

Project Details

Project Title	Increase in brightness ceiling of agro residue pulps in cost effective manner specifically for wheat straw
Project Executed by	Indian Agro & Recycled Paper Mills Association (IARPMA)
Participating Agency	Avantha Centre for Industrial R & D (ACIRD), Yamuna Nagar (Haryana) Central Pulp & Paper Research Institute (CPPRI), Saharanpur (UP)
Project Duration, Months	24
Date of Start	February 2014
Date of Completion	February 2017
Project Cost, Rs Lacs	50.0 (ACIRD - 22.5, CPPRI - 22.5, IARPMA - 5.0)
Funds Released	50.0
Balance Funds to be Released	Nil

Objectives

- **To find out reasons behind low brightness development in agro pulps**
- **To study the effect of different bleaching chemicals and modern bleaching processes on agro pulps**
- **To suggest best process for bleaching of agro pulps to +85% brightness**
- **Demonstration of results in the plant**

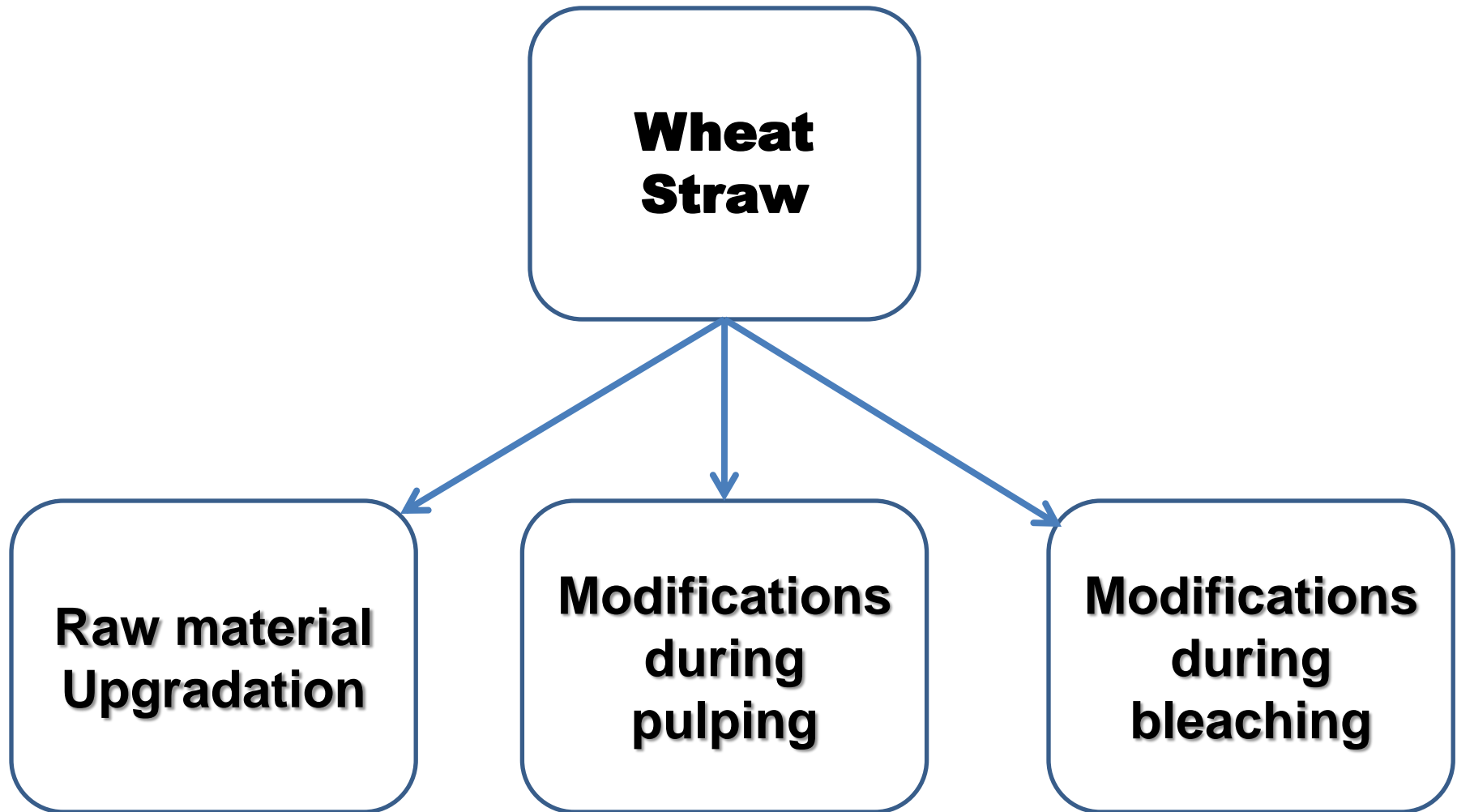
Quantified Deliverables

- **Production of +85% ISO brightness pulp from wheat straw in cost effective manner**

Work Progress (Summary)

Sl. No	Activities as per Work Plan	Status
1.	Literature survey	Completed
2.	Collection of mixed agro raw material, wheat straw and unbleached & bleached pulps from different mills	Completed
3.	Determination of optical and physical strength properties of pulps	Completed
4.	Study on effect of washing of wheat straw on pulp brightness	Completed
5.	Study on different pulping additives	Completed
6.	Study on different pre-bleaching treatments	Completed
7.	Study on different post-bleaching treatments	Completed
8.	Study on different in-situ-bleaching treatments	Completed
9.	Plant scale study with selected process	Completed
10.	Preparation of report	Completed and submitted

APPROACH TOWARDS PROBLEM



APPROACH TOWARDS PROBLEM

Raw material Upgradation

- **Effect of washing of the wheat straw**
 - Hot water washing (Effect of temperature)
 - Effect of use of back water
- **Effect of additives during washing**
 - Surfactants
 - Chelating agents
 - Mild Alkali
 - Acid pre treatment

APPROACH TOWARDS PROBLEM

Modifications during pulping

- **Optimization of pulping conditions**
 - Effect of kappa no.
 - Use of chelating agents
- Effect of pulping additives
 - Oxidative
 - Reductive
 - Redox

APPROACH TOWARDS PROBLEM

Modifications during bleaching

- **Pre-treatment of pulp before bleaching**
 - Acid treatment of pulps
 - Acidic peroxide
 - Chelating Agents
 - Enzymatic treatment
- **Optimization of bleaching chemicals during bleaching**
 - Hydrogen peroxide stabilizers and boosters
- **Post-bleaching treatment**
 - Acid treatment
 - Treatment of pulp by PAA

Results

Reasons behind low brightness

Hexenuronic acid

Particular	Set-1	Set-2	Set-3	Set-4
Kappa no.	17.7	14.1	13.1	12.0
Initial HexA ($\mu\text{mol/g}$)	8.3	11.5	13.5	19.1
<i>Acid treatment (time 2h, temperature- 90 °C)</i>				
Kappa no. <i>(Eliminating contribution of HexA)</i>	17.2	13.6	12.1	10.6
HexA ($\mu\text{mol/g}$)	2.1	3.5	3.5	4.6
Decrease in HexA (units)	6.2	8.0	10.0	14.5
Decrease in Kappa no.	0.5	0.5	1.0	1.40

Reasons behind low brightness

Metal ions

Metals	Value in unbleached pulp (ppm)	
	Wheat straw	Eucalyptus
Copper (Cu)	4.6	4.3
Manganese (Mn)	44.5	11.2
Zinc (Zn)	52.3	--
Iron (Fe)	204	37.9
Magnesium (Mg)	2,140	688
Calcium (Ca)	6,135	6503

Reasons behind low brightness

Removal of metal ions in pulping

Metals	Value in unbleached pulp (ppm)		
	Control	EDTA	HEDP
Copper (Cu)	4.6	1.6	1.2
Manganese (Mn)	44.5	42	42
Zinc (Zn)	52.3	38	36
Iron (Fe)	204	95	126
Magnesium (Mg)	2,140	1920	1843
Calcium (Ca)	6,135	--	--

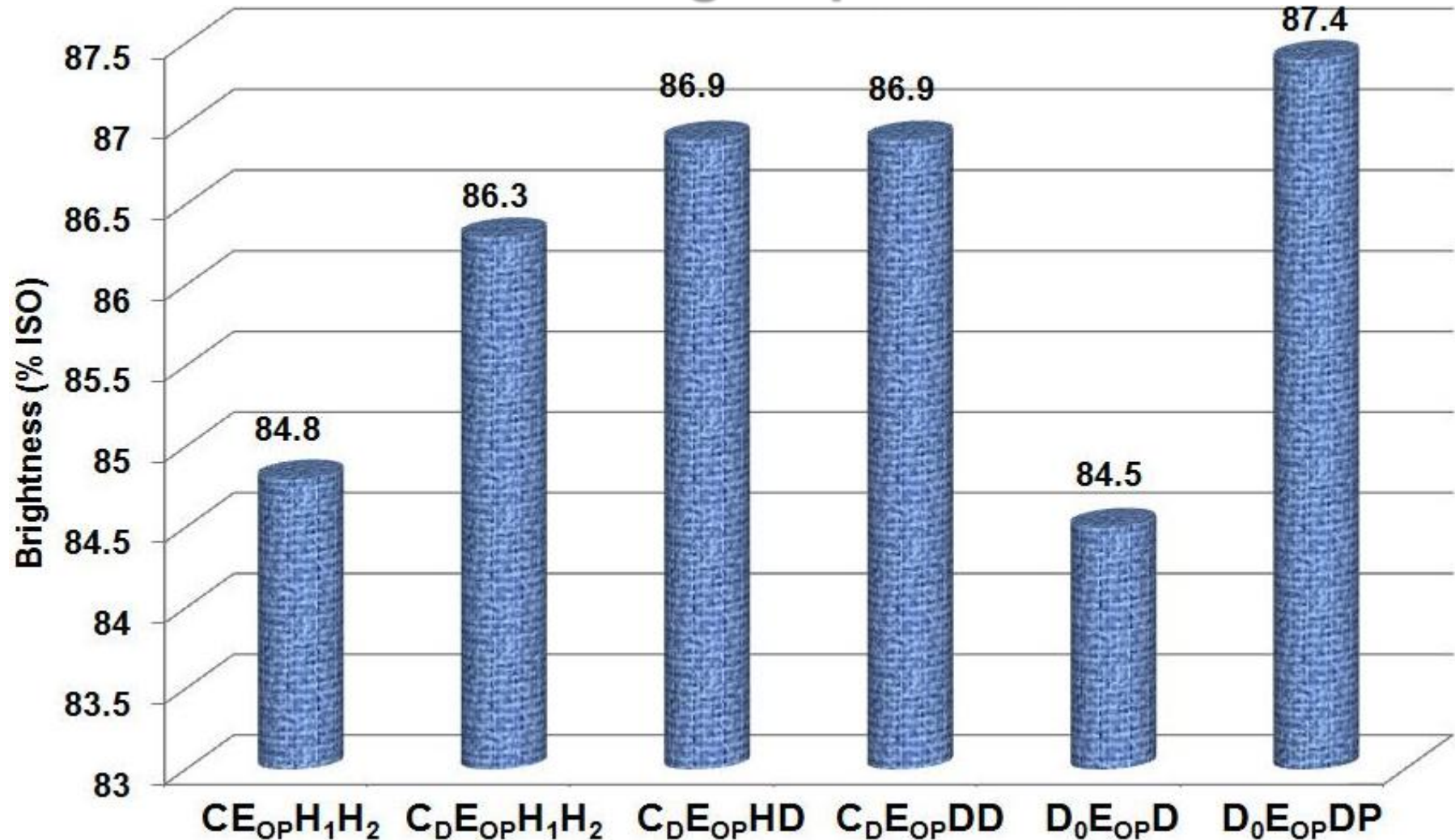
Reasons behind low brightness

Removal of metal ions in acid treatment

Metals	Value in unbleached pulp (ppm)	
	Control	Acid treated
Copper (Cu)	4.6	1.8
Manganese (Mn)	44.5	32.2
Zinc (Zn)	52.3	12.2
Iron (Fe)	204	65
Magnesium (Mg)	2,140	916
Calcium (Ca)	6,135	--

Effect of different bleaching chemicals

Final brightness achieved by using different bleaching sequences



Effect of raw material cleaning on wheat straw

Effect on ash, silica, iron and calcium

Parameters	As Such wheat Straw	Dry Depithed wheat Straw	Wet cleaned Wheat Straw
Ash (%)	12.3	11.7	7.9
Silica (%)	6.5	6.3	6.1
Calcium (ppm)	4500	4080	3750
Iron (ppm)	260	240	150

Effect of raw material cleaning on wheat straw

Effect of raw material cleaning on pulping

Particular	Set-1	Set-2	Set-3	Set-4
	Unwashed		Washed	
NaOH (%)	15.0			
AQ (%)	0.05			
Screened pulp yield (%)	49.2	49.4	53.6	53.8
Rejects (%)	1.1	1.2	0.9	1.0
Kappa no.	10.3	10.8	9.3	9.7
pH	11.3	11.3	11.6	11.8
Free alkali (g/l)	2.1	1.9	3.9	4.1
Black liquor solids, w/w (%)	11.2	11.3	10.7	10.8

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Bath ratio - 1:4, Temperature - 166°C, Cooking time - 30 min

Effect of raw material cleaning on wheat straw

Effect of raw material cleaning on bleaching

Parameter	Unwashed		
Kappa Number	10.5		
<i>D₀ Stage (Consistency -5%, Temp.- 55°C, R.T. – 45 min, Kappa factor -0.25)</i>			
ClO ₂ added (%)	1.0		
Residual Cl ₂ (ppm)	10.8		
End pH	2.5		
<i>E_{OP} Stage (Consistency -10%, Temp.- 80 °C, time - 90 min)</i>			
NaOH added (%)	1.36		
H ₂ O ₂ added (%)	0.5		
Final pH	10.3		
Residual H ₂ O ₂ (ppm)	20		
Kappa Number	1.7		
Brightness (% ISO)	77.6		
<i>D Stage (Consistency -10%, Temp.- 80 °C, R.T. – 180 min)*</i>			
ClO ₂ added (%)	0.4	0.6	0.8
Final pH	3.5	3.6	3.8
Residual ClO ₂ (ppm)	10.8	27	59.4
Brightness (% ISO)	83.0	83.6	84.1

Effect of raw material cleaning on wheat straw

Effect of raw material cleaning on bleaching

Parameter	Wet cleaned		
Kappa Number	9.5		
<i>D₀ Stage (Consistency -5%, Temp.- 55°C, R.T. – 45 min, Kappa factor -0.25)</i>			
ClO ₂ added (%)	0.9		
Residual Cl ₂ (ppm)	10.8		
End pH	2.3		
<i>E_{OP} Stage (Consistency -10%, Temp.- 80 °C, time - 90 min)</i>			
NaOH added (%)	1.2		
H ₂ O ₂ added (%)	0.5		
Final pH	10.2		
Residual H ₂ O ₂ (ppm)	12.2		
Kappa Number	1.5		
Brightness (% ISO)	78.3		
<i>D Stage (Consistency -10%, Temp.- 80 °C, R.T. – 180 min)*</i>			
ClO ₂ added (%)	0.4	0.6	0.8
Initial pH	3.0	2.9	2.8
Final pH	3.6	3.8	3.7
Residual ClO ₂ (ppm)	16.2	37.8	48.6
Brightness (% ISO)	84.8 (+1.8)	85.7 (+2.1)	86.3 (+2.2)

Effect of raw material cleaning on wheat straw

Effect of raw material cleaning on strength

Parameters	Control	Dry Depithed	Wet Cleaned
Brightness (% ISO)	83.2	83.8	85.7
°SR	29	28	28
Grammage (g/m ²)	61.0	61.2	60.1
Bulk (cc/g)	1.33	1.32	1.31
Tensile index (Nm/g)	46.7	49.1	50.9
Burst index (kN/g)	4.1	4.1	4.0
Tear index (mN m ² /g)	5.7	5.8	6.2
Roughness (ml/min)	83	70	66

Effect of using b/w during washing

Effect on pulping

Particular	Set-1	Set-2	Set-3	Set-4
TDS in wash water (g/l)	5000	4000	3000	2000
TSS in wash water (g/l)	1000	800	600	400
Pulping condition (<i>Bath ratio - 1:4, Temp. - 166°C, time - 30 min, AA - 15%, AQ - 0.05%</i>)				
Screened pulp yield (%)	52.2	52.6	52.8	53.1
Rejects (%)	1.7	1.5	1.4	1.2
Kappa no.	11.6	11.2	10.7	10.2
pH	11.1	11.3	11.4	11.5
Free alkali (g/l)	2.3	2.7	3.3	3.6
Black liquor solids, w/w (%)	11.6	11.2	11.1	11.0

Study on pre and post bleaching treatments

Effect of acid treatment on pulp brightness

Particulars	Brightness achieved* (% ISO)
Acid treatment (<i>Consistency -10%, Temp.- 80 °C, time – 60 min, 2 pH</i>)	
Control	84.5
Pre bleaching acid (H ₂ SO ₄) treatment	86.5 (+2.0)
Acid (H ₂ SO ₄) treatment (in situ with D ₀ stage)	86.0 (+1.5)
Post bleaching acid (H ₂ SO ₄) treatment	86.6 (+2.1)

**Bleaching sequence followed D₀E_{OP}D*

Study on pre and post bleaching treatments

Effect of acid treatment on pulp brightness

Particulars	Brightness achieved* (% ISO)	PC number
Pre bleaching acid treatment (<i>Consistency -10%, Temp.- 80 °C, time - 60 min, 2 pH</i>)		
Control	84.8	2.17
Acid Pre-treated (H ₂ SO ₄)	86.4 (+1.6)	1.56
Acid Post-treated (H ₂ SO ₄)	86.6 (+1.8)	1.41

*Bleaching sequence followed $CE_{OP}H_1H_2$

Study on pre and post bleaching treatments

Hydrogen peroxide pre-treatment of pulp

Particular	Control	ODL	Peroxide treated
<i>Peroxide treatment</i> (Peroxide – 1.5%, Cy - 10%, Temp. - 80°C, Time - 2 h, EDTA – 0.2%) <i>ODL</i> (Oxygen – 1.6%, Cy - 10%, Temp. - 90°C, Time – 90 min)			
Kappa Number	14.3	9.2	10.3
Brightness (% ISO)	40.3	48.3	54.2
Properties of D ₀ E _{OP} D bleached pulp			
ClO ₂ /NaOH used (%)	2.27 /2.3	1.69 /1.6	1.66 /1.7
Brightness (% ISO)	84.6	85.6	86.7
CIE (Whiteness)	75.1	76.7	77.9
Viscosity (cP)	12.1	11.9	11.7
Properties of the effluent generated during bleaching			
Color (kg/t)	20.1	--	14.7
COD (kg/t)	44.5	--	29.6
AOX (kg/t)	0.66	--	0.43

Study on pre and post bleaching treatments

Hydrogen peroxide pre-treatment of pulp

Particular	Control	ODL	Peroxide treated
Peroxide treatment (Peroxide – 1.5%, Cy - 10%, Temp. - 80°C, Time - 2 h, EDTA – 0.2%) ODL (Oxygen – 1.6%, Cy - 10%, Temp. - 90°C, Time – 90 min)			
Kappa Number	14.3	9.2	10.3
Brightness (% ISO)	40.3	48.3	54.2
Viscosity (cP)	14.1	12.3	12.2
Properties of CE_{OP}HH bleached pulp			
Cl ₂ /NaOH used (%)	4.9 /2.6	3.6 /1.7	3.7 /1.8
Brightness (% ISO)	84.9	85.9	86.6
CIE (Whiteness)	76.1	76.9	77.8
Viscosity (cP)	10.2	10.0	9.9
Properties of the Effluent generated during bleaching			
Color (kg/t)	47.7	32.8	29.8
COD (kg/t)	61.7	42.5	47.4
AOX (kg/t)	2.5	1.7	1.8

Study on pre and post bleaching treatments

Enzymatic pre-bleaching treatment of pulp

Particular	Control	Enzyme treated
Kappa no.	14.7	
Enzyme pre-treatment (Consistency – 8.0%, Temperature - 65°C, Retention time - 1 h)		
Enzyme* dose (g/T)	-	150
C - Stage (Consistency – 2.5%, Temperature - 40°C, Time - 45 min., KF-0.26)		
End pH	2.3	2.3
E _{OP} - Stage (H ₂ O ₂ -0.7%, Cy - 10%, Temp. - 75°C, Retention time - 120 min.)		
NaOH added (%)	2.3	2.3
End pH	10.0	10.1
Residual H ₂ O ₂ (ppm)	40.8	20.4
Brightness (% ISO)	75.9	77.2
E _{OP} pulp kappa no.	1.2	1.0

* Xylanase activity – 10,300 IU/g.

Study on pre and post bleaching treatments

Enzymatic pre-bleaching treatment of pulp

Particular	Control	Enzyme treated
H_1 - Stage (Consistency - 10%, Temperature - 40°C, Retention time - 120 min.)		
Hypo added (%)	0.5	0.5
End pH	8.7	8.7
Brightness (% ISO)	81.6	82.4
H_2 - Stage (Consistency - 10%, Temperature - 40°C, Retention time - 120 min.)		
Hypo added (%)	0.25	0.25
End pH	7.6	7.5
Residual hypo (ppm)	32.6	49.7
Brightness (% ISO)	84.5	85.2
Whiteness (CIE)	72.7	74.1
Viscosity (cP)	9.3	8.9

Study on pre and post bleaching treatments

Effect of enzymatic pre-treatment on effluent

Particular	Control	Enzyme
pH	2.64	2.67
Total solids (%)	0.37	0.37
Colour (PCU)	384	329
COD (kg/t)	110	98

Study on bleaching additives

Effect of using hydrogen peroxide activator

Parameter	Control	With activator	
		Same bleaching chemicals	Reduced bleaching chemicals
Kappa Number	12.9		
C stage (Kappa factor - 0.26, Cy - 3.0%, Temp. - 35°C, Time - 45 Min.)			
E _{OP} stage (Cy - 10.0%, Temp. - 70°C, Time - 120 Min, O ₂ - 0.6%)			
Activator dose (g/TP)	0	100	
H ₂ O ₂ /NaOH added (%)	0.5/2.2	0.5/2.2	0.4/2.05 (- 1.0/1.5 kg/T)
Residual H ₂ O ₂ (ppm)	10.2	6.8	3.4
Kappa No.	1.1	0.9	1.0
Brightness (% ISO)	76.8	78.3	77.9

Study on bleaching additives

Effect of using hydrogen peroxide activator

Parameter	Control	With activator	
		Same bleaching chemicals	Reduced bleaching chemicals
H ₁ stage (Cy - 10.0%, hypo - 0.80%, Temp. - 45°C, Time - 120 Min.)			
Activator dose (g/TP)	0	50	
Brightness (% ISO)	83.6	84.2	83.8
H ₂ stage (Cy - 10.0%, Hypo - 0.3%, Temp. - 45°C, Time - 120 Min.)			
Brightness (% ISO)	84.2	85.1 (+0.9)	84.8
Whiteness (CIE)	75.8	77.1 (+1.3)	76.1
P. C. number	2.63	2.21 (-0.42)	2.21
Viscosity (cP)	10.6	11.3 (+0.7)	12.0

Major Achievements...

Laboratory scale studies

- With the pre-bleaching acid treatment of pulp, final brightness of pulp achieved was 86.5% (Gain in brightness by 1.6-2.0 units).
- With the post-bleaching acid treatment of pulp or in-situ acid treatment of pulp, final brightness of pulp achieved was 86.0% (Gain in brightness by 1.1-1.5 units).
- Hydrogen peroxide (H_2O_2) pre-treatment of unbleached wheat straw pulp improved final bleached pulp brightness by 1.7-2.1 units with substantial reduction in chlorine based bleaching chemicals and pollutants generated during bleaching.
- With the replacement of 20% Cl_2 with ClO_2 in chlorination stage of bleaching, final brightness of pulp achieved was 86.3% (Gain in brightness by 1.5-1.8 units).
- Use of hydrogen peroxide activator in E_{OP} stage of bleaching sequence improved the final bleached pulp brightness by 1.1 units and whiteness by 1.5 units.

Major Achievements...

Plant scale studies...

- Average final pulp brightness improved by 1.1 units to 83.6% from a control of 82.5%. During the trial a few values also crossed the brightness ceiling of 85%, whereas during control run none of the value obtained was above 85% brightness.
- Average final pulp whiteness obtained was 69.9 indicating the whiteness improvement by 3.2 units.
- Average final pulp yellowness reduced to 7.6 indicating the reduction in yellowness of 1.1 point.
- As expected viscosity of final bleached pulp was improved marginally to 5.6 cP compared to 5.2 cP obtained during control.

Major Achievements

Plant scale studies

Table 1: Number of occurrence in final pulp brightness slabs

Brightness slab	Pre-trial	During trial
≥85.0	0 (0%)	3 (4.5%)
≥83.5 <85.0	8 (24.2%)	40 (60.6%)
≥82.5 <83.5	10 (30.3%)	12 (18.2%)
≥80 <82.5	14 (42.5%)	11 (16.7%)
<80	1 (3.0%)	0 (0%)

Table 2: Number of occurrence in final pulp whiteness slabs

Whiteness slab	Pre-trial	During trial
≥75	0 (0%)	3 (4.5%)
≥74 <75.0	0 (0%)	4 (6.1%)
≥70 <74	9 (27.3%)	27 (40.9%)
≥65 <70	13 (39.4%)	25 (37.9%)
≥60 <65	8 (24.2%)	7 (10.6%)
<60	3 (9.1%)	0 (0%)