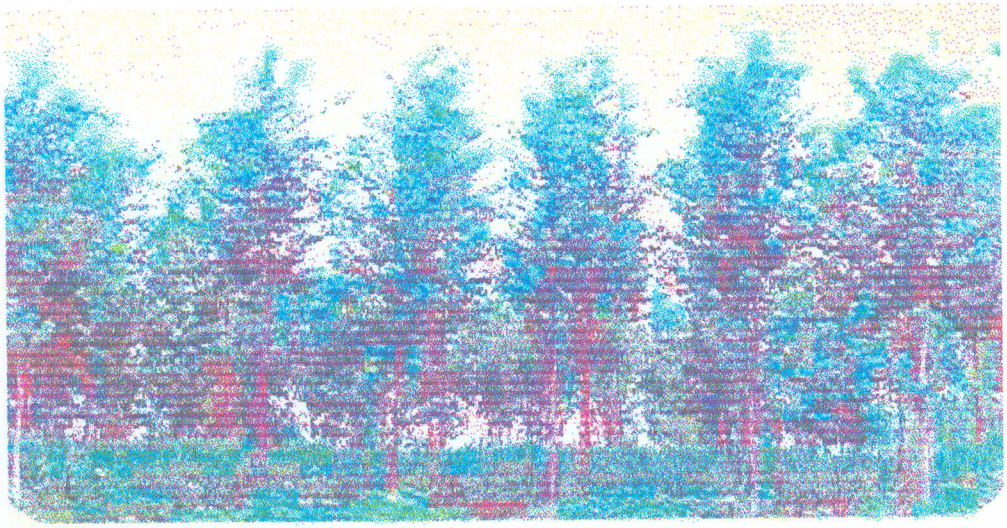
IMPA-FRI PROJECT

HIGH YIELD PULPING AND BLEACHING OF PLANTATION GROWN WOOD SPECIES AND BAGASSE USING ALKALINE PEROXIDE MECHANICAL PULPING/BLEACHING (APMP) PROCESS



CONSOLIDATED REPORT

ON

ALKALINE PEROXIDE MECHANICAL PULP FROM *POPULUS DELTOIDES*

CELLULOSE AND PAPER DIVISION
FOREST RESEARCH INSTITUTE
P.O. NEW FOREST
DEHRA-DUN

High yield pulping and bleaching of plantation grown wood species and bagasse using Alkaline Peroxide Mechanical Pulping/bleaching (APMP) Process

Project : IPMA- FRI Project

Objective : The objective of the project are:

i) To produce high yield pulps from plantation grown wood species viz., Poplars, Eucalypts(*Eucalyptus grandis, Eucalyptus globulus), Casuraina equisetifolia, Ailenthus excelus Anthocephalus indicus* and bagasse in 75-85 % yield and 70-85 % brightness by using Alkaline Peroxide Mechanical Pulping/bleaching (APMP) Process.

ii) To study techno- economic feasibility of mini high yield pulping plant based on APMP process

Period: 4 Years (The project was started after the MOU was signed between IPMA and FRI on 19-1- 99

Results of work done on *Populus deltoides*

APMP Process : A brief review

Alkaline Peroxide Mechanical Pulping (APMP) advances the technology of producing Bleached Chemi-Thermo- Mechanical pulp (BCTMP) into the 90's. APMP uses advanced chip per treatment techniques to pulp and bleach wood prior to refining. The combination of pulping and bleaching in a single unit operation reduces the capital cost for an installation by decreasing the amount of equipment to produce pulp. There are also significant operating cost advantages through reduced electrical power consumption, which is achieved by refining the pulp under alkaline conditions To better understand the APMP process and the savings potential this technology offers, let's proceed step-by step through the process.

Chip Impregnation- "Better chip impregnation means better pulp" APMP consists of multistage of chip impregnation. The first stage consists of pre-steaming the wood chips to soften the wood and prevent damage when chips are compressed. The first stage of compression squeezes out some of the water-soluble materials that naturally occur in wood. The removal of water-soluble material enhances the responsiveness of the chemical treatments. Mixing chemicals to the pulp can be difficult; adding chemicals to chips is even harder. The reason for this is that the structure of the wood is often not conductive to complete and uniform penetration of chemical across the chip so, to increase the bleach response wood is impregnated with solution of chemical stabilizers and chelants to tie-up the organic and inorganic inhibitors to peroxide bleaching. This impregnation stage can consist of various inorganic or organic acids sequesterant or a weak alkaline peroxide liquor, i.e. whatever chemicals are necessary to wash the contaminants from a specific wood, that could affect the performance of subsequent stage. To efficiently bleach wood, the combination of perfect impregnation and maximum squeezing capability is critical.

Bleaching - The wood thoroughly compressed to squeezed or washed out much of the spent chemical and reaction products. what's left is a wood chip ideally suited for bleaching. Bleaching was done with peroxide and caustic soda.

The Refining process- Chips from two stages bleaching are almost pulp so the energy required to finish the process is reduced compared to more conventional techniques.

Advantages - APMP offer two significant advantages to a site with limited wood and power supply. APMP can be installed economically with relatively small pieces of equipment. APMP technology uses no sulphur compounds thus the treatment can be easier. It is environmental friendly process giving much higher productivity of pulp suitable for fine papers.

Visit to Indian paper mills producing high yield pulps

It will be seen from the brief review given above on APMP process that it is highly specialised technical process based on sophisticated specially designed equipment capable of producing high yield high brightness pulp economically suitable for production of fine papers.
Therefore before undergoing the exercise of drawing up the specifications for procuring laboratory equipment's suitable for APMP process, it was considered desirable to look at the conventional facilities being employed by Indian pulp and paper mills for production of high yield pulps.
There are three big mills in India that are producing high yield pulps for manufacture of newsprint:viz, Tamilnadu Newsprint and Paper mills, Hindustan Newsprint Mills and Mysore Paper mills.

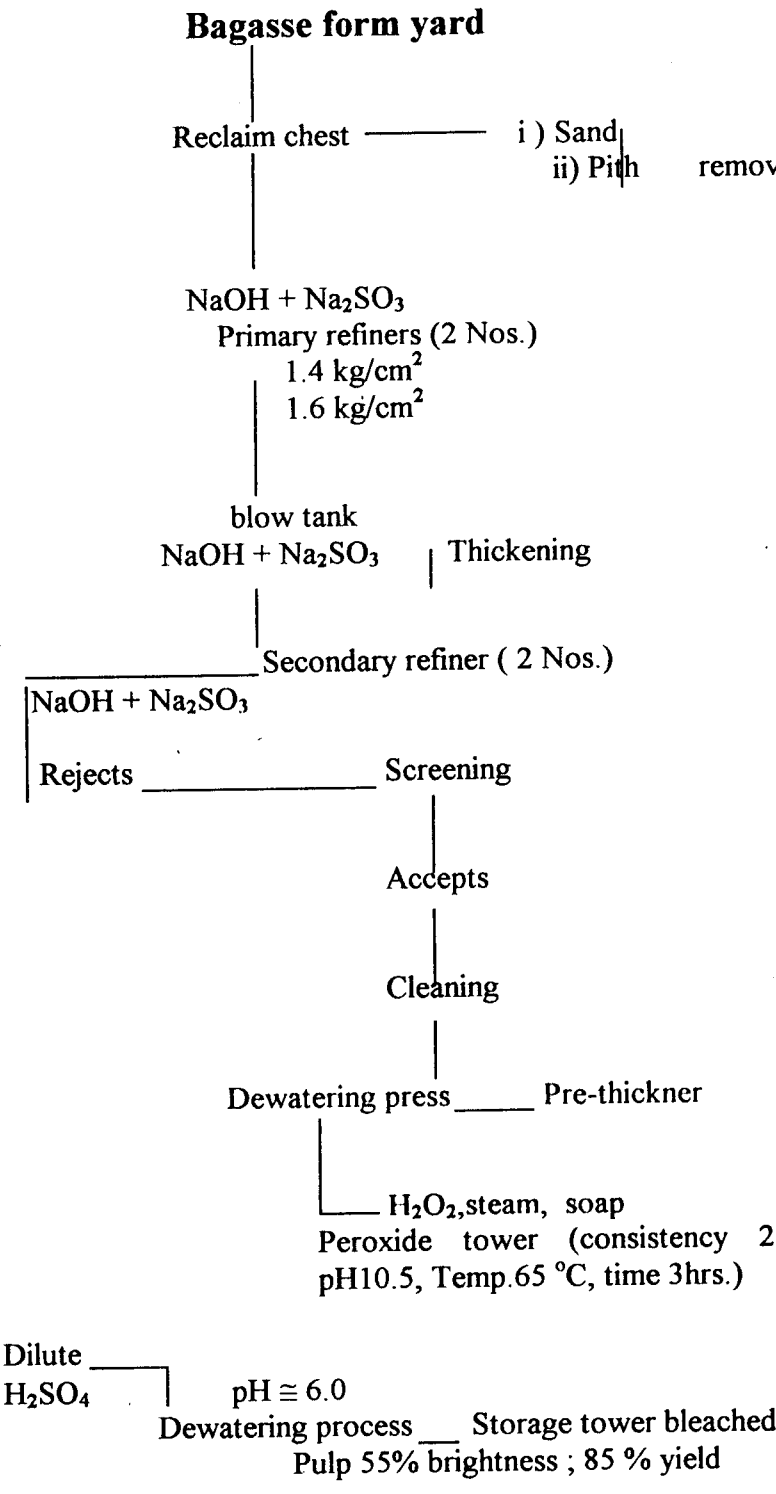
The observations made during visit of above named three mills are given below:

Tamilnadu Newsprint and Paper Mills

The mill is using bagasse mechanical bleached pulp (40 %), bagasse chemical pulp (30 %), imported CRMP (15 %) and mill broke (15 %) for making newsprint. Bagasse is subjected to dry depithing followed by wet cleaning and is stored wet.

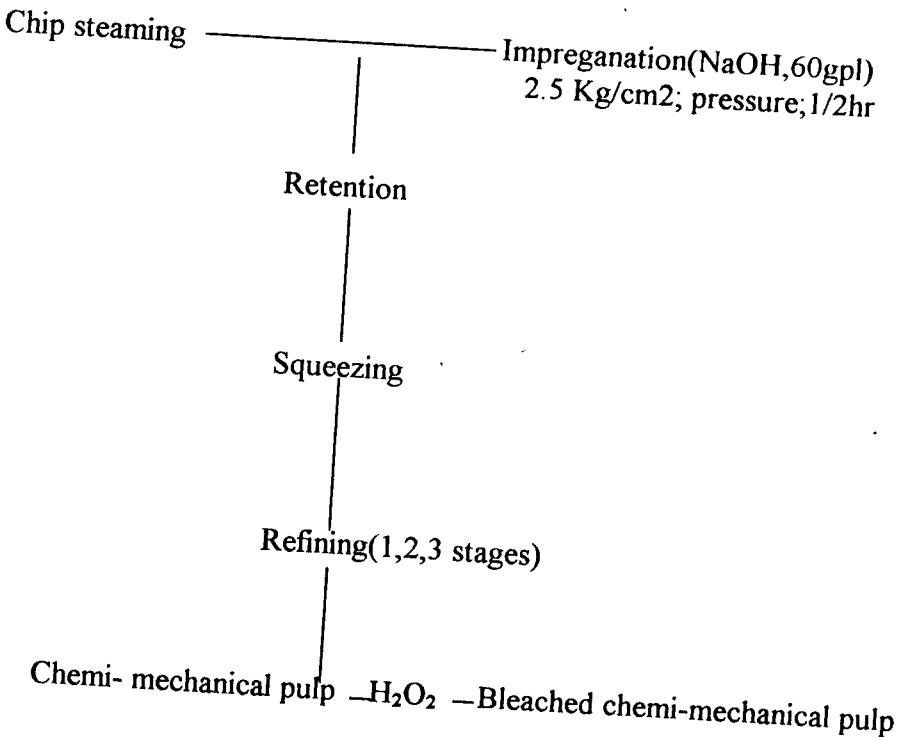
For mechanical pulping wet bagasse from yard is brought to reclaim chest where sand and pith are removed. It is then subjected to primary chemical treatment (3.0% NaOH and #.0% Na_2SO_3 at 25 % consistency) during pre-steaming followed by pressurized refining (primary refiners 2 nos.) at 1.4 Kg/Cm^2 and 110-115°C. After refining it is sent to blow tank and thickened to a consistency of 35%. Again it is subjected to chemical treatment (NaOH 1.5%, Na2SO3 1.5%0 followed by atmospheric refining (secondary refiners 2 nos). The refined pulp thus obtained is screened; accepts are send to cleaning system, dewatering press and subsequent bleaching by H_2O_2(2.0% at 25% consistency, pH 10.5, temp. 65°C.time 3 hr.). The peroxide bleached pulp is treated with dilute H_2SO_4 (final pH of pulp 6.0).The pulp thus obtained has 55-60% brightness and 85% yield (on depithed bagasse).

The flow diagram of the production of bleached chemical-mechanical pulp from bagasse given below.



Hindustan Newsprint limited

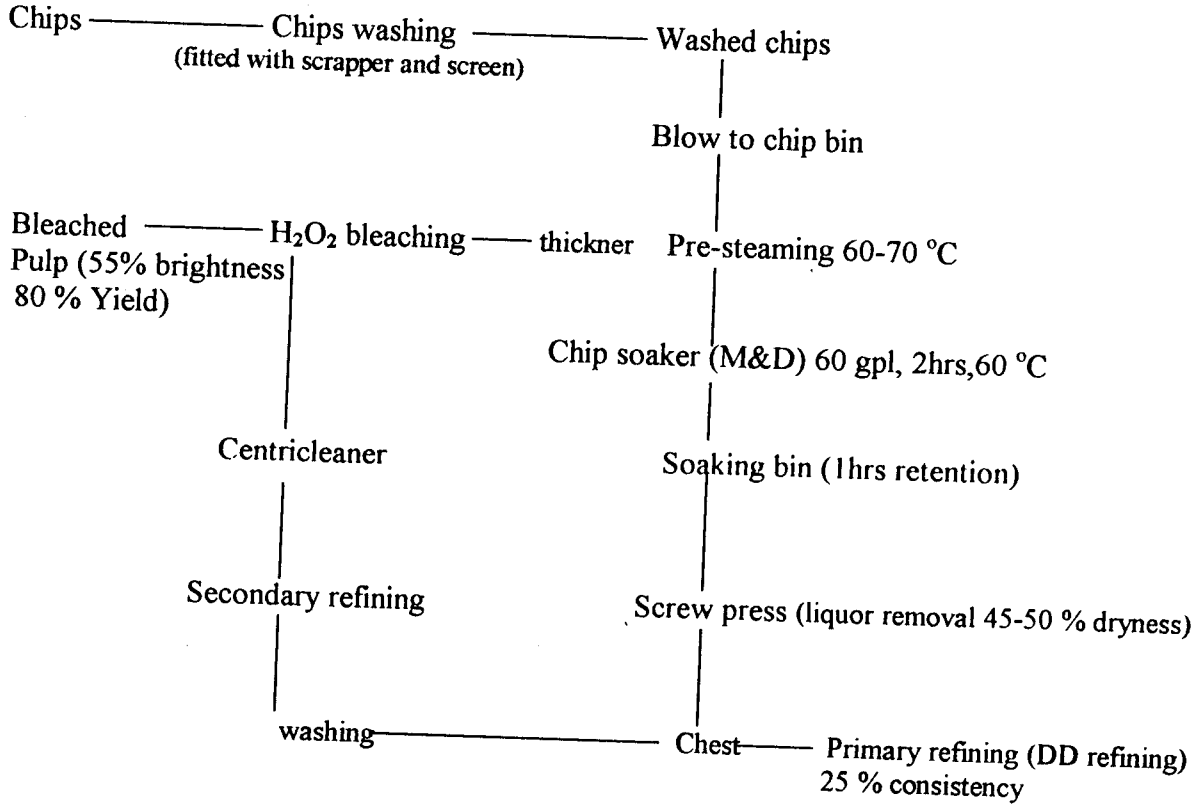
The mill is using hardwood viz. *Eucalyptus hybrid, Eucalyptus grandis* and *Acacia auriculiformus* and bamboo (Eta reed) for papermaking. Bleached chemi-mechanical pulp is being made from hardwoods and chemical pulp from Eta reed .Newsprint furnish is 65% bleached chemi-mechanical pulp from hardwoods, 25% chemical pulp from Eta reed and 10% imported pulp .The brightness of the newsprint is 55-58%.
Chemi-mechanical pulp is bleached by H_2O_2 to 60% brightness; the unbleached brightness being 28% for *Eucalyptus hybrid* and 35% for *Eucalyptus grandis*. The flow diagram for preparation of chemi-mechanical pulp is given below.



Mysore Paper Mills

The mill is using hardwoods viz, *Acacia auriculiformus, Eucalyptus hybrid* and bamboo for making newsprint.Bleached cold soda refiner mechanical pulp (CRMP) is being made form , *Acacia auriculiformus* and chemical pulp is being made from *Eucalyptus hybrid* plus bamboo. The newsprnt furnish is : CSRMP 55%, chemical pulp from *Eucalyptus hybrid* plus

bamboo 25%, imported CTMP 20%.The final brightness of newsprint is 55%.The CSRMP is bleached by H_2O_2 to 55% brightness; pulp yield being 80%.
The flow diagram of the process is given below:



Based on the observations made on the process/ equipment being used by the three big mills in India for production of high yield pulps for newsprint and on the basis of available literature on APMP process for production of high brightness high yield pulp for fine paper, specifications have been drawn up for the laboratory APMP system envisaged for procurement in IPMA-FRI project.

High yield pulping experiment (APMP process) with *Populus deltoides* using Prex de-watering / impregnation system

MATERIAL AND METHODS

Raw material : *Populus deltoides* (stored at 10-15 % moisture content; age 3 yrs old)

Pulping /bleaching chemicals : Caustic soda, hydrogen peroxide, sodium silicate, EDTA, Magnesium sulphate

Process : Alkaline Peroxide mechanical pulping

- -Pre-steaming of wood chips
- -Dewatering of pre-steamed chips
- -Chemical impregnation ($NaOH$,H_2O_2, Chelating agent)
- -Deliquoring
- -Defibration
- -Bleaching (H_2O_2 + $NaOH$ + Chelating agent)
- -Refining ($\simeq$ 250 ml CSF)

Pre-steaming of wood chips Wood chips (10-15 % moisture content) were Pre-steamed under pressure as well as atmospheric pressure to attain a moisture level at which the chips are sufficiently softened. It was observed that air dried chips having 10-15 % moisture content got softened after attaining about 100 % moisture content. The data on pre-steaming are recorded in Table 1

Table 1 Pre-steaming of chips

Sl.No	Pressure (Kg/cm^2)	Time (min)	Moisture attained (%)	Remarks
1	8	60	209.65	Chips were highly soft
2	8	15	195.86	Chips were highly soft
3	8	5	152.76	Chips were very soft
4	8	4	145.26	Chips were very soft
5	5	5	142.50	Chips were very soft
6	5	3	96.27	Chips were mildly soft
7	Atmospheric	30	176.97	Chips were soft
8	Atmospheric (over sieve)	30	125.67	Chips were less soft

Initial moisture in chips was 10-15 % on o.d. basis

Dewatering of pre-steamed chips Pre-steamed chips about 100 % moisture content were used in these experiments. For dewatering a stainless steel (3161) dewatering unit was designed and got fabricated. The unit consisted of circular casing with perforation at the bottom and on side wall and plunger to fit into the casing for compressing the chips contained in the casing by a hydraulic press.
The pre-steamed chips were kept in the casing and the plunger was placed over the chips. This unit was placed in a stainless steel tray (for collecting the effluent i.e ., dewatering material) and the whole assembly was placed between the two plates of the hydraulic press. Pressure was applied to compress and dewatered the chips (1000-3000 psi). the compression process was carried out 3-4 times to achieve maximum dewatering. The maximum dewatering achievable by the above device was 50 % from 100 % moisture content in the pre-steamed chips.

Chemical impregnation /Deliquoring/Bleaching A series of experiments were carried out at varying doses of caustic soda , hydrogen peroxide and chelating agent. The dewatered chips were impregnated with caustic soda, hydrogen peroxide and chelating agent. The chemical impregnated chips were deliquoring in the same unit in which the dewatering was carried out at 1000-3000 psi. The liquor thus removed was collected and analysed. The deliquored chips so obtained were defibrated and further treated with hydrogen peroxide in alkaline medium using chelating agent in polythene bags to obtained bleached pulp.

It was observed that chemical impregnation with caustic soda alone followed by deliquoring followed by hydrogen peroxide treatments did not give satisfactory results with respect to brightness.

It was seen that chemical impregnation with caustic soda with hydrogen peroxide and chelating agent followed by deliquoring and defibration followed by hydrogen peroxide treatments in presence of alkali and chelating agent gave better results with respect to brightness.

Further it was observed that application of hydrogen peroxide in two stages i.e first at chemical impregnation stage and secondly at subsequent at treatment stage of deliquored defibrated material gave better results than applying in three stages the results are recorded in Table 3-6.

Refining / Beating The pulps obtained after mentioned treated were refined in laboratory refiner /beaten in valley beater to about 200ml CSF.

Pulp evaluation Pulp yield and brightness was determined. Standard hand sheets were prepared and tested of strength properties.

Microscopic study Photo graphs 1-8 reveal effect of treatment on the fibre structure in the chip/ pulp

Effluent characterisation The effluent collected on dewatering of pre-steamed chips was analysed for COD, TS, Residual alkali etc. The results are recorded in Table 2

Stored chips were pre-steamed under 5Kg/cm^2 for three minutes to attain moisture level about 100% and pre-steamed chips were dewatered in dewatering/compressing unit at 3000psi and compression process was carried out 3-4 times to achieve maximum dewatering. The maximum dewatering achievable was 50 to 55% from the level of 100% moisture content in the pre-steamed chips. The dewatered chips were treated with different doses of chemicals.

Table 2 : Characteristics of effluent generated on dewatering of pre-steamed chips

Parameters	Value
Effluent generated, ml/100gm of O.D chips	50-60
Total solids, %(w/w)	2.1-2.6
Total dissolved solids, %(w/w)	1.90-2.20
Suspended solids, %(w/w)	600
COD, mg/l	8640

Table 3: Experimental condition of stored *Populus deltoides* with and without sodium hydroxide impregnation followed by three stages alkaline peroxide treatment

Treatment/ properties	Experiments			
	I	II	III	IV
Chemical impregnation stage				
NaOH, gpl	10.0	20.0	-	-
Residual NaOH, gpl	7.2	17.2	-	-
Total solid,gpl, %	3.8	4.2	-	-
COD,gpl	38 80	-	-	-
Ist stage				
H_2O_2,%	1.0	1.0	1.0	1.0
NaOH,%	2.5	2.0	2.5	2.5
Residual, H_2O_2,%	Nil	Nil	Nil	Nil
IInd stage				
H_2O_2,%	1.5	1.0	1.0	1.0
NaOH,%	2.5	2.0	2.0	Nil
Residual, H_2O_2,%	0.31	0.35	Nil	Nil
IIIrd stage				
H_2O_2,%	1.0	1.0	2.0	1.0
NaOH,%	2.0	2.0	2.0	1.5
Residual, H_2O_2,%	0.47	0.41	0.34	2.5
Pulp yield, %	81.0	80.6	83.72	84.0
Brightness, %	62.44	62.25	63.50	65.0
Tensile index, Nm/g	63.75	68.84	47.20	46.26
Burst index,kPam^2/g	3.46	3.94	2.33	2.12

* 3.0 % Na_2SiO_3, 0.25 % $MgSO_4$, 0.25 % EDTA is given with every H_2O_2 stages

Table 4: Experimental condition of stored *Populus deltoides* two stages alkaline peroxide treatment

Treatment/ properties	Experiments			
	I	II	III	IV
I[st] stage				
H_2O_2,%	2.0	2.5	3.0	3.0
NaOH,%	3.0	3.0	4.0	5.0
Residual, H_2O_2,%	0.12	0.23	0.63	0.59
II[nd] stage				
H_2O_2,%	1.0	1.0	1.0	1.0
NaOH,%	1.0	1.0	2.0	2.0
Residual, H_2O_2,%	0.21	0.31	0.56	0.50
Pulp yield, %	86.0	85.21	84.72	83.63
Brightness, %	66.63	66.58	65.50	64.85
Tensile index, Nm/g	42.36	42.12	43.26	43.76
Burst index,$kPam^2/g$	2.03	2.00	2.16	2.18

* 3.0 % $Na_2Si_2O_3$, 0.25 % $MgSO_4$, 0.25 % EDTA is given with every H_2O_2 stages

Experiments with freshly felled *Populus deltoides* (Moisture content 55.6 %)

Without pre-steaming

Freshly felled green ***Populus deltoides*** logs were brought form Haryana in log form.The initial moisture content of the chips was about 55.6%. Chips (200gm o.d.) were dewatered 3-4 times in dewatering/ compressing unit at 3000 psi to remove maximum possible water present in the chips. It was possible to remove about 40% of the water present in the chips. The dewatered chips were treated with peroxide in three stages (total peroxide charged was 3%) along with alkali and chelating agents.

It is observed that 70 % brightness and adequate strength properties were achievable without pre-steaming in case of freshly felled chips (initial moisture content more than 50%). The results are summarized in Table –5.

With pre-steaming

Freshly felled chips were pre-steamed under $5Kg/cm^2$ for three minutes to attain moisture level about 100% and pre-steamed chips were dewatered in dewatering/compressing unit at 3000psi and compression process was carried out 3-4 times to achieve maximum dewatering.The maximum dewatering achievable was 50 to 55% from the level of 100% moisture content in the pre-steamed chips. The dewatered chips were treated with same doses of chemical as practiced in without pre-steamed chips.

It was observed that there was only marginal improvement in brightness and strength properties as compared to the pulp obtained without pre-steaming(Table –5).

The advantage of processing freshly felled chips of *Populus deltoides* having more than 50% moisture is that it does not require pre-steaming stage thus avoiding one step of the process resulting in less effluent generation and energy consumption.

Table- 5 Three stage Alkaline Peroxide Treatment without pre-steaming and chemical impregnation of freshly felled *Populus deltoides*

Treatment/ properties	Experiments		
	I	II	III
Ist stage			
H_2O_2,%	2.0	3.0	2.0
NaOH,%	3.0	4.0	4.0
Residual, H_2O_2,%	0.33	0.63	0.26
IInd stage			
H_2O_2,%	1.0	1.0	1.5
NaOH,%	1.0	1.0	1.0
Residual, H_2O_2,%	0.62	0.51	0.52
Pulp yield, %	89.0	88.21	87.72
Brightness, %	67.84	68.32	69.85
Tensile index, Nm/g	47.28	48.78	48.02
Burst index,kPam^2/g	1.98	2.32	2.02

* 3.0 % $Na_2Si_2O_3$, 0.25 % $MgSO_4$, 0.25 % EDTA is given with every H_2O_2 stages

Table-6 Alkaline peroxide treatment in freshly felled *Populus deltoides*

Treatment/ properties	Experiments		
	I	II	III
Ist stage			
H_2O_2,%	1.0	1.50	2.0
NaOH,%	3.0	4.0	4.0
Residual, H_2O_2,%	nil	0.43	0.21
IInd stage			
H_2O_2,%	1.0	1.0	1.5
NaOH,%	1.0	1.0	1.0
Residual, H_2O_2,%	0.12	0.41	0.52
Pulp yield, %	89.0	87.21	86.14
Brightness, %	67.84	69.32	70.71
Tensile index, Nm/g	47.28	48.82	48.92
Burst index,kPam^2/g	1.98	2.30	2.31

* 3.0 % $Na_2Si_2O_3$, 0.25 % $MgSO_4$, 0.25 % EDTA is given with every H_2O_2 stages

High yield pulping experiment (APMP process) with *Populus deltoides* using Continuous Compressing Cum De-Watering Unit

Design parameters of the unit

Capacity : 2 - 5 kg/ hr

De-watering efficiency : 100 : 20-25 (w/w) water/ liquor removal to residual water/ liquor in chips

Pressure : 10,000 psi

Number of trials were carried out on compression-cum-dewatering unit at different operating conditions to achieve targeted wood to liquor/water ratio in de-watered chips (4:1) .The soaked chips were de-watered at optimised rpm/pressure. The de-watered chip were bleached at different does of H_2O_2 to achieve maximum brightness, minimum pulp yield loss with higher strength properties. The energy consumed at each stage of dewatering(I^{st} /II^{nd} stage) was noted. The effluent generated at each stage was analysed. The summarized results of the experiments are as follows

Table 7 :Characteristic of effluent generated on Continuous Compressing Cum De-Watering Unit with different doses of H_2O_2

Parameters	Values			
I^{st} Stage Dewatering stage				
Effluent generated	1.14 - 1.18 liter			
COD	5600 - 5640 mg/l			
Total solids	0.76- 0.89%			
Energy required	0.44 -0.49 KWH/4 Kg A.D.Chips			
Chemical Treatment				
Hydrogen Peroxide	2.0%	2.0%	2.0%	2.0%
Sodium hydroxide	2.0%	2.0%	2.0%	2.0%
II^{nd} Stage Dewatering stage:				
Effluent generated	7.1- 8.08 liter			
COD	10500-11200 mg/l			
Total solids	2.11 -2.78 %			
Energy required	0.39 -0.42 KWH			
Chemical Treatment				
Hydrogen Peroxide	1.0%	1.5%	1.5%	2.0%
Sodium hydroxide	2.0%	2.0%	2.5%	2.5%
Yield	86.22	86.12	85.96	85.75
Brightness % ISO	75.26	75.38	76.83	78.03
Tensile index, Nm/g	42.06	43.68	45.93	46.82
Burst index,kPam^2/g	1.95	1.98	2.00	2.05
Tear index mNm^2/g	4.32	4.32	4.34	4.35

3.0 % Na_2SiO_3, 0.25 % $MgSO_4$, 0.25 % EDTA is given with every H_2O_2 stages

No. Dis/2001-C & P/3 (13)/IPMA
Cellulose and Paper Division
Forest Research Institute.
P.O.New Forest Dehra Dun. (U.P.)

Dated 19.07.2001

To:-

Sri R. Narayan Moorthy
Secretary
Indian Paper Manufacturer Association
PHD House (4th Floor),
4/2 Siri Institutional Area (Opp. Asian Games Village)
NEW DELHI 110016.

Subject : IPMA-FRI project - additional funds requirement for Fy 2001-2002 - regarding.

Sir,

We are enclosing herewith the consolidated report of work done on APMP of *Poplus deltoides* using (1) prex dewatering system (hydraulic) and (2) continuous compression - cum - dewatering unit (screw conveyer type).

The request for carry forward of the balance money Rs 6,12669/=00 for the Fy 2001-2002 has already been made vide our letter No 1771/2K/C&P/3(13)/IPMA dated 7th June 2001. Commitments for the expenditure of this amount have already been made.

We require now additional funds for the Fy 2001-2002 as per the details given below:

Additional Funds Requirement For Fy 2001-2002

(1)	Research Associate (two) w.e.f. Sept 2001 to march 2002	- Rs 1,15,000/- (@ Rs 8000/pm for each)
(2)	Equipments	
	(i) Pressurised Refiner	- Rs 12,00,000/- (Tenders already invited)
	(ii) Reaction Vessel	- Rs 6,00,000/- (Tenders being invited)
	(iii)Centrifugal ball mill	- Rs5,50,000/- (Tenders already invited)
	(iv) Pulp Classifier	- Rs 5,00,000/- (Tenders being invited)
(3)	Travel	- Rs 1,00,000/-
(4)	Chemical/Glassware	- Rs 1,00,000/-
(5)	Miscellaneous	- Rs 50,000/-
		Rs 32,15,000/-

In this connection kindly refer to the minutes of the meeting of Cess Fund Sub-Committee held under the chairmanship of Shri P.K.Jain Industrial Adviser on 12th June 2000 in Udyog Bhavan,Ministry of Industry New Delhi.In the meeting it was decided as reproduced below (reference- last line of the minutes of the meeting) :

" The balance of cess fund could be refunded and again considers by the Cess Committee after assessment of the results and getting full details of the other equipments with factual cost estimates and expenditure involved for pilot plant trial run and commercilisation."

Accordingly a sum of Rs 37.00 lacs was refunded to IPMA.

Now the requirement of additional fund for purchase of equipments with cost and under other heads has been finalised as enumerated above for Fy 2001-2002.

It is requested that the additional fund of Rs 32.15 lacs may kindly be released to us immediately for execution of the project as scheduled and envisaged.

Encl: As above

19.7.2k

Dr.S.V.Singh)
Head
Cellulose and Paper Division