF(ml)	--	+5	+5	+10
Drainage time (sec) for 900 ml	46.5	46.7	44.5	42.4
Improvement in drainage %	--	--	4.2	8.7

Table 37: Effect of two stage treatment of Enzyme-4 on physical strength properties of Mill-1 pulp

Particular	Results			
CSF(ml)	495	500	500	505
Substance (g/m^2)	71.3	71.7	70.7	73.1
Bulk (cc/g)	1.26	1.26	1.27	1.26
Tensile index (Nm/g)	55.44	55.22	56.13	56.03
Breaking length (m)	5653	5630	5723	5713
Burst factor	42.8	43.9	43.9	44.9
Burst index (kN/g)	4.2	4.3	4.3	4.4
Tear factor	49.0	47.9	46.9	46.9
Tear index (mN m^2/g)	4.8	4.7	4.6	4.6
Gurley Porosity (sec/100 ml)	12.84	15.71	15.87	17.56
Double fold (no.)	75	83	90	92
Smoothness (ml/min)	117	97	95	87

Table 38: Effect of two stage treatment of Enzyme-2 on drainage of Mill-2 pulp

Particular	Results		
1st Enzyme Treatment			
Enzyme dose (g/t)	--	75	100
Revolution (no.)	1400	1200	1100
CSF(ml)	495	498	500
2nd Enzyme Treatment			
Enzyme dose (g/t)	--	75	75
CSF(ml)	490	505	505
Difference in CSF(ml)	--	+15	+15
Drainage time (sec) for 800 ml	31.3	27.8	26.9
Improvement in drainage (%)	--	11.2	14.2

Table 39: Effect of two stage treatment of Enzyme-2 on physical strength properties of Mill-2 pulp

Particular	Results		
CSF(ml)	490	505	505
Substance (g/m^2)	71.9	73.2	70.2
Bulk (cc/g)	1.20	1.21	1.21
Tensile index (Nm/g)	63.6	65.7	64.4
Breaking length (m)	6485	6698	6566
Burst factor	47.9	48.9	46.9
Burst index (kN/g)	4.7	4.8	4.6
Tear index (mN m^2/g)	5.3	4.8	4.8
Tear factor	54.1	49.1	49.1
Gurley Porosity (sec/100 ml)	51.7	54.2	52.4
Double fold (no.)	47	66	50
Smoothness (ml/min)	65	60	62

Table 40: Effect of two stage treatment of Enzyme-3 on drainage of Mill-2 pulp

Particular	Results			
1st Enzyme Treatment				
Enzyme dose (g/t)	--	--	50	100
Revolution (no.)	1350	1350	1150	1100
CSF(ml)	498	500	500	505
2nd Enzyme Treatment				
Enzyme Dose (g/t)	--	75	75	75
CSF(ml)	495	507	520	527
Difference in CSF(ml)	--	+12	+25	+32
Drainage time (sec) for 900 ml	49.18	46.48	42.46	41.17
Improvement in drainage (%)	--	5.4	14.2	16.3

Table 41: Effect of two stage treatment of Enzyme-3 on physical strength properties of Mill-2 pulp

Particular	Ist Enzyme Treatment			
CSF(ml)	495	507	520	527
Substance (g/m^2)	71.9	73.2	70.2	70.4
Bulk (cc/g)	1.28	1.27	1.29	1.29
Tensile index (Nm/g)	62.74	64.54	63.80	64.34
Breaking length (m)	6398	6581	6505	6561
Burst factor	43.9	44.9	46.0	46.0
Burst index (kN/g)	4.3	4.4	4.6	4.5
Tear factor	59.2	58.1	56.1	55.1
Tear index (mN m^2/g)	5.8	5.7	5.5	5.4
Gurley Porosity (sec/100 ml)	35.73	38.63	41.68	47.3
Double fold (no.)	47	66	79	83
Smoothness (ml/min)	59	57	50	48

Table 42: Effect of two stage treatment of Enzyme-4 on drainage of Mill-2 pulp

Particular	Results			
1st Enzyme Treatment				
Enzyme dose (g/t)	--	--	50	100
Revolution (no.)	1350	1350	1150	1100
CSF(ml)	495	495	493	500
2nd Enzyme Treatment				
Enzyme dose (g/t)	--	75	75	75
CSF(ml)	490	490	485	490
Difference in CSF(ml)	--	--	-5	--
Drainage time (sec) for 900 ml	47.18	47.44	46.75	45.88
Improvement in drainage %	--	--	0.9	2.8

Table 43: Effect of two stage treatment of Enzyme-4 on physical strength properties of Mill-2 pulp

Particular	Results			
CSF(ml)	490	490	485	490
Substance (g/m^2)	72.7	72.9	73.1	73.3
Bulk (cc/g)	1.28	1.27	1.27	1.27
Tensile index (Nm/g)	62.59	63.04	62.50	63.42
Breaking length (m)	6383	6428	6373	6467
Burst factor	45.9	45.9	46.9	46.9
Burst index (kN/g)	4.5	4.5	4.6	4.6
Tear index (mN m^2/g)	6.1	6.0	5.9	5.9
Tear factor	62.2	61.2	60.2	60.2
Gurley Porosity (sec/100 ml)	35.63	38.63	41.41	44.52
Double fold (no.)	58	74	85	84
Smoothness (ml/min)	57	45	41	40

Enzymatic refining of recycled fiber pulps

7. RESULTS *AND DISCUSSION - Enzymatic refining of recycled fiber pulp*

7a. Evaluation of different enzymes

The enzymes were characterized for their cellulase (CMCase & FPase) and xylanase and other activities. Enzyme activities are shown in Table 44. There was much variation in enzyme activities of the first three refining enzymes and the highest enzyme activities were observed in enzyme 9 in all cases CMCase, FPase and xylanase.

7b. Studies on enzymatic refining of Mix office waste (Recycled fibre-1)

Studies on enzymatic refining were carried out on MOW using refining enzymes enzyme 8 and enzyme 9. Enzyme activity and dose of enzyme is one of the important parameter for the success of enzyme applications having greater influence on the yield & strength properties of the pulp. In order to avoid strength losses a careful optimization of enzyme doses and treatment condition is important. Further, there is a need to optimize the dose of the enzyme as it is very much varied with the fiber furnish. Reduction in pulp yield might be expected from severe hydrolytic activity of cellulases and hemicellulases. Published data indicate that losses can be restricted to acceptable levels provided enzyme dosage and reaction time are optimized.

Enzyme pretreatment of pulps

Enzyme treatment of pulp was carried out by adding enzyme 8 and 9 to the unrefined pulp after sufficient dilution & mixed properly by kneading mechanism. Enzymes in respective doses (0.03 %, 0.05% and 0.07%) were added and enzyme treatment of pulp was done using the conditions shown in Table 45. Control was run parallelly with maintaining all the conditions except the enzyme.

Refining

Both the untreated and the enzymatically treated pulps were refined in a laboratory PFI mill at different revolutions to obtain desired freeness values.

Evaluation of pulp properties

After refining, the pulps were characterized for drainability and strength properties.

Enzymatic refining with Enzyme 8 – Optimization of enzyme dose

Studies on enzymatic refining of mix office waste were conducted with enzyme 8 and enzyme 9.

Effect of enzyme 8 on Drainability & Strength properties

Results shown in table 46 & 47 indicated that enzyme 8 showed marginal improvement in drainability and significant improvement in strength properties of pulp compared with the properties of the untreated pulp. Maximum strength improvement was observed with 0.03% enzyme dose.

Effect of enzyme 9 on Drainability & strength properties

Results shown in table 48 & 49 indicated that enzyme 9 showed improvement in drainability and marginal improvement in strength properties of pulp.

7c. Studies on enzymatic refining of NDLKC (Recycled fibre-2)

Conventional Pulping vs Enzymatic Pulping of RCF (OCC & NDLKC)

Conventional alkaline pulping is the traditional process widely practiced in Indian recycled paper mills which involved pulping of the RCF with sodium hydroxide for fibre swelling and the process generates greater amounts of dissolved material i.e. anionic trash along with the higher pollution loads especially COD and colour in effluents. A comparison was therefore made to evaluate the efficacy of enzyme induced neutral pulping with the conventional alkaline process with an objective that if found comparable, then enzymes could become an alternate to conventional process for the processing of the unbleached varieties of NDLKC and OCC in paper mills. A further benefit excepted from enzyme use is in decreasing COD levels and an increase in the COD/BOD ratio in mill waste water. Enzymes partially hydrolyse cellulase fine fibrils and colloidal material to low molecular weight saccharides that are easily biodegraded in the waste water treatment plant. However it is utmost important to optimize the dose of the enzymes for treatment of the RCF.

Pulping and Enzyme treatment of recycled fibre

NDLKC and OCC teared into small pieces (5-10 cms) before processing to maintain the homogeneity of the raw material. High-density pulper (500 g capacity) was used for a batch of 250g of OD sample. Conventional pulping in the high density pulper was carried out as the conditions adopted in the mill employing Sodium hydroxide and surfactant. In case of enzymatic process enzyme and surfactant were added. The reference pulps were processed using the same conditions as the enzyme treated pulps prior to refining.

In the present study enzyme dose is optimized for NDLKC using the enzyme 8 with doses of 0.03 %, 0.05% and 0.075% and enzyme 9 0.7 in case of NDLKC. Enzyme 9 Enzymes in respective doses were added to the pulper and pulping was done using the conditions shown in Table 50. After pulping, the pulps were subjected to refining upto the desired freeness levels.

Effect of Enzyme dose on refining energy and fines content of pulp

Pulp yield of the enzyme treated pulps of NDLKC pulps were decreased as the dose of the enzyme was increased from 0.03% to 0.075%. Data is shown in Table 51. High dose of enzyme slightly affected the yield as it is slightly decreased when compared to the low doses. 20-40% savings in refining energy was observed as enzyme dose is increased from 0.03% to 0.075%. Data showed that varying in enzyme dose showed very slight increase in NDLKC pulps.

Fiber classification of the pulp - Bauer-McNett classification

Both enzyme treated and untreated pulps were subjected to Bauer-McNett apparatus. Results are shown in table 52. Both the enzymes increased the fibre fractions which are suitable for paper making and good formation .

Strength properties of pulp

The strength properties of enzyme treated pulps of NDLKC were shown in table 53. It was observed that the strength properties of the enzyme treated pulp with a dose of 0.03% are maximum and decreased as the enzyme dose increased from 0.03% to 0.075% when compared with the strength properties of control pulp.

All the above results of yield and strength properties of NDLKC pulps showed that 0.03% enzyme dose can be used as optimal dose for NDLKC processing and the same dose was used for further studies.

Comparison of chemical pulping vs enzymatic treatment of RCF

Pulp Yield & Drainability

The results of the enzyme induced pulping shown in Table 54 indicated improvement in fiber recovery with enzymes. Fibre recovery of enzyme processing of NDLKC is higher than that of the conventional alkaline process i.e. 94% i.e. 3% improvement in yield was observed.

Fines contents and drainability of Pulp

Fines analysis showed that the NDLKC enzyme treated pulps showed a little decrease in fines content when compared with the conventional pulps.

One of the major benefit of the enzyme application is the improvement in drainability of the pulp which affects the runnability of pulp on paper machine. Present results indicated that the drainability of enzyme treated NDLKC pulps is greater than that of chemically treated pulps i.e 6.2 gm/sec against 5.8 gm /sec of chemical pulp of NDLKC.

Strength properties

Table 55 shows the physical properties of laboratory hand sheets made from the refined pulps. Enzymatic hydrolysis did not affect the strength properties of the pulp adversely and are very much comparable with those from the conventionally treated pulps. The tensile Index of the enzyme treated pulps of NDLKC pulps is increased i.e. 40.1 Nm/g against 38.8 Nm/g of chemical pulp of NDLKC .

The tear index and burst indexes of the pulps showed no significant change. Porosity of the enzyme treated pulp is increased by 12% in case of NDLKC.

Environmental Impact

Enzymatic processes may yield a number of additional benefits. The feasibility of enzyme processes in neutral environment reduces the overall chemical requirements, which means less impact on the environment.

Effluents

Results shown in Table 56 indicated that enzymes added great benefits especially in environmental point of view. A huge decrease is observed in levels of COD, Colour and lignin of effluents of enzyme treated pulps of NDLKC verity. i.e 23.2 kg/tp, 2.7 kg/tp and 1.3 kg/tp against 56.8 kg/tp, 34.3 kg/tp and 5.8 kg/tp of chemically treated NDLKC pulp effluents respectively. The reducing sugar contents in enzyme treated pulp effluent showed that enzyme partially hydrolyzed the fine fibers and released low molecular weight sugar in effluents increasing the biodegradability of the effluent. Data shown in Table 56 clearly showed that enzymatic pulping process showed huge positive impact on environment by reducing the pollution loads of the effluent of paper mills.

Fibre morphology of pulps

Photo micrographs of chemical and enzymatically treated pulps of NDLKC were shown in fig 1. Better fibrillation was observed in case of enzyme treated pulps when compared with the chemically treated pulps increasing the bonding strength of the pulp.

7d. Studies on enzymatic refining of OCC (Recycled fibre-3)

Enzymatic refining of OCC was carried out using two enzymes enzyme 8 and enzyme 9. In the present study enzyme dose is optimized for both enzymes using the enzyme doses of 0.03%, 0.04% and 0.05%. Enzymes in respective doses were added to the pulper and pulping was done using the conditions shown in Table 57. After pulping, the pulps were subjected to refining upto the desired freeness levels.

Effect of Enzyme dose on fines content of pulp

Pulp yield of the OCC pulps treated with both enzymes were decreased as the dose of the enzyme was increased from 0.03% to 0.05% in OCC processing. Data is shown in Table 58 & 59. High dose of enzyme slightly affected the yield as it is slightly decreased when compared to the low doses. Savings in refining energy 10-20 % in case of enzyme 8 and 20% in case of enzyme 9 was observed as it is increased from 0.03% to 0.05% & 0.075% . Data showed that varying in enzyme dose does not show much effect on fines content of OCC pulps.

Strength properties of pulp

The strength properties of enzyme treated pulps of OCC were shown in table 60 & 61. It was observed that the strength properties of the both enzyme treated pulp with a dose of 0.03% are maximum and decreased as the enzyme dose increased from 0.03% to 0.05% in case of OCC when compared with the strength properties of control pulp.

All the above results of yield and strength properties of OCC pulps with both enzymes showed that 0.03% enzyme dose can be used as optimal dose for OCC processing and the same dose was used for further studies.

Comparison of chemical pulping vs enzymatic treatment of RCF

Pulp Yield & Drainability

The results of the enzyme induced pulping shown in Table 62 indicated improvement in fiber recovery with enzymes. Fibre recovery of enzyme processing of OCC is 96% which is higher than that of the conventional alkaline process i.e. 92% when compared with the alkaline process yield of chemical pulping.

Fines contents and drainability of Pulp

Fines analysis showed that the fines portion of the enzyme treated OCC pulp is significantly lower than the fines content of chemically treated pulp showing improvement in long fiber fraction when compared with the conventional pulps.

One of the major benefit of the enzyme application is the improvement in drainability of the pulp which affects the runnability of pulp on paper machine. Present results indicated that the drainability of enzyme treated OCC pulps is greater than that of chemically treated pulps i.e. 7.5 gm/sec against 7.05 gm/sec.

Strength properties

Table 63 shows the physical properties of laboratory hand sheets made from the refined pulps. Enzymatic hydrolysis did not affect the strength properties of the pulp adversely and are very much comparable with those from the conventionally treated pulps. The tensile and burst Index of the enzyme treated pulps of OCC pulps is increased i.e 46.84 Nm/g and 2.67 kPam^2/g against 44.34 Nm/g and 2.59 kPam^2/g of chemical pulp of OCC. Porosity of the enzyme treated pulps of OCC is increased by 12%.

Effluents

Results shown in Table 64 indicated that enzymes added great benefits especially in environmental point of view. A huge decrease is observed in levels of COD, Colour and lignin of effluents of enzyme treated pulps of OCC. i.e 12.9 kg/tp, 2.9 kg/tp and 1.0 kg/tp against 36.9 kg/tp, 23.3 kg/tp and 5.1 kg/tp of chemical OCC pulp effluents respectively. The reducing sugar contents in enzyme treated pulp effluent showed that enzyme partially hydrolyzed the fine fibers and released low molecular weight sugar in effluents increasing the biodegradability of the effluent. Data shown in Table 64 clearly showed that enzymatic pulping process showed huge positive impact on environment by reducing the pollution loads of the effluent of paper mills.

Fibre morphology of pulps

Photo micrographs of chemical and enzymatically treated pulps of OCC pulps were shown in fig 2. Better fibrillation was observed in case of enzyme treated pulps when compared with the chemically treated pulps increasing the bonding strength of the pulp.

7e. Observations and conclusion

(i) Enzymatic refining of Mix office waste

Results of enzyme treatment of mix office waste pulp before refining with enzyme 8 and enzyme 9 showed that –

- Improvement in strength properties along with marginal improvement in drianability of pulp was observed with enzyme 8.
- Improvement in drianability of pulp along with marginal improvement in strength properties was observed with enzyme 9.

(ii) Enzymatic refining of NDLKC and OCC varieties

Results of enzyme treatment of NDLKC and OCC varieties are promising and their use offers significant advantage over conventional chemical process. Enzyme treatment showed following benefits -

- It is possible to eliminate the use of caustic (2%) used by the industry by replacement of enzymes under optimized conditions.
- Savings in energy during refining of enzyme tretaed pulps is 10-40% with varying doses of enzymes
- Improvement in yield -2- 4%
- Improved strength properties in respect of tensile index by 3-5% , in burst index , 3.0 % and in proosity by 12 -20%.
- Reduction in fines content & better drainability.
- Reduction in pollution loads of colour (80-90 %) & COD (50-60%).

Addition of enzymes during pulping of unbleach varieties is expected to particularly benefit those mills with an aim to reduce energy consumption and pollution load of effluents and also the mill having the limited refining capacity.

The use of enzymes in the processing of these varieties is expected to be beneficial and may prove an alternate to conventional as chemical processing. Further research efforts in this direction should demonstrate the commercial utility of the enzyme in mills.

7f. Tables and figures

Table 44: Enzyme Activity of different enzymes used in present study

Enzymes	**Activity (IU/ml)**		
	CMCase	**FPase**	**Xylanase**
Enzyme 8	31.00	8.8	95.5
Enzyme 9	129	40	195

Table 45: Enzyme Pretreatment Conditions

Particulars	**Control**	**Enzyme treated pulp**
Enzyme dose, %	-	0.03, 0.05, 0.07
Surfactant dose,%	-	0.1%
Treatment Time, (min)	30	30
Temperature,^{0} C	50	50
Consistency, %	10.0	10
pH	8.0	8.0

Table 46: Effect of enzyme 8 on Drainability of mix office waste pulp

Parameters	Control	Enzyme 8 – dose of enzyme		
		0.03%	**0.05%**	**0.07%**
Drainability, gm/sec	9.3	9.45	9.55	9.75
Improvement in drainability, %	**-**	**1. 1**	**2.7**	**4.8**

Table 47: Effect of enzyme 8 on strength Properties of mix office waste pulp

Parameters	Control	Enzyme 1 – dose of enzyme		
		0.03%	**0.05%**	**0.07%**
Tear index, $mN.m^2/g$	5.6	5.9	5.2	5.0
Improvement in Tear **index**, %	-	5.**4**	-	-
Tensile index, N.m/g	36.8	45.4	45.2	39.0
Improvement in Tensile **index**, %	-	**23**	**22.8**	**6.0**
Burst index, $kPam^2/g$	2.2	2.5	2.3	2.2
Improvement in Burst **index**, %	-	**14**	4.**5**	-
Double fold	15	15	16	19
Improvement in Double fold , %	-	-	6.**7**	26.**7**
Porosity , ml/min.	608	617	694	594
App. Density , g/cm^3	0.63	0.63	0.68	0.64

Table 48: Effect of enzyme 9 on Drainability of mix office waste pulp

Parameters	**Control**	**Enzyme 9– dose of enzyme**		
		0.03%	**0.05%**	**0.07%**
Drainability, gm/sec	9.3	9.6	10.48	10.3
Improvement in drainability, %	**-**	**3.2**	**12**	**10.8**

Table 49: Effect of enzyme 8 on strength Properties of mix office waste pulp

Parameters	**Control**	**Enzyme 9 – dose of enzyme**		
		0.03%	**0.05%**	**0.07%**
Tear index, mN.m^2/g	5.6	5.3	5.3	5.6
Improvement in Tear index, %	-	-	-	**-**
Tensile index, N.m/g	36.8	38.1	37.5	36.8
Improvement in Tensile i**ndex**, %	-	3.4	2.**0**	**-**
Burst index, kPam^2/g	1.9	1.92	1.9	1.7
Improvement in Burst index, %	-	-	-	-
Double fold	15	14.5	14.3	17
Improvement in Double fold , %	-	-	-	**13**
Porosity , ml/min.	**6**08	768	600	598
App. Density , g/cm^3	0.63	0.64	0.64	0.70

Table 50: Process conditions used for pulping of NDLKC

Process	**Conventional pulping**	**Enzymatic pulping**
Pulping		
Time, min.	30	30
Consistency, %	10	10
Temp. °C	70	50
pH	10-11	7.0-7.5
Chemicals & Enzymes		
NaOH ,%	2.0	--
Enzyme ,%	--	0.03, 0.05% & (0.075)
Surfactant , %	--	0.03%

Table 51: Effect of enzymes on pulp yield and refining energy during treatment of NDLKC

Parameter		**Control**	**Enzyme 9**	**Enzyme -8 (dose)**		
			0.075%	**0.03%**	**0.05%**	**0.075%**
Yield, %		97.8	96.0	97.6	96.3	95.1
Refining energy consumed, KWH		0.05		0.05	0.04	0.03
Energy saving,%		**-**		**-**	**20**	**40**
Freeness ,CSF, ml		300		300	300	290
Fines study	Fines,%	27.48	23.24	26.5	27.0	27.4
	Fiber,%	72.52	76.76	73.5	73.0	72.6

Table 52: Effect of enzymes on Fiber Classification of pulp during treatment of NDLKC

Parameter	Control	Enzyme 9	Enzyme -8 (dose)		
		0.075%	0.03%	0.05%	0.075%
CSF	280	280	280	280	280
+ 30 fraction	28.9	28.95	24.4	31.2	30.9
+50 fraction	28.5	29.5	29.4	28.1	34.2
+100 fraction	12.4	12.4	13.4	14	13.55
+200 fraction	5.2	4.25	4.5	1.5	6.35
-200 fraction	25.0	24.9	28.3	25.2	15.0

Table 53: Effect of enzyme dose on strength properties of NDLKC pulp

Parameters	Control	Enzyme 9	Enzyme 8 (dose)		
		0.07%	0.03%	0.05%	0.075%
Freeness, CSF, ml	280	280	280	280	280
Tear index , $mN.m^2/g$	7.0	7.0	7.0	6.58	7.0
Tensile index, N.m/g	34.8	45.85	40.1	35.10	35.67
Burst index , $kPam^2/g$	2.0	2.4	2.20	2.0	2.04
Porosity , ml/min	505	450	430	430	391

Table 54: Pulp yield and drainability of chemical and enzyme treated NDLKC pulps

Parameter		**NDLKC pulp**	
		Chemical	**Enzymatic**
Yield, %		94	97
Drainability, gm/sec		5.8	6.2
Fines study	Fines, %	30.3	27.0
	Fiber, %	69.7	73.0

Table 55: Strength properties of chemical and enzyme treated NDLKC pulps

Parameter	**NDLKC pulp**	
	Chemical	**Enzymatic**
Tear index , $mN.m^2/g$	7.4	7.0
Tensile index, N.m/g	38.8	40.1
Improvement in Tensile index, %	-	3.2
Burst index, $kPam^2/g$	2.2	2.2
Improvement in burst index, %	-	-
Porosity , ml/min	376	430
Improvement in Porosity, %	-	12.6

Table 56: Characterization of effluents of chemical and enzyme treated NDLKC pulps

Parameters	**NDLKC pulp**	
	Chemical	**Enzymatic**
Colour, Kg/tp	34.3	2.7
Reduction in colour , %	**-**	**92**
Lignin, Kg/tp	5.8	1.3
Reduction in lignin , %	**-**	**78**
COD, Kg/tp	56.8	23.2
Reduction in COD , %	**-**	**59**
Reducing Sugars, Kg/tp	1.37	3.58
Total solids, Kg/tp	48.2	20.4
Reduction in solids , %	**-**	**58**

Table 57: Process conditions used for pulping of OCC

Process	Conventional pulping	Enzymatic pulping
Pulping		
Time, min.	30	30
Consistency, %	10	10
Temp. °C	70	50
pH	10-11	7.0-7.5
Chemicals & Enzymes		
NaOH ,%	2.0	--
Enzyme 8,%	--	0.03, 0.04 , 0.05%
Enzyme 9,%		0.03, 0.04 , 0.05%
Surfactant , %	--	0.03%

Table 58: Effect of enzyme 8 on pulp yield and refining energy during treatment of OCC

Parameter		Control	Enzyme dose		
			0.03%	**0.04%**	**0.05%**
Yield, %		96.5	96.0	95.5	94.5
Refining energy consumed, KWH		0.10	0.09	0.08	0.08
Savings in Energy,%		**-**	**10**	**20**	**20**
Freeness ,CSF, ml		300	305	300	290
Fines study	Fines, %	29.3	26.3	26.5	26.4
	Fiber,%	70.7	73.7	73.5	73.6

Table 59: Effect of enzyme 9 on pulp yield and refining energy during enzyme treatment of OCC

Parameter		**Control**	**Enzyme 9 (dose)**		
			0.03%	**0.04%**	**0.05%**
Yield, %		96.5	96.0	95.5	94.0
Refining energy consumed, KWH		0.10	0.08	0.08	0.08
Savings in Energy,%		**-**	**20**	**20**	**20**
Freeness ,CSF, ml		300	305	290	295
Fines study	Fines, %	29.3	32.47	32.30	32.10
	Fiber,%	70.7	67.53	67.70	67.90

Table 60: Effect of enzyme 8 on strength properties of OCC pulp

Parameters	**Control**	**Enzyme 8 (dose)**		
		0.03%	**0.04%**	**0.05%**
Freeness ,CSF,ml	300	305	300	290
Tear index , $mN.m^2/g$	8.0	8.41	8.23	8.07
Tensile index, N.m/g	45.4	46.84	45.64	44.58
Burst index , $kPam^2/g$	2.9	2.9	2.88	2.75
App. Density , g/cm^3	0.6	0.58	0.60	0.62
Porosity , ml/min	307	338	390	441

Table 61: Effect of enzyme 9 on strength properties of OCC pulp

Parameters	**Control**	**Enzyme 9 (dose)**		
		0.03%	**0.04%**	**0.05%**
Freeness ,CSF,ml	300	305	290	295
Tear index , mN.m^2/g	8.0	7.7	7.65	7.5
Tensile index, N.m/g	45.4	44.59	43.6	43.61
Burst index , kPam^2/g	2.9	2.7	2.7	2.6
App. Density , g/cm^3	0.6	0.61	0.62	0.64
Porosity , ml/min	307	422	420	413

Table 62: Pulp yield and drainability of chemical and enzyme treated OCC pulps

Parameter		**OCC pulp**	
		Chemical	**Enzymatic**
Yield, %		92	96
Drainability, gm/sec		7.05	7.5
Fines study	Fines, %	36.48	26.3
	Fiber, %	63.6	73.7

Table 63: Strength properties of chemical and enzyme treated OCC pulps

Parameter	**OCC pulp**	
	Chemical	**Enzymatic**
Tear index , $mN.m^2/g$	8.5	8.41
Tensile index, N.m/g	44.34	46.84
Improvement in Tensile index, %	-	5.3
Burst index, $kPam^2/g$	2.59	2.67
Improvement in burst index, %	-	3.0
Porosity , ml/min	331	413
Improvement in Porosity, %	-	12.6

Table 64: Characterization of effluents of chemical and enzyme treated OCC

Parameters	**OCC pulp effluents**	
	Chemical	**Enzymatic**
Colour, Kg/tp	23.3	2.9
Reduction in colour , %	**-**	**88**
Lignin, Kg/tp	5.1	1.0
Reduction in lignin , %	**-**	**83**
COD, Kg/tp	36.9	12.9
Reduction in COD , %	**-**	**65**
Reducing Sugars, Kg/tp	1.2	2.3
Total solids, Kg/tp	42.5	25.9
Reduction in solids , %	**-**	**39**

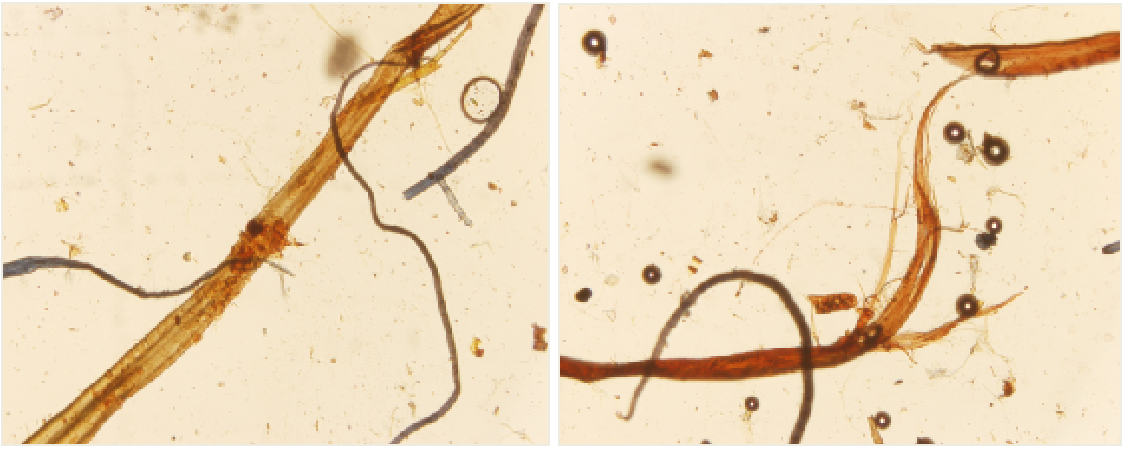

Fig 1 Microphotographs of chemical treated and enzyme treated NDLKC pulps

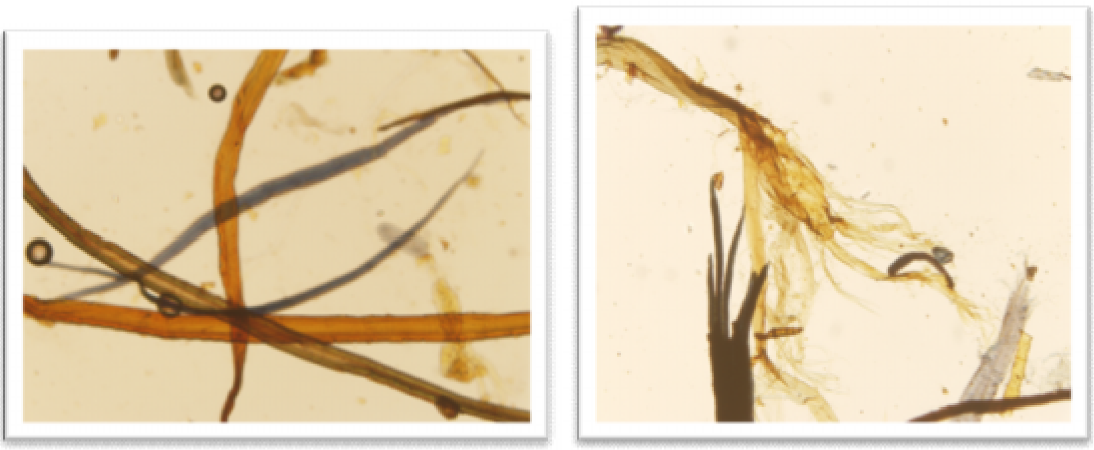

Fig 2 Microphotographs of chemical treated and enzyme treated OCC pulps

Plant trial with screened enzyme

8. RESULTS AND DISCUSSION - *Plant trial with screened enzyme*

Plant scale trial was conducted in a wood based paper mill situated in western part of India. The mill was producing writing printing papers using Hardwood, Softwood and BCTMP pulp in their pulp furnish. The trial was conducted using two screened enzymes based on the study conducted at TCIRD. Control data were collected for 10 days plant running in normal conditions.

8a. Trial with Enzyme-3

Plant scale trial was taken with the use of Enzyme-3 for a period of 12 days. The trial was taken with enzyme dose level of 50-100 g/TP. During the trial improvement of 2°SR in freeness of the pulp was observed. During the trial with enzyme-3 machine DDR was bypassed. Detailed results of refining energy reduction observed during plant scale trials taken with Enzyme-3 are given in Table 65.

In the wet- end area FPAR and FPR were improved with the use of Enzyme-3 during plant trial. Slight improvement in freeness of the Head box and Machine chest pulp was also observed. Detailed results of wet end properties observed during plant scale trial taken with Enzyme-3 are given in Table 66.

Breaking length of the paper was improved in both the directions i.e. MD and CD from 2961 m to 3720 m, and 1567 to 1813 m, respectively. Tear factor of the paper was reduced marginally during the trial run with Enzyme-3. Detailed results of physical strength properties of the paper obtained during plant scale trials taken with Enzyme-3 are given in Table 67.

8b. Trial with Enzyme-4

Plant scale trial was taken with the use of Enzyme-4 for the period of five days. The trial was taken with enzyme dose level of 60-100 g/TP. During the trial improvement of 5 units of °SR in freeness of the pulp was observed. During the trial with enzyme-4 a reduction of ~20% in refining energy was observed. In addition the machine DDR was also bypassed during the trial run. Detailed results of refining energy reduction observed during plant scale trials taken with Enzyme-4 are given in Table 68.

In the wet- end area FPAR and FPR were improved with the use of Enzyme-4 during plant trial. Significant improvement in freeness (5 units of °SR) in the Head box and Machine chest pulp was observed. Detailed results of wet end properties obtained during plant scale trial taken with Enzyme-4 are given in Table 69.

2.0% increase in the ash percentage of the paper was obtained during trial run with Enzyme-4. Breaking length of the paper was improved in both the directions i.e. MD and CD from 2961 to 3475 m, and 1567 to 1798 m respectively. Tear factor of the paper was reduced marginally during the trial run with Enzyme-4. Detailed results of physical strength properties of the paper obtained during plant scale trials taken with Enzyme-4 are given in Table 70.

8c. Observations and conclusion

Trial with Enzyme-3

- 50-100 g/TP dose of enzyme was found suitable.
- Improvement of 2 units in ^oSR of the refined pulp was observed.
- During the trial with enzyme-3 machine DDR was bypassed.
- FPAR and FPR were improved with the use of Enzyme-3.
- Slight improvement in freeness of the Head box and Machine chest pulp was observed.
- Breaking length of the paper was improved in both the directions i.e. MD and CD.
- Tear factor of the paper was reduced marginally.

Trial with Enzyme-4

- 60-100 g/TP dose of enzyme was found suitable.
- Improvement of 5 units in ^oSR of the refined pulp was observed.
- Reduction of ~20% in refining energy was observed .
- During the trial with enzyme-4 machine DDR was bypassed.
- FPAR and FPR were improved with the use of Enzyme-4.
- Significant improvement in freeness of the Head box and Machine chest pulp was observed.
- Breaking length of the paper was improved in both the directions i.e. MD and CD.
- Tear factor of the paper was reduced marginally.

8d. Tables and figures

Table 65: Results of the plant scale trials taken with Enzyme-3 *(Effect on refining energy reduction)*

Particulars	**BLANK DATA**			**Trial with Enzyme-3**		
	Min	Max	Avg.	Min	Max	Avg.
Enzyme dose (g/TP)	--	---	--	50	100	75
Refining						
^{0}SR (SW)	--	--	--	26	34	29
^{0}SR (SW+HW)	18	21	19	--	--	--
^{0}SR (HW)	--	--	--	22	25	23
^{0}SR (Head Box)	22	27	24	24	29	26
Refiner loads						
Pulp DDR (kWh)	75	110	101	100	108	105
Stock DDR (kWh)	98	108	103	97	106	103
M/C DDR (AMP)	31	71	61	--	--	--

Table 66: Results of the plant scale trials taken with enzyme-3 *(Effect on wet end properties)*

Particulars		**BLANK DATA**			**Trial with Enzyme 3**		
		Min	Max	Avg.	Min	Max	Avg.
Enzyme dose (g/TP)		--	---	--	50	100	75
Wet end properties							
Soft wood	Cy (%)	--	--	--	3.7	4.2	3.9
	°SR	--	--	--	26	34	29
Hardwood + Softwood	Cy (%)	4.5	4.9	4.7	--	--	--
	°SR	18	21	19	--	--	--
BCTMP	Cy (%)	3.71	4.0	3.8	3.7	4.1	3.9
	°SR	24	25	25	22	24	23
Hardwood	Cy (%)	--	--	--	4.6	5.0	4.8
	°SR	--	--	--	22	25	23
M/C Chest	Cy (%)	3.6	3.8	3.7	3.9	4.4	4.1
	°SR	20	24	22	23	25	24
Head Box	Cy (%)	0.44	0.59	0.55	0.51	0.61	0.55
	°SR	22	27	24	24	29	26
Back Water	Cy (%)	0.10	0.15	0.12	0.09	0.14	0.11
FPR	(%)	72.1	80.8	77.1	75.7	82.8	79.4
FPAR	(%)	41.4	50.1	46.9	44.9	53.0	49.5

Table 67: Results of the plant scale trials taken with enzyme-3 *(Effect on paper properties)*

Particulars		**BLANK DATA**			**Trial with Enzyme-3**		
		Min	Max	Avg.	Min	Max	Avg.
Grammage *(g/m²)*		73.3	77.6	75.5	74.4	77.3	75.9
Thickness (μm)		105	111	109	107	112	109
Bulk (cc/g)		1.38	1.49	1.44	1.40	1.47	1.43
Smoothness *(ml/min)*	Top	194	303	258	254	328	285
	Wire	181	320	257	256	348	296
G. porosity *(s/100ml)*		6	10	8	6	10	8
Ash (%)		11.7	15.6	14.2	12.1	15.8	14.2
Tear Factor	MD	49	65	57	50	58	54
	CD	54	70	62	56	64	59
Br. Length (m)	MD	2481	3386	2961	3277	4416	3720
	CD	1387	1768	1567	1539	2175	1813
Burst Factor		9.7	13.6	11.3	10.0	14.6	11.8
Wax Pick (no.)	Top	14	14	14	14	14	14
	Wire	14	16	16	14	16	16

Table 68: Results of the plant scale trials taken with enzyme-4 *(Effect on Refining energy reduction)*

Particulars	BLANK DATA			Trial with Enzyme 4		
	Min	Max	Avg.	Min	Max	Avg.
Enzyme dose (g/TP)	--	--	--	60	100	80
Refining Degree SR						
^{0}SR (SW)	--	--	--	--	--	--
^{0}SR (SW+HW)	18	21	19	27	43	34
^{0}SR (HW)	--	--	--	23	26	25
^{0}SR (Head Box)	22	27	24	27	33	29
Paper Properties						
Tear Factor (MD)	49	65	57	47	54	50
Tear Factor (CD)	54	70	62	52	61	56
Breaking length (MD), m	2481	3386	2961	3190	3953	3475
Breaking length (CD), m	1387	1768	1567	1712	1921	1798
Burst Factor	9.7	13.6	11.3	9.11	13.46	10.83
Ash (%)	11.7	15.6	14.2	14.20	18.00	16.26
Refiner loads						
Pulp DDR (kWh)	75	110	101	100	110	106
Stock DDR (kWh)	98	108	103	62	96	83
M/C DDR (AMP)	31	71	61	--	--	--

Table 69: Results of the plant scale trials taken with enzyme-4 *(Effect on wet end properties)*

Particulars		**BLANK DATA**			**Trial with Enzyme-4**		
		Min	Max	Avg.	Min	Max	Avg.
Enzyme dose (g/TP)		--	--	--	60	100	80
Wet end properties							
Soft wood	Cy (%)	--	--	--	--	--	--
	°SR	--	--	--	--	--	--
Hardwood + Softwood	Cy (%)	4.5	4.9	4.7	3.80	4.30	3.96
	°SR	18	21	19	27	43	34
BCTMP	Cy (%)	3.71	4.0	3.8	3.77	4.03	3.90
	°SR	24	25	25	24	26	25
Hardwood	Cy (%)	--	--	--	3.96	4.61	4.22
	°SR	--	--	--	23	26	24
M/C Chest	Cy (%)	3.6	3.8	3.7	4.07	4.32	4.18
	°SR	20	24	22	24	30	26
Head Box	Cy (%)	0.44	0.59	0.55	0.47	0.58	0.55
	°SR	22	27	24	27	33	29
Back Water	Cy (%)	0.10	0.15	0.12	0.09	0.13	0.11
FPR	(%)	72.1	80.8	77.1	77.2	83.2	79.4
FPAR	(%)	41.4	50.1	46.9	47.1	53.1	48.6

Table 70: Results of the plant scale trials taken with enzyme-4 *(Effect on paper properties)*

Particulars		**BLANK DATA**			**Trial with Enzyme-4**		
		Min	Max	Avg.	Min	Max	Avg.
Grammage *(g/m²)*		73.3	77.6	75.5	73.8	77.9	75.7
Thickness (μm)		105	111	109	105	113	109
Bulk (cc/g)		1.38	1.49	1.44	1.40	1.46	1.44
Smoothness *(ml/min)*	Top	194	303	258	234	359	295
	Wire	181	320	257	240	365	309
G. porosity *(s/100ml)*		6	10	8	6	9	7
Ash (%)		11.7	15.6	14.2	14.2	18.0	16.2
Tear Factor	MD	49	65	57	47	53	50
	CD	54	70	62	52	61	56
Br. Length (m)	MD	2481	3386	2961	3190	3953	3475
	CD	1387	1768	1567	1712	1921	1798
Burst Factor		9.7	13.6	11.3	9.11	12.29	10.85
Wax Pick (no.)	Top	14	14	14	13	14	13
	Wire	14	16	16	14	14	14

SEM, XRD analysis *of* *Enzyme treated and* untreated wood pulp fibers

9. RESULTS AND DISCUSSION - SEM, XRD analysis of enzyme treated and *untreated wood pulp fibers* (Experiments carried out at TCIRD)

9a. SEM analysis

Study on mixed hardwood pulp

SEM micrographs of enzyme treated and untreated mixed hardwood pulps were taken to analyze the effect of enzymes on the surface of the pulp fiber. There was no fibrillation as well as delamination of cell wall/ cell wall collapse was observed in the unrefined untreated fibers of mixed hardwood pulp fibers. SEM micrographs of mixed hardwood pulp fibers are shown in the Figure 3.

With the mechanical refining of the pulp marginal fibrillation on the surface of fiber as well as delamination of cell wall/ cell wall collapse was observed. SEM micrographs are shown in the Figure 4.

With the addition of enzymes there was marginal delamination of cell wall as well as cell wall collapse was observed even in unrefined pulps. SEM micrographs of Enzyme-3 and Enzyme-4 treated mixed hardwood pulp fiber are shown in the Figure 5 and Figure 6, respectively.

Enzymatic refining (Treatment of pulp with enzyme afterwards mechanical refining) of mixed hardwood pulp was resulted to improved fibrillation on the surface of pulp fibers, delamination of cell wall and collapsed cell wall. SEM micrographs of Enzyme-3 and Enzyme-4 treated refined mixed hardwood pulp fibers are shown in the Figure 7 and Figure 8, respectively.

Study on softwood pulp

SEM micrographs of enzyme treated and untreated softwood pulps were taken to analyze the effect of enzymes on the surface of the pulp fiber. There was no fibrillation as well as delamination of cell wall/ cell wall collapse was observed in the unrefined untreated fibers of softwood pulp fibers. SEM micrographs of softwood pulp fibers are shown in the Figure 9.

With the mechanical refining of the pulp marginal fibrillation on the surface of fiber as well as delamination of cell wall/ cell wall collapse was observed. SEM micrographs of refined softwood pulp fibers without enzymatic treatment are shown in the Figure 10.

With the addition of enzymes there was marginal delamination of cell wall as well as cell wall collapse was observed even in unrefined softwood pulp fibers. SEM micrograph of Enzyme-3 and Enzyme-4 treated softwood pulp fiber are shown in the Figure 11 and Figure 12, respectively.

Enzymatic refining (Treatment of pulp with enzyme afterwards mechanical refining) of softwood pulp was resulted in improved fibrillation on the surface of pulp fibers, delamination of cell wall and collapsed cell wall. SEM micrograph of Enzyme-3 and Enzyme-4 treated refined softwood pulp fibers are shown in the Figure 13 and Figure 14, respectively.

9b. XRD analysis

Study on mixed hardwood pulp

To access the effect of enzymes on the crystallinity of cellulosic fiber, XRD analysis of control as well as enzyme treated mixed hardwood pulps was performed. Results showed that treatment of pulp with Enzyme-3 decreases the amorphous part of the cellulose, whereas Enzyme-4 decreases the crystalline part of the cellulose. Detailed results are given in the Table 71 and Figures 15, 16 and 17.

Study on Softwood pulp

To access the effect of enzymes on the crystallinity of cellulosic fiber, XRD analysis of control as well as enzyme treated softwood pulps was also performed. Results showed that treatment of pulp with Enzyme-3 decreases the amorphous part of the cellulose, whereas Enzyme-4 decreases the crystalline part of the cellulose. Detailed results are given in the Table 72 and Figures 18, 19 and 20.

9c. Tables and Figures

Table 71: Crystallinity index of untreated and enzyme treated mixed hardwood pulps

Particulars	Crystallinity Index (%)
Mixed hardwood pulp (Untreated, Unrefined)	93.75
Enzyme-3 treated mixed hardwood unrefined pulp	96.42
Enzyme-4 treated mixed hardwood unrefined pulp	89.74

Table 72: Crystallinity index of untreated and enzyme treated softwood pulps

Particulars	Crystallinity Index (%)
Softwood pulp (Untreated, Unrefined)	92.59
Enzyme-3 treated softwood unrefined pulp	96.05
Enzyme-4 treated softwood unrefined pulp	88.79

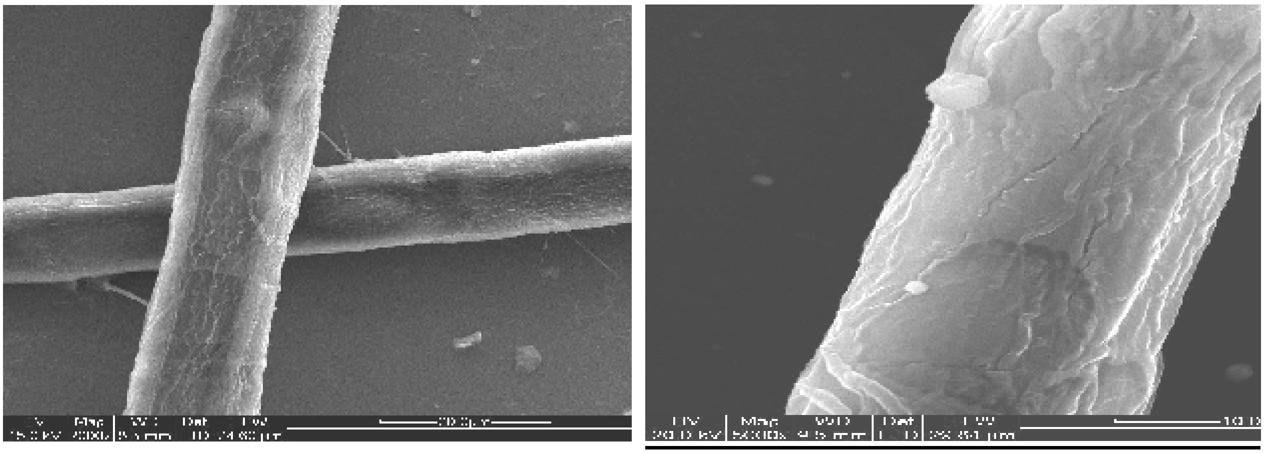

Figure 3: SEM micrographs of Mixed hardwood pulp fibers (Control, Unrefined)

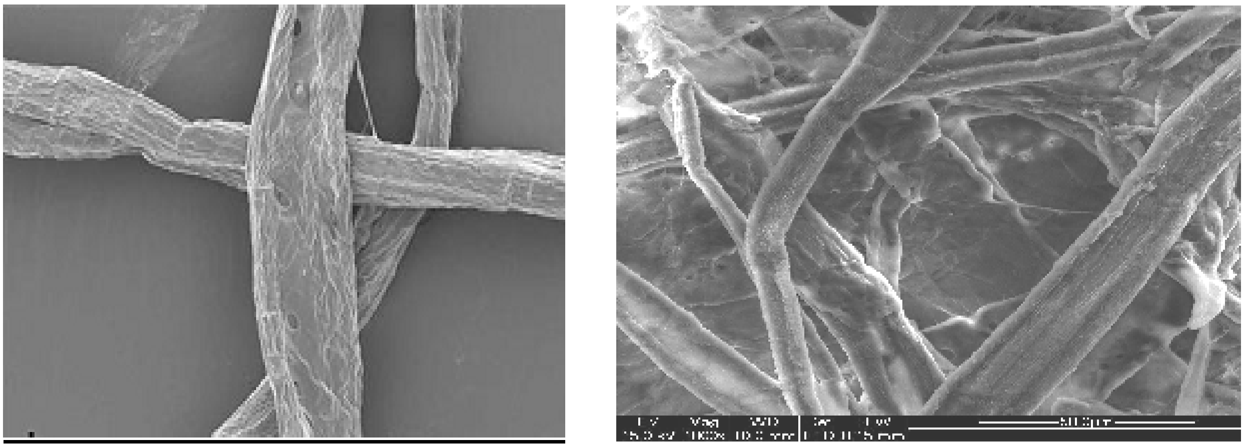

Figure 4: SEM micrographs of mixed hardwood pulp fibers (Control, Refined)

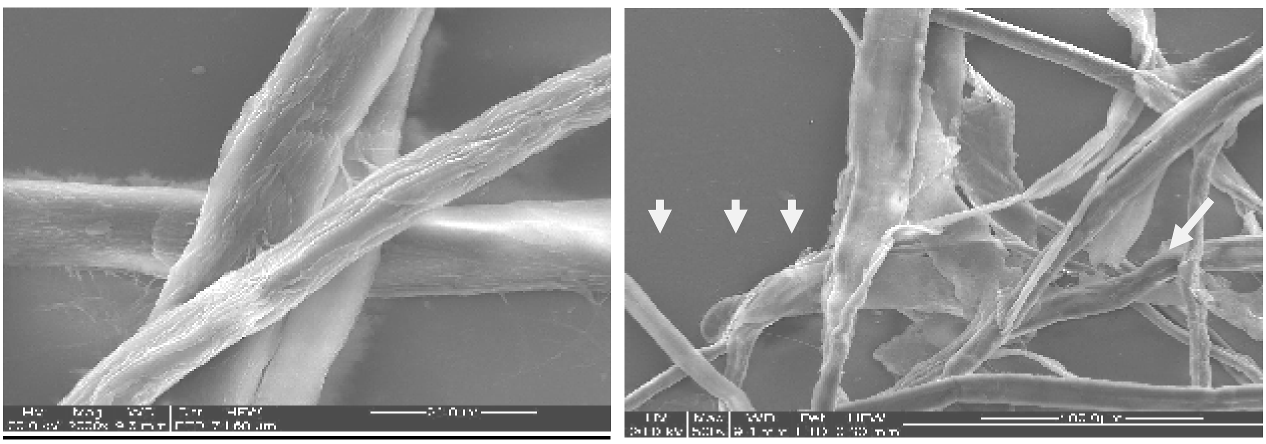

Figure 5: SEM micrographs of Enzyme-3 treated mixed hardwood pulp fiber (Unrefined)

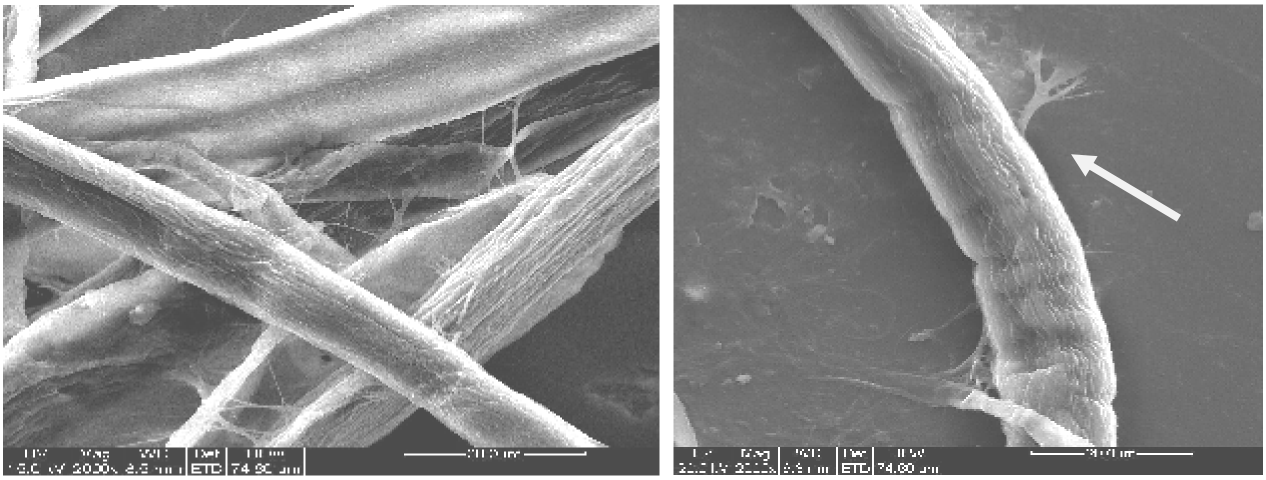

Figure 6: SEM micrographs of Enzyme-4 treated mixed hardwood pulp fiber (Unrefined)

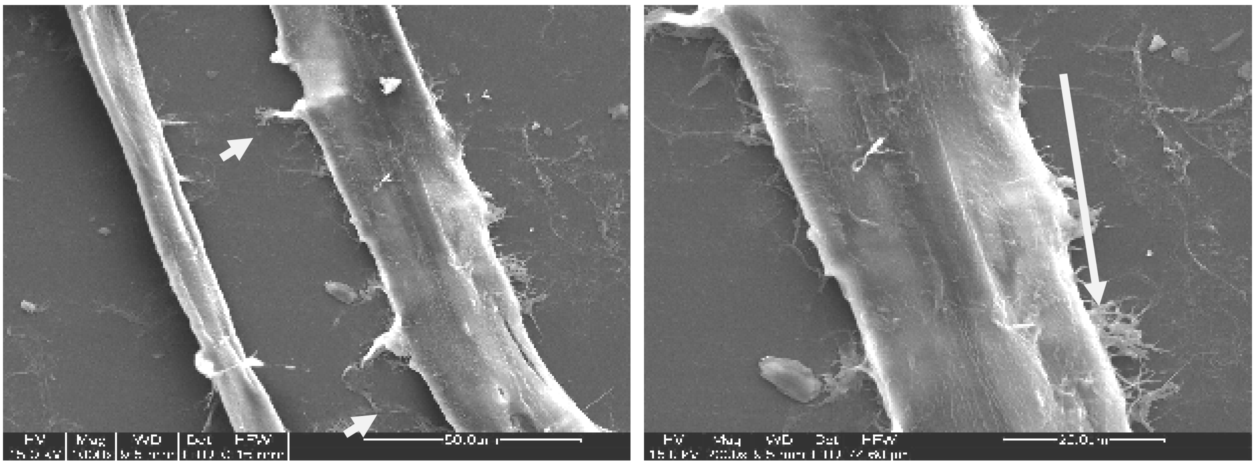

Figure 7: SEM micrographs of Enzyme-3 treated mixed hardwood pulp fiber (refined)

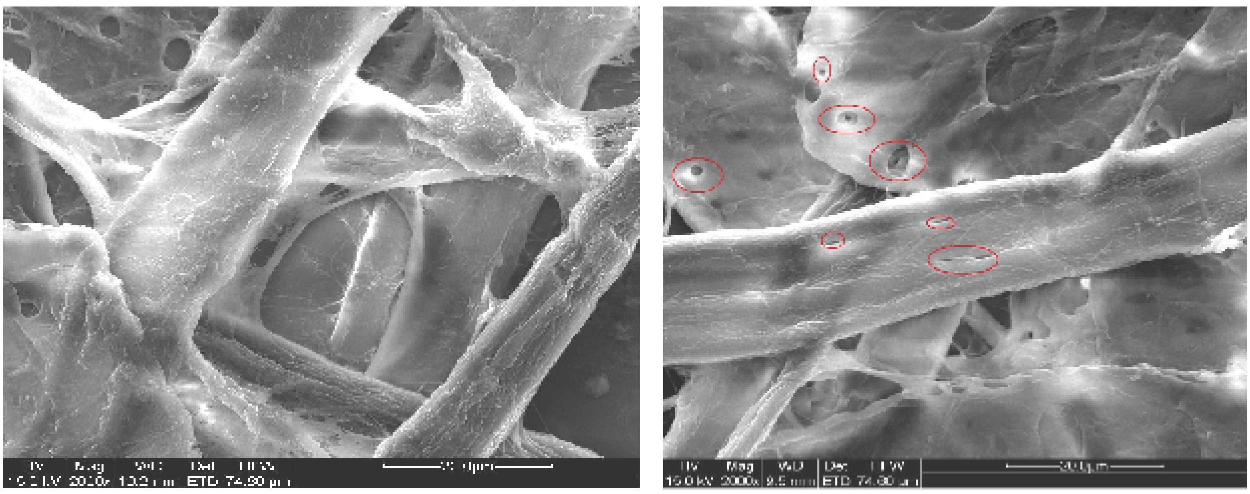

Figure 8: SEM micrographs of Enzyme-4 treated mixed hardwood pulp fiber (refined)

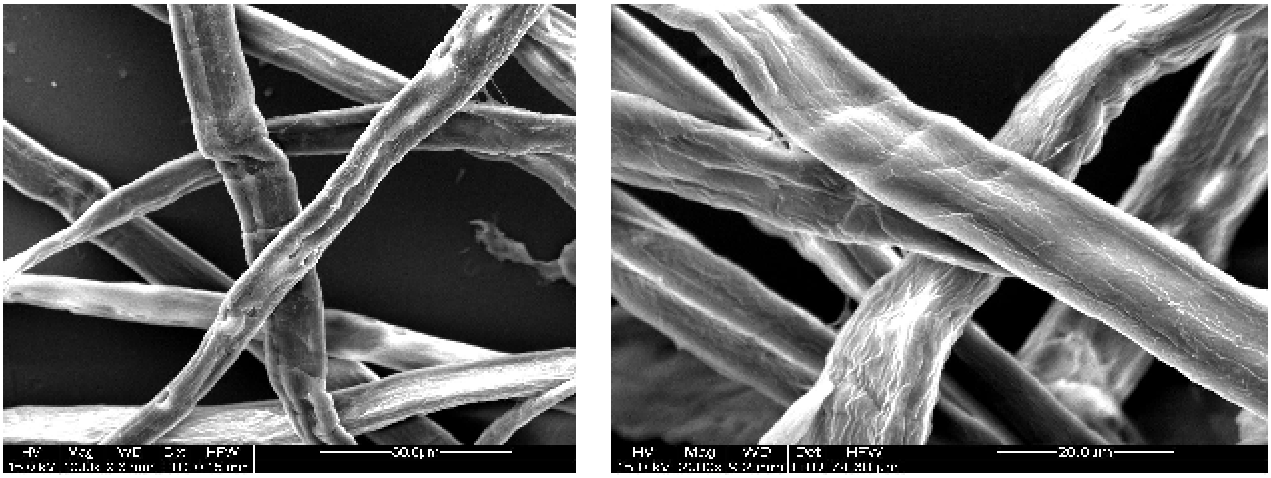

Figure 9: SEM micrographs of softwood pulp fiber (Control, Unrefined)

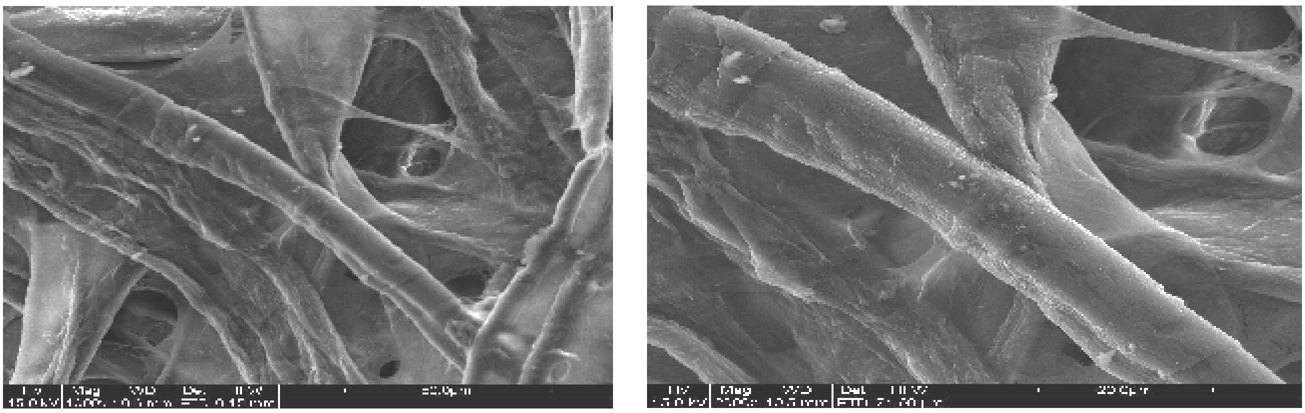

Figure 10: SEM micrographs of softwood pulp fiber (Control, Refined)

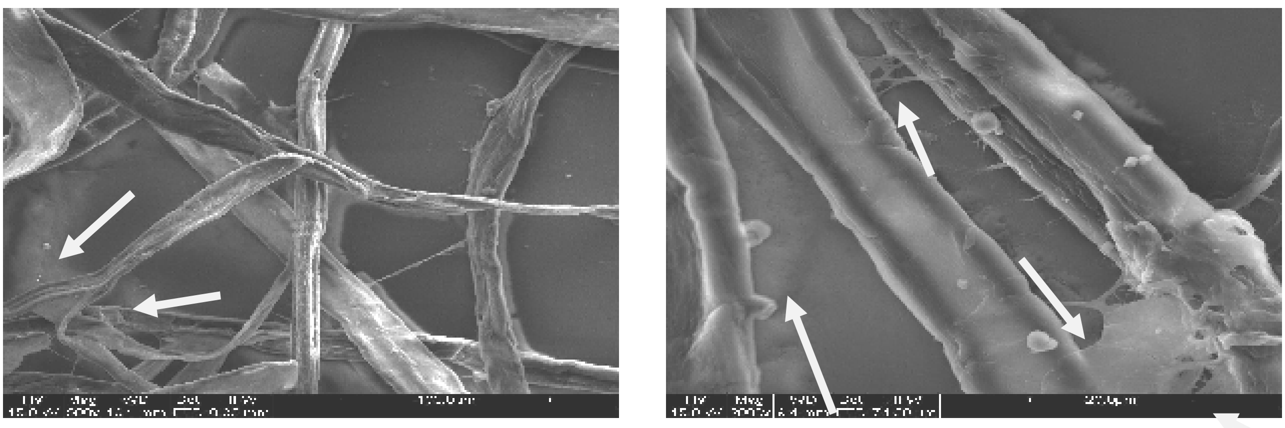

Figure 11: SEM micrographs of Enzyme-3 treated softwood pulp fiber (Unrefined)

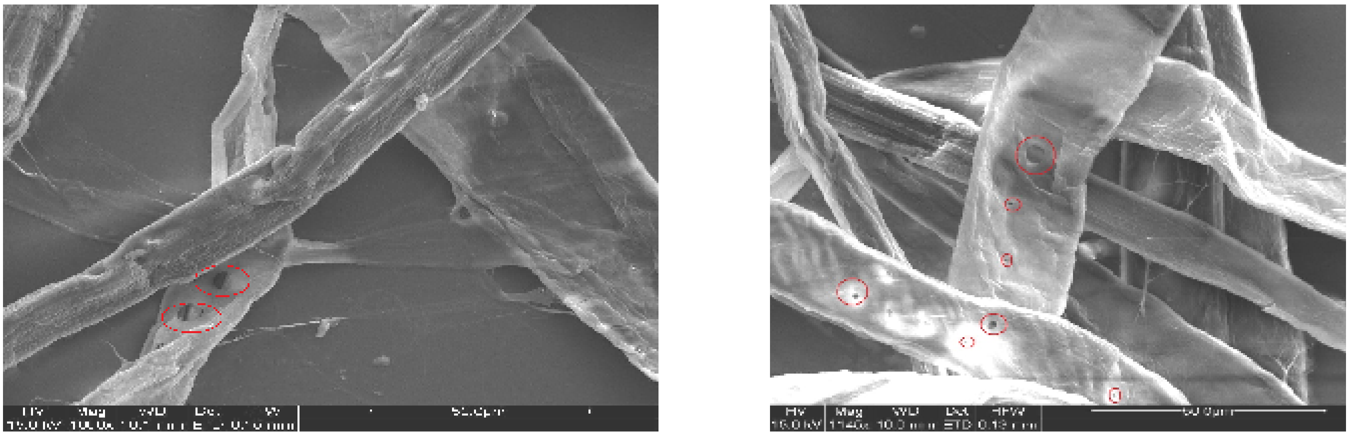

Figure 12: SEM micrographs of Enzyme-4 treated softwood pulp fiber (Unrefined)

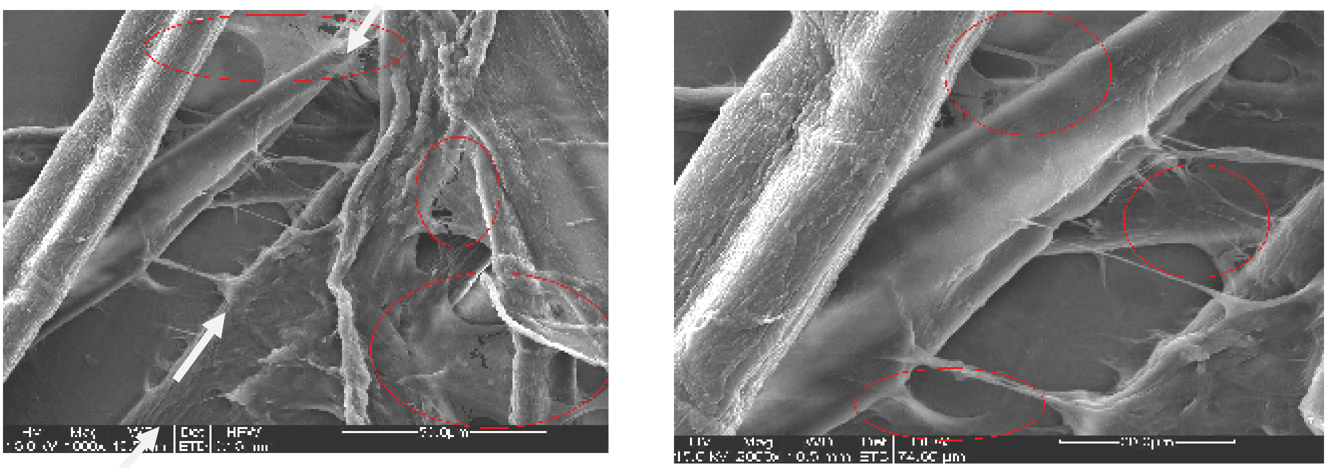

Figure 13: SEM micrographs of Enzyme-3 treated softwood pulp fiber (Refined)

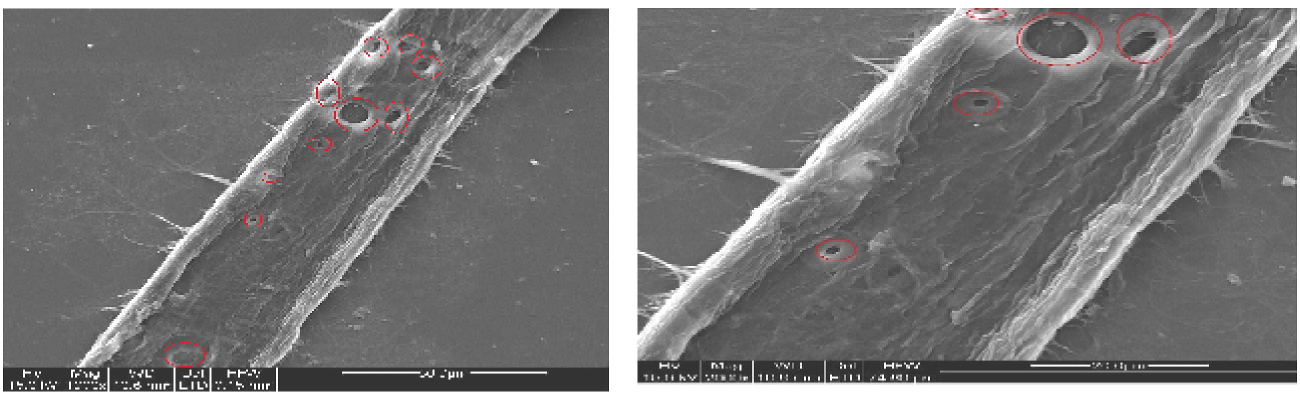

Figure 14: SEM micrographs of Enzyme-4 treated softwood pulp fiber (Refined)

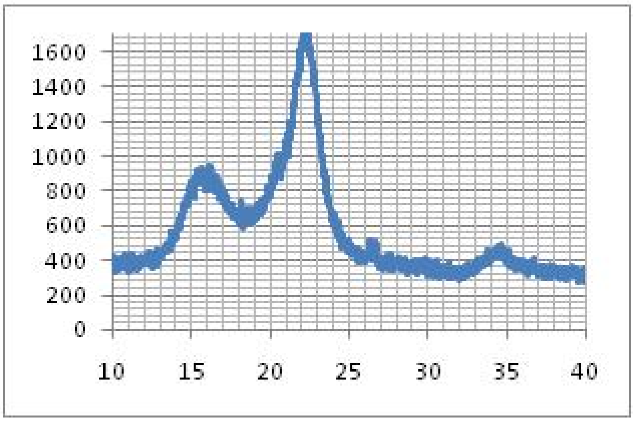

Figure 15: XRD diffractogram of untreated unrefined mixed hardwood pulp

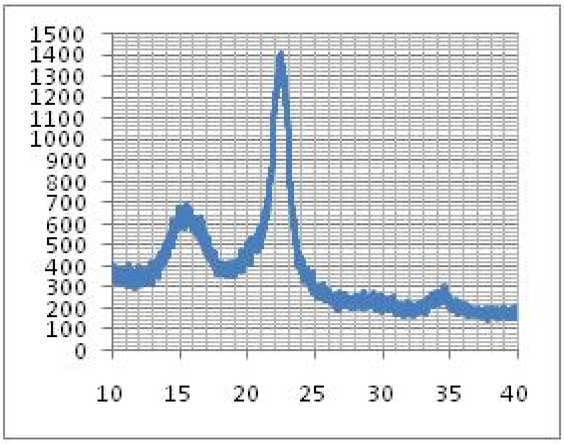

Figure 16: XRD diffractogram of Enzyme-3 treated unrefined mixed hardwood pulp

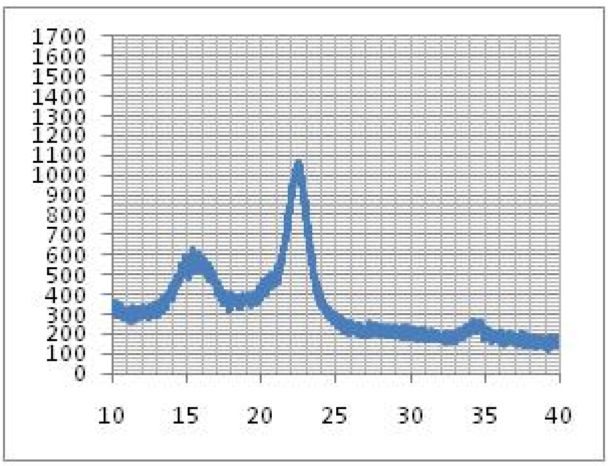

Figure 17: XRD diffractogram of Enzyme-4 treated unrefined mixed hardwood pulp

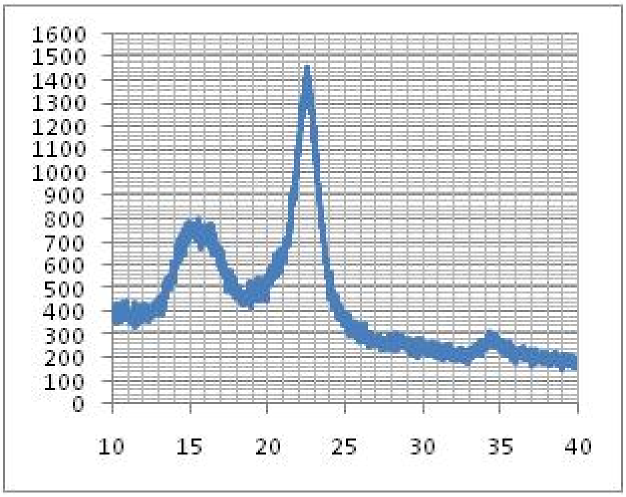

Figure 18: XRD diffractogram of untreated unrefined softwood pulp

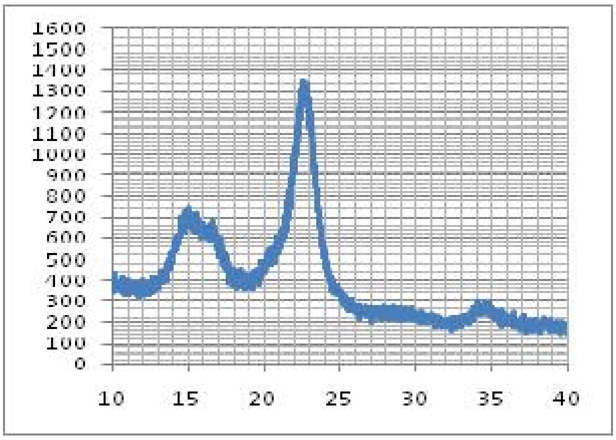

Figure 19: XRD diffractogram of Enzyme-3 treated unrefined softwood pulp

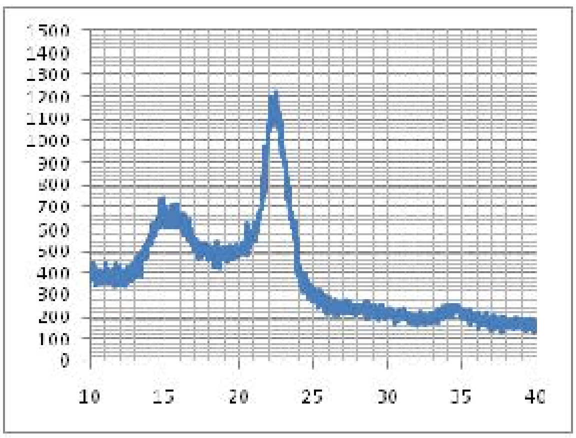

Figure 20: XRD diffractogram of Enzyme-4 treated unrefined softwood pulp

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11. ANNEXURE

Copy of the manuscript published in proceedings of 2nd Paper+ Conference-2012:

Improved Papermaking of Recycle Fibers through Fiber Modification with Enzymes

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Dr. Jain holds the Masters in Chemistry with PhD in pulp & paper. He has more than 30 years of research & development experience of working in industry and research institutes in the area of Chemical Recovery, Environmental management & Biotechnology. Having worked as Director of Kumarappa National Handmade Paper Institute (KNHPI), Jaipur during the year 2007-09, now working as a senior scientist at Central Pulp & Paper Research Institute (CPPRI).

He has been nominated as an expert member of various committees of Dept. of Biotechnology, Dept. of Science & Technology and CSIR. He was awarded with N. N. Mohan Memorial Award for the best research paper in the year 1984 and has more than 100 research papers and eight patents to his credit. Widely travelled to USA, Australia, Sweden, China, Thailand, Singapore under various programmes connected to UNIDO & International conferences.

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ABSTRACT

Recycle fiber (RCF) has emerged as one of the important raw material for paper manufacturing in India contributing to about 5 million tons of (47%) papers, paperboards and newsprints production.

In the present study, enzymatic treatment of the RCF played an important role while improving the quality of paper with improvement of strength properties, productivity, with an additional improved environmental status.

Cellulase and Hemicellulase are the principal enzymes used for fiber modification. Present study covers findings of the enzymatic treatment of the recycled fibers viz. New Double Lined Kraft Cuttings (ÑDLKC) and old corrugated cartoons (OCC) with the identified enzyme containing the mixture of cellulases & hemicellulase in a definite proportion. The process conditions of the enzyme treatment of these two raw materials were optimized in respect of doses of enzyme under the process conditions prevalent in the mill in respect of temperature & time. It has been possible to eliminate the use of caustic used during pulping. Microscopic studies of the enzyme treated fiber have also been covered which indicated that the fibrillation and bonding increases among the fibers due to fibre modification after enzymatic treatment of RCF.

INTRODUCTION

Indian Paper Industry produces more than 10 million tons of paper, paper board &newsprint against global average production of 390 million tons. Industry utilizes diversified raw materials viz. wood , agro residue based and recycled waste paper producing nearly 10 million tons of paper, paper board & newsprint of the total production.

Recycled fibre (RCF) contributes to around 47% of the total production thereby emerging as one of the major raw material throughout all the developed and developing countries which are the largest producers and consumers of paper. Worldwide, approximately over 100 million tons of recycle fiber is presently collected and reused for paper manufacture. Recycle paper is used not only reduce overall cost, but also protect the environment. Therefore, recycle fiber based product must complete with their virgin equivalents and must perform well during conversion while meeting the same end product specifications. (1,2,3)

Literature survey revealed that around 50% of paper produced globally is manufactured using the recycled fibre (2). Generally, it could be stated that pulp properties of recycled fibers decline during the reprocessing thus there is a limitation in paper recycling. Deterioration of recycled fiber properties is mainly due to the irreversible changes in fiber structure caused by repeated chemical, mechanical treatments and drying. However, in these steps, originally content of fines increases in the recycled pulp. The main problem with fines is due to their high relative surface area and dewatering rate is lower compared to primary pulp. (2)

The original properties of pulps, especially of chemical pulps, change according to the number of utilization cycles applied in the commercial recycling of paper and paperboard. Pulp drainability and paper strength diminish as a result of repeated refining mechanical actions, thus increasing cuts and generating more fines in the recycled pulps.(4)

The application of biotechnology into papermaking processes are also expanded form small scaled batch process to large mill trials, or from simple fibre modification to speciality paper manufactures. (5)

Production of release and high density papers requires high amount of refining energy. The use of cellulase / hemicellulase enzymes prior to refining is proposed to modify the fiber surface and improve the fibrillation in the following mechanical treatment. Thus, less energy is needed to obtain a target refining level. In order to maintain good strength properties the use of cellulase containing enzyme mixtures requires thorough optimization. (6)

The enzymatic refining of recycle fibers can increase the paper strength, expand the specific surface area of the fibers, improve the interfiber bonding, develop the freeness level, and decrease the fiber fines. (7)

Polymeric additives are widely used in paper manufacturing to improve paper quality, which allows the use of low quality fibres (like fibres from waste papers) to produce paper with good properties. Usually, the use of recycled fibres in papermaking results in a decrease in paper strength properties. In general, it is assumed that the poor properties of recycled paper are mainly due to structural changes in the fibre caused by drying. Moreover, the paper machine runnability is deteriorated by the bad drainability of recycled pulp. Cellulases and hemicellulases are a class of additives, which may be used with the objective of to modify the fibre properties in order to improve or to change paper properties. The use of selected enzymes, cellulases and hemicellulases to modify or restore fibre properties in the recycling paper industry proved to a useful method. (7, 8)

Recycled fibres can be upgraded through treatments with cellulases. In fact, the enzymes modify the interfacial properties of fibres, increasing the water affinity, which in turn change the technical properties of pulp and paper, such as drainability and strength (9). According to other investigations, fibres treatment with different cellulases or hemicellulases lead to energy savings in beating and refining by reducing the beating time to achieve the same beating level, without affecting the pulp strength properties (5,10)

The present paper explores the possibility of modification of the recycled fibres using identified/cocktail of enzymes specific for a given quality of recycled paper i.e. NDLKC & OCC for improved quality of paper compared with that over conventional process being employed in Indian paper industry.

MATERIALS AND METHODS

Paper Furnish

Recovered paper, new double lined Kraft cuttings (NDLKC) and old corrugated cartoons (OCC) collected from paper mills were used in the present study.

Enzymes and their enzyme activity

Commercial enzyme used in the present study was procured from an international reputed company. The enzyme was characterized for its Cellulase (CMCase & FPase) and xylanase activities. All the enzyme activities were determined by measuring the released reducing sugar content. CMCase and FPase activities were analysed using Ghosh method (1987) (11) and xylanase activity measured by Bailey's method (1992) (12) and the activities were 31 IU/ml and 9.0 IU/ml 96.0 IU/ml respectively.

Pulping and Enzyme treatment of NDLKC and OCC

NDLKC and OCC teared into small pieces (5-10 cms) before processing to maintain the homogeneity of the raw material. High-density pulper (500 g capacity) was used for a batch of 250g of OD sample. Conventional pulping in the high density pulper was carried out as the conditions adopted in the mill employing Sodium hydroxide and surfactant. In case of enzymatic process enzyme and surfactant were added. The process conditions used for pulping during conventional and enzymatic process are shown in Table 1. The reference pulps were processed using the same conditions as the enzyme treated pulps prior to refining.

Table-1: Process conditions used for pulping of NDLKC & OCC

Process	Conventional pulping	Enzymatic pulping
Pulping		
Time, min.	30	30
Consistency, %	10	10
Temp. ?C	70	50
pH 10-11	7.0-7.5	
Chemicals & Enzymes		
NaOH ,%	2.0	---
Enzyme ,%	---	0.03, 0.04 , 0.05% & (.075)
Surfactant , %	---	0.03%

Refining of the Pulp

Refining of all the untreated chemical and enzyme treated pulps was done in laboratory PFI mill.

Hand sheet preparation and Testing

Hand sheets formation was done by ISO method 5364. Fines content was processed through Britt's dynamic jar using -200 screen.The freeness of pulp fibers was measured using a CSF tester according to ISO 5267-2. Drainability was determined by using DFR-04 (Dynamic drainage, freeness and retention tester). Fines analysis was performed with a 200 mesh Screen, according to TAPPI test method T261cm-90. Handsheets were evaluated for their strength according to standard ISO test methods.

Effluent Characterization

The effluents obtained from enzymatic and chemical pulping processes were analyzed in terms of chemical oxygen demand (COD) and lignin. Reducing sugar content in effluent was measured by DNS as per the procedure followed by Miller, 1959 (13). The determination of COD, Color and lignin were carried out accordingly to APHA (American Public Health Association) Standard Methods.

Microscopic studies of untreated and treated pulp

Photomicrography of the untreated, chemical treated and enzyme treated pulps after refining was done with image analysis to see the effect of enzymes on fiber structure, swelling and fibrillation.

RESULTS AND DISCUSSION

Conventional Pulping vs Enzymatic Pulping of RCF (OCC & NDLKC)

Conventional alkaline pulping is the traditional process widely practiced in Indian recycled paper mills which involved pulping of the RCF with sodium hydroxide for fibre swelling and the process generates greater amounts of dissolved material i.e. anionic trash along with the higher pollution loads especially COD and colour in effluents. A comparison was therefore made to evaluate the efficacy of enzyme induced neutral pulping with the conventional alkaline process with an objective that if found comparable, then enzymes could become an alternate to conventional process for the processing of the unbleach varieties of NDLKC and OCC in paper mills. A further benefit excepted from enzyme use is in decreasing COD levels and an increase in the COD/ BOD ratio in mill waste water. Enzymes partially hydrolyse cellulase fine fibrils and colloidal material to low molecular weight saccharides that are easily biodegraded in the waste water treatment plant. However it is utmost important to optimize the dose of the enzymes for treatment of the RCF.

Optimization of enzyme dose

Dose of enzyme is one of the important parameter for the success of enzyme applications having greater influence on the yield & strength properties of the pulp. In order to avoid strength losses a careful optimization of enzyme doses and treatment condition is important. Further, there is a need to optimize the dose of the enzyme as it is very much varied with the fiber furnish. Reduction in pulp yield might be expected from severe hydrolytic activity of cellulases and hemicellulases. Published data indicate that losses can be restricted to acceptable levels provided enzyme dosage and reaction time are optimized.

In the present study enzyme dose is optimized for both NDLKC and OCC using the enzyme doses of 0.03 %, 0.05% and 0.075% in case of NDLKC and 0.03%, 0.04% and 0.05% in case of OCC. Enzymes in respective doses were added to the pulper and pulping was done using the conditions shown in Table 1. After pulping, the pulps were subjected to refining upto the desired freeness levels.

Effect of Enzyme dose on fines content of pulp.

Pulp yield of the enzyme treated pulps of both NDLKC and OCC pulps were decreased as the dose of the enzyme was increased from 0.03% to 0.075% in case of NDLKC and from 0.03% to 0.05% in OCC processing. Data is shown in Table 2 & 3. High dose of enzyme slightly affected the yield as it is slightly decreased when compared to the low doses.

Table-2: Effect of enzyme dose on pulp yield and fines content during enzyme treatment of OCC

Parameter		Enzyme dose		
		0.03%	0.04%	0.05%
Yield, %		96.0	95.5	94.5
PFI revolutions		3000	3000	3000
Freeness ,CSF, ml		305	300	290
Fines study	Fines, %	26.3	26.5	26.4
	Fiber,%	73.7	73.5	73.6

Table-3: Effect of enzyme dose on pulp yield and fines content during enzyme treatment of NDLKC

Parameter		Enzyme dose		
		0.03%	0.05%	0.075%
Yield, %		97.6	96.3	95.1
PFI revolutions		3000	3000	3000
Freeness ,CSF, ml		300	300	290
Fines study	Fines, %	26.5	27.0	27.4
	Fiber,%	73.5	73.0	72.6

The fines content after enzymatic treatment are reported in Table 2 & 3. Data showed that varying in enzyme dose does not show much effect on fines content of OCC and a very slight increase in NDLKC pulps. Bajpai et al., (2006) described that the Cellulase in the enzyme mixture mainly attaches to fines instead of long fiber protecting the long fibers from severe hydrolysis. (9)

Strength properties of pulp

The strength properties of enzyme treated pulps of both NDLKC and OCC were shown in table 4 & 5. It was observed that the strength properties of the enzyme treated pulp with a dose of 0.03% are maximum and decreased as the enzyme dose increased from 0.03% to 0.05% in case of OCC and 0.03% to 0.075% in case of NDLKC pulp processing.

Table- 4: Effect of enzyme dose on strength properties of OCC pulp

Parameters	Enzyme dose		
	0.03%	0.04%	0.05%
Freeness ,CSF,ml	305	300	290
Tear index , mN.m²/g	8.41	8.23	8.07
Tensile index, N.m/g	46.84	45.64	44.58
Burst index , kPam²/g	2.9	2.88	2.75
App. Density , g/cm³	0.58	0.60	0.62
Porosity , ml/min	338	390	441

Table- 5: Effect of enzyme dose on strength properties of NDLKC pulp

Parameters	Enzyme dose		
	0.03%	0.05%	0.075%
Freeness, CSF, ml	280	280	280
Tear index , mN.m²/g	7.0	6.58	7.0
Tensile index, N.m/g	40.1	35.10	35.67
Burst index , kPam²/g	2.20	2.0	2.04
Porosity , ml/min	430	430	391

All the above results of yield and strength properties of both NDLKC and OCC showed that 0.03% enzyme dose can be used as optimal dose for OCC and NDLKC processing and the same dose was used for further studies.

Comparison of chemical pulping vs enzymatic treatment of RCF

Pulp Yield & Drainability

The results of the enzyme induced pulping shown in Table 6 indicated improvement in fiber recovery with enzymes. Fibre recovery of enzyme processing of OCC is 96% which is higher than that of the conventional alkaline process i.e 92% and the same trend is observed in case of NDLKC, 3% improvement in yield was observed when compared with the alkaline process yield i.e 97.0% against 94.0% of chemical pulping.

Table-6: Pulp yield and drainability of chemical and enzyme treated OCC and NDLKC pulps

Parameter		NDLKC pulp		OCC pulp	
		Chemical	Enzymatic	Chemical	Enzymatic
Yield, %		94	97	92	96
Drainability, gm/sec		5.8	6.2	7.05	7.5
Fines study	Fines, %	30.3	27.0	36.48	26.3
	Fiber, %	69.7	73.0	63.6	73.7

Fines contents and drain ability of Pulp

Fines analysis showed that the fines portion of the enzyme treated OCC pulp is significantly lower than the fines content of chemically treated pulp showing improvement in long fiber fraction whereas the NDLKC enzyme treated pulps showed a little decrease in fines content when compared with the conventional pulps. One of the major benefit of the enzyme application is the improvement in drainability of the pulp which affects the runnability of pulp on paper machine. Present results indicated that the drainability of both enzyme treated NDLKC and OCC pulps is greater than that of chemically treated pulps i.e 6.2 gm/sec against 5.8 gm /sec of chemical pulp of NDLKC and 7.5 gm/sec against 7.05 gm/sec of OCC pulps.

Strength properties

Table 7 shows the physical properties of laboratory hand sheets made from the refined pulps. Enzymatic hydrolysis did not affect the strength properties of the pulp adversely and are very much comparable with those from the conventionally treated pulps. The tensile Index of the enzyme treated pulps of both NDLKC and OCC pulps is increased i.e 40.1 Nm/g against 38.8 Nm/g of chemical pulp of NDLKC and 46.84 Nm/g against 44.34 Nm/g of chemical pulp of OCC.

Table-7: Strength properties of chemical and enzyme treated OCC and NDLKC pulps

Parameter	NDLKC pulp		OCC pulp	
	Chemical	Enzymatic	Chemical	Enzymatic
Tear index , $mN.m^2/g$	7.4	7.0	8.5	8.41
Tensile index, N.m/g	38.8	40.1	44.34	46.84
Improvement in Tensile index, %	-	3.2	-	5.3
Burst index, $kPam^2/g$	2.2	2.2	2.59	2.67
Improvement in burst index, %	-	-	-	3.0
Porosity , ml/min 376	430	331	413	
Improvement in Porosity, %	-	12.6	-	20.0

The tear index and burst indexes of the both variety pulps showed no significant change as the burst index of the OCC pulp is slightly improved was observed when compared with the strength of the chemically treated pulps. Porosity of the enzyme treated pulps of both verities is increased by 12% in case of NDLKC and improvement in OCC is 20% .

Environmental Impact

Enzymatic processes may yield a number of additional benefits. The feasibility of enzyme processes in neutral environment reduces the overall chemical requirements, which means less impact on the environment.

Effluents

Results shown in Table 8 indicated that enzymes added great benefits especially in environmental point of view. A huge decrease is observed in levels of COD, Colour and lignin of effluents of enzyme treated pulps of both OCC and NDLKC verities. i.e 12.9 kg/tp, 2.9 kg/tp and 1.0 kg/tp against 36.9 kg/tp, 23.3 kg/tp and 5.1 kg/tp of chemical OCC pulp effluents where as 23.2 kg/tp, 2.7 kg/tp and 1.3 kg/tp against 56.8 kg/tp, 34.3 kg/tp and 5.8 kg/tp of chemically treated NDLKC pulp effluents respectively. The reducing sugar contents in enzyme treated pulp effluent of both verities showed that enzyme partially hydrolyzed the fine fibers and released low molecular weight sugar in effluents increasing the biodegradability of the effluent. Data shown in Table 8 clearly showed that enzymatic pulping process showed huge positive impact on environment by reducing the pollution loads of the effluent of paper mills.

Table 8: Characterization of effluents of chemical and enzyme treated OCC and NDLKC pulps

Parametrs	NDLKC pulp		OCC pulp	
	Chemical	Enzymatic	Chemical	Enzymatic
Colour, Kg/tp	34.3	2.7	23.3	2.9
Reduction in colour , %	-	92	-	88
Lignin, Kg/tp	5.8	1.3	5.1	1.0
Reduction in lignin , %	-	78	-	83
COD, Kg/tp	56.8	23.2	36.9	12.9
Reduction in COD , %	-	59	-	65
Reducing Sugars, Kg/tp	1.37	3.58	1.2	2.3
Total solids, Kg/tp	48.2	20.4	42.5	25.9
Reduction in solids , %	-	58	-	39

Fibre morphology of pulps

Photo micrographs of chemical and enzymatically treated pulps of both verities were shown in fig 1 and 2. Better fibrillation was observed in case of enzyme treated pulps when compared with the chemically treated pulps increasing the bonding strength of the pulp. Long fibers when collide with enzymes, main effect is brushing of long fibers i.e damage the bonds on exposed cellulose chains. This is a partial depolymerization of cellulose chains on the fiber surface that causes a weakening but not a complete cutting of external micro fibers, allowing the less intake of refining energy or more easily defibrillated during refining. This effect is clearly seen in enzyme treated pulps of OCC i.e fig 1.

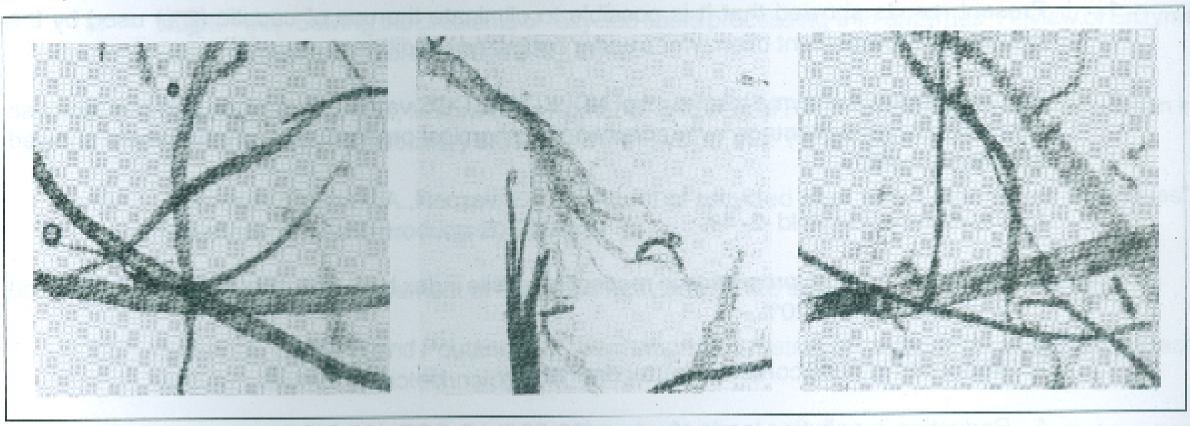

Fig.1: Microphotographs of chemical treated and enzyme treated OCC pulps

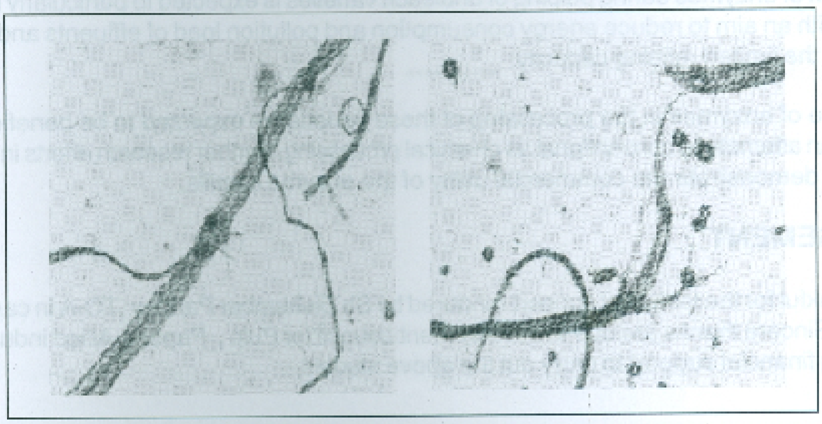

Fig.2: Microphotographs of chemical treated and enzyme treated NDLKC pulps

Enzyme Action on Pulps

Based on the results obtained during the studies , it is observed that the combination of cellulase & hemicellulase enzymes mainly functions by the partial hydrolysis of the fines & fibrils is due to the action of cellulase enzyme (cocktail of CMCase, FPase & Cellobiohydrolases), perforation and brushing of long fibres is by xylanase enzyme.

1. As a result of partial hydrolysis of fibrils & fines and the colloidal particals in the paper machine,the mixture of enzymes results –Improved drainage at paper machine thereby improved productivity by way of increasing the machine speed
2. Reduction in the fines content in the pulp helps to improve the strength properties due to the increase in percentage of long fibres. Reason beind is that the cellulase enzyme in the enzyme mixture preferrred to be attached to the fines instead of attaching to the long fibers from drastic hydrolysis conditions which results into other added benefits in respect of pulp yield.
3. The other important action due to the presence of xylanase in the enzyme mixture is the fiber perforation which results in hydration of fibre thereby promoting delamination and internal fibrillation, ultimately improving fibre properties.(9)

CONCLUSION

1. Present results showed that It is possible to eliminate the use of caustic (2%) used by the industry by replacement of enzymes under optimized conditions

2. Present results of enzyme treatment of NDLKC and OCC varieties are promising and their use offers significant advantage over conventional chemical process. Enzyme treatment showed following benefits –

    1. Improvement in yield -2- 4%

    2. Improved strength properties in respect of tensile index by 3-5% , in burst index , 3.0 % and in proosity by 12 -20%.

    3. Reduction in fines content & better drainability

    4. Reduction in pollution loads of colour (80-90 %) & COD (50-60%).

3. Addition of enzymes during pulping of unbleach varieties is expected to particularly benefit those mills with an aim to reduce energy consumption and pollution load of effluents and also the mill having the limited refining capacity.

4. The use of enzymes in the processing of these varieties is expected to be beneficial and may prove an alternate to conventional as chemical processing. Further research efforts in this direction should demonstrate the commercial utility of the enzyme in mills.

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