Application of Per-acetic Acid in Chemical Pulp Bleaching

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Per-acetic acid (PAA) treatment of different mill pulps (paper grade and dissolving grade) having different proportions of hardwood and bamboo was carried out. The PAA treatment of final bleached pulps was done at varying pH, reaction time, temperature and dose to get maximum gain in optical properties. Use of per-acetic acid at a dose of 0.5-1.0 kg/TP at ambient temperature and normal plant pulp pH, increased the final pulp brightness by 1.0-1.5 points and whiteness by 2.0-3.5 points. Yellowness and post colour number of pulp also reduced without affecting the viscosity. Almost similar increase in brightness and whiteness was observed when PAA treated pulp was used for the production of high brightness paper (+96% ISO). The brightness and CIE whiteness of PAA treated (1.0 kg/TP) high brightness paper were 97.9% ISO (+1.1) and 142.7 points (+3.2) respectively whereas the same of control (no PAA treatment) were 96.8% ISO and 139.5 points respectively.

The effects of addition of PAA in E_{OP} stage and dioxide stage were also studied. Not much improvement in final brightness and whiteness was observed when PAA was used at a dose level of 0.5-1.0 kg/TP in E_{OP} and D stages. It was noted that in the D stage about 1.0 and 3.0 kg of ClO_2 could be replaced by treatment with PAA at a dose level of 1.0 and 1.5 kg/TP respectively. Use of per-acetic acid in D stage can partially replace ClO_2 where ClO_2 is a limitation.

INTRODUCTION

Per-acetic acid (PAA) has been evaluated as a delignification or bleaching agent for chemical pulp and textile since early 1950's (1). Bailey and Dence published a research paper describing the use of per-acetic acid for both delignification and brightening of chemical pulp (2). Liebergott has reported use of peroxy acids in different stages of pulping and multistage bleaching process for chemical pulps (Table 1) (3).

The brightness and whiteness of fully bleached kraft pulps were efficiently increased with 1-2 kg PAA/TP. Brightness reversion of bleached pulps and during beating in paper machines was also prevented by use of PAA (4).

Use of per-acetic acid in bleaching stages or in the storage tower for bleached pulp increases pulp brightness and bleaching selectivity. The optical properties of fibres are improved and pulp brightness is stabilized when per-acetic acid is used in stock preparation or post bleaching (5). In spite of their known delignifying and oxidizing power, poor stability and high chemical cost has been considered as a major disadvantage for their potential use in prebleaching or bleaching applications (6).

In the present study, an attempt has been made to improve the optical properties of final bleached paper grade and dissolving grade pulps and also of the high brightness paper by treatment with PAA. The effects of addition of PAA in E_{OP} stage and dioxide stage were also studied.

Table 1: Peracid peroxy acid use in the bleaching sequence

1)	Delignifying agent for: 1) Wood (pulping process such as Milox) 2) Pulp (replacement for an initial chlorination stage) or in an E_{OP} stage
2)	Activation agent: 1. To activate lignin before or between O_2 stage(s) 2. To activate a peroxide stage together with oxygen under acidic conditions
3)	Brightening agent: 1. ECF bleaching as a replacement for a chlorine dioxide or hypochlorite stage 2. TCF bleaching as a replacement of peroxide stage

EXPERIMENTAL

Unbleached and bleached pulps from Mill A (20% bamboo + 80% hardwood), Mill B (60% bamboo + 40% hardwood), Mill C (35% bamboo + 65% hardwood) and Mill D (100% hardwood dissolving grade) were taken for PAA treatments. PAA treatment on bleached pulps were given at 10% consistency with per-acetic acid at different conditions as given in the corresponding tables. Bleached pulps were characterized for brightness, CIE whiteness, L*a*b* values, yellowness, post colour (PC) number and viscosity.

Analytical techniques

Moisture content of the pulp was determined as per Tappi Test Method T 210 cm-86.

Brightness and whiteness of pulp was determined using Technibrite Brightness Meter (Model TB 1c) as per Tappi Test Method T 525 om-02 and T 562 pm-96.

Viscosity, of pulp was measured as per Tappi Test Method T 230 om-82.

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PC no. of pulp was measured as per Tappi Test Method T 260 om-85.

RESULTS AND DISCUSSION

PAA treatment of different mill pulps

Mill A pulp (20% bamboo + 80% hardwood)

Pulps of different initial brightness 88.0 to 89.1% ISO were treated with PAA. Treatment was done at ambient temperature (~25^{0}C) for 3 h at a dose of 1.0-2.0 kg/TP, pH 5.0-8.0. Developme--nt in brightness was not affected by variation in the initial pulp pH. Final brightness increased by 1.4-1.6 ISO points and 1.6-1.8 ISO points at a PAA dose of 1.0 kg/TP and 2.0 kg/TP respectively. CIE whiteness increased by 2.2-2.9 points and 2.6-3.7 points at a PAA dose of 1.0 kg/TP and 2.0 kg/TP respectively (Table 2.1). Yellowness was also lower (lower b* values) in case of the treated samples.

Table 2.1: Treatment of Mill A bleached pulp* at different pH and PAA dose and ambient conditions

Parameter	Control				PAA (1.0 kg/TP)				PAA (2.0 kg/TP)			
Initial pH	5.0	6.0	7.0	8.0	5.0	6.0	7.0	8.0	5.0	6.0	7.0	8.0
Final pH	5.1	6.1	7.0	8.0	3.0	3.5	4.0	4.2	3.1	3.4	3.8	4.0
Temp. (°C)	25	25	25	25	25	25	25	25	25	25	25	25
Time (min)	180	180	180	180	180	180	180	180	180	180	180	180
Bleached pulp properties												
Final brightness (% ISO)	88.4	88.4	88.5	88.4	90.0 (+1.6)	90.0 (+1.6)	89.9 (+1.4)	90.1 (+1.7)	90.0 (+1.6)	90.2 (+1.8)	90.3 (+1.8)	90.0 (+1.6)
CIE whiteness	83.6	83.8	83.4	83.8	85.8 (+2.2)	86.2 (+2.4)	86.3 (+2.9)	86.3 (+2.5)	86.6 (+3.0)	86.9 (+3.1)	87.1 (+3.7)	86.4 (+2.6)
L*	96.36	96.37	96.39	96.37	96.75	96.82	96.83	96.82	96.80	96.84	96.80	96.47
a*	0.93	0.88	0.86	0.85	0.80	0.83	0.86	0.83	0.85	0.84	0.80	0.86
b*	1.89	1.77	1.71	1.79	1.34	1.26	1.25	1.25	1.18	1.13	1.05	1.25
ASTM yellowness	2.44	2.71	2.63	2.74	2.05	1.93	1.91	1.92	1.81	1.73	1.62	1.93

** Initial pulp brightness 88.4% (PAA 40% concentration)*

Table 2.2: Effect of reaction time and PAA dose at ambient temperature on bleached pulp properties

Reaction time (h)	PAA dose (kg/TP)	Brightness (% ISO)	CIE whiteness	PC no.
3.0	0	88.0	81.6	1.04
	0.5	89.0(+1.0)	84.1(+2.5)	0.97
	1.0	89.2(+1.2)	84.8(+3.2)	0.94
	1.5	89.4 (+1.4)	85.0(+3.4)	0.98
	2.0	89.4 (+1.4)	85.0(+3.4)	0.92
6.0	0	88.0	82.1	1.01
	0.5	89.0(+1.0)	83.7(+1.6)	0.97
	1.0	89.5(+1.5)	85.1(+3.0)	0.95
	1.5	89.5(+1.5)	85.6(+3.5)	0.94
	2.0	89.7 (+1.7)	84.9(+3.3)	0.87
15.0	0	88.1	82.0	1.20
	0.5	89.3(+1.2)	85.1(+3.1)	0.94
	1.0	89.5(+1.4)	85.9(+3.9)	0.88
	1.5	89.8 (+1.7)	86.4(+3.4)	0.91
	2.0	89.8 (+1.7)	86.4(+3.4)	0.80
18.0	0	88.1	82.4	1.20
	0.5	89.5(+1.4)	85.3(+2.9)	1.12
	1.0	89.9(+1.8)	86.2(+3.8)	1.10
	1.5	90.0(+1.9)	86.2(+3.8)	1.07
	2.0	90.0 (+1.9)	84.9(+3.3)	1.07

There was no significant increase in final brightness when the reaction time was increased from 3.0 h to 18.0 h and PAA dose was increased from 0.5 to 2.0 kg/TP (Table 2.2). Brightness increased by 1.0, 1.4, 1.4 and 1.4 ISO points & CIE whiteness increased by 2.5, 3.2, 3.4 and 3.4 points at PAA dose level of 0.5, 1.0, 1.5 and 2.0 kg/TP respectively in 3.0 h. The brightness increased by 1.4, 1.8, 1.9 and 1.9 ISO points and CIE whiteness increased by 2.9, 3.8, 3.8 and 3.6 points at a PAA dose level of 0.5, 1.0, 1.5 and 2.0 kg/TP respectively in 18 h. Brightness stability was found to be better as compared to control in all the cases (Table 2.2).

Reaction time was reduced from 3.0 h to 1.0 h to see the effect on brightness development. Increase in brightness and whiteness was almost same in 1.0, 2.0 and 3.0 h reaction time (Table 2.3). Viscosity of treated samples was comparable to control.

Table 2.3: Treatment of Mill A bleached pulp* with different PAA dose and reaction time at ambient temperature

Parameter	Control	PAA treatment 1.0 h		Control	PAA treatment 2.0 h		Control	PAA treatment 3.0 h	
PAA added (kg/TP)	--	0.5	1.0	--	0.5	1.0	--	0.5	1.0
Initial pH	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
Final pH	7.0	4.5	4.3	7.0	4.6	4.4	7.0	4.5	4.3
Time (min)	60	60	60	120	120	120	180	180	180
Bleached pulp properties									
Final brightness (% ISO)	87.8	88.9 (+1.1)	89.0 (+1.2)	87.9	88.9 (+1.0)	89.1 (+1.2)	87.8	89.0 (+1.2)	89.1 (+1.3)
CIE whiteness	81.7	83.4 (+1.7)	84.1 (+2.4)	81.8	83.8 (+2.1)	84.4 (+2.7)	81.8	84.1 (+2.4)	84.2 (+2.4)
L*	96.11	96.38	96.41	96.34	96.31	96.41	96.11	96.36	96.25
a*	0.82	0.82	0.80	0.85	0.80	0.80	0.81	0.76	0.77
b*	1.87	1.65	1.52	1.73	1.65	1.45	1.98	1.49	1.59
ASTM yellowness	2.87	2.54	2.33	2.71	2.53	2.23	3.04	2.28	2.30
Viscosity (cp)	9.6	9.6	9.3	9.7	9.6	9.5	9.7	9.7	9.7

** Initial pulp brightness 87.8% (PAA 40% concentration)*

Table 2.4: Treatment of Mill A bleached pulp* at varying PAA dose for 3 h at ambient temperature

Parameter	Control	PAA treated				
PAA added (kg/TP)	--	0.25	0.50	1.0	1.5	2.0
Initial pH	7.0	7.0	7.0	7.0	7.0	7.0
Final pH	7.0	5.7	4.8	4.5	4.2	3.8
Bleached pulp properties						
Final brightness (% ISO)	87.8	88.5 (+0.7)	88.9 (+1.1)	89.1 (+1.3)	89.2 (+1.4)	89.1 (+1.3)
CIE whiteness	79.3	80.5 (+1.2)	81.3 (+2.0)	81.8 (+2.5)	82.4 (3.1)	82.6 (+3.3)
L*	96.39	96.57	96.77	96.85	96.87	96.75
a*	0.64	0.61	0.56	0.50	0.49	0.58
b*	2.55	2.40	2.32	2.26	2.16	2.04
ASTM yellowness	3.90	3.66	3.54	3.44	3.29	3.12

** Initial pulp brightness 87.8% (PAA 40% concentration)*

Table 2.5: Impact of PAA dose on pulp properties (Mill A) for 3 h reaction time at ambient temperature

Parameter	Control	PAA treated	
PAA added (kg/TP)	--	0.5	1.0
Initial pH	7.0	7.0	7.0
Final pH	7.0	4.5	4.2
Consistency (%)	10	10	10
Bleached pulp properties			
Final brightness (% ISO)	89.1	90.1 (+1.0)	90.2 (+1.1)
CIE whiteness	84.0	86.3 (+2.3)	86.6 (+2.6)
L*	96.45	96.77	96.77
a*	0.77	0.84	0.91
b*	1.57	1.23	1.17
ASTM yellowness	2.41	1.88	1.79
Viscosity (cp)	8.5	8.4	8.3

** Initial pulp brightness 89.1% (PAA 40% concentration)*

Increase in brightness and whiteness was slightly lower when PAA dose was reduced to 0.25 kg/TP. Increase in brightness was 0.7, 1.1, 1.3, 1.4 and 1.3 ISO points and increase in CIE whiteness was 1.2, 2.0, 2.5, 3.1 and 3.3 points at PAA dose level of 0.25, 0.50, 1.00, 1.50 and 2.00 kg/TP respectively (Table 2.4).

Increase in brightness and whiteness was almost comparable when pulps of higher initial brightness (89.1% ISO) were treated with PAA at a dose level of 0.5 and 1.0 kg/TP (Table 2.5). PAA treatment did not result in any adverse effect on viscosity.

Mill B pulp (60% bamboo + 40% hardwood)

Bleached pulp was treated with PAA at ambient temperature and 50°C for 3 h at

Table 3: Effect of PAA dose on bleached pulp properties (Mill B) on treatment at ambient temperature and 50^{0}C for 3 h

PAA dose (kg/TP)	Reaction temp (^{0}C)	Brightness (% ISO)	CIE whiteness	Viscosity (cp)
0	25	87.8	79.3	9.2
	50	87.8	79.3	9.2
0.5	25	88.9(+1.1)	81.3(+2.0)	9.1
	50	89.0(+1.2)	81.5(+2.2)	9.1
1.0	25	89.1 (+1.3)	81.8(+2.5)	9.1
	50	89.1 (+1.3)	82.0(+2.7)	9.0
1.5	25	89.2 (+1.4)	82.4(+3.1)	9.0
	50	89.2 (+1.4)	82.5(+3.2)	9.0

Table 4: Effect of PAA dose on bleached pulp properties (Mill C) on treatment at ambient temperature and 50^{0}C for 3 h

PAA dose (kg/TP)	Reaction temp (^{0}C)	Brightness (% ISO)	CIE whiteness	Viscosity (cp)
0	25	86.8	77.9	8.8
	50	86.9	78.0	8.7
0.5	25	88.0(+1.2)	80.9(+3.0)	8.7
	50	88.1(+1.2)	81.2(+3.2)	8.6
1.0	25	88.3(+1.5)	81.0(+3.1)	8.7
	50	88.3(+1.4)	81.3(+3.3)	8.6
1.5	25	88.4(+1.6)	81.4(+3.5)	8.6
	50	88.4(+1.5)	81.6(+3.6)	8.5

Table 5: Impact of PAA dose on properties of dissolving grade pulp (Mill D)

Parameter	Control	Treated (0.5kg/TP)	Treated (1.0 kg/TP)
Brightness (% ISO)	89.0	89.7(+0.7)	90.0(+1.0)
CIE whiteness	84.5	86.1(+1.6)	86.7(+2.2)
L*	96.76	96.90	96.97
a*	0.62	0.67	0.80
b*	1.62	1.35	1.25
ASTM Yellowness	2.49	2.06	1.92

Reaction time 3 h at ambient temperature

Table 6.1: Treatment of Mill A pulp with PAA for 3 h at ambient temperature

Parameter	Control	Treated (0.5kg/TP)	Treated (1.0 kg/TP)
Brightness (% ISO)	87.3	88.0(+0.7)	88.4(+1.1)
CIE whiteness	82.7	84.4(+2.3)	85.5(+2.8)
L*	96.36	96.22	95.14
a*	0.71	0.56	0.78
b*	1.80	1.57	1.54
ASTM Yellowness	2.76	2.01	1.84

Table 6.2: Improvement in optical properties* of high brightness paper (70 gsm) by pretreatment of pulp with PAA

Parameter	Control	Treated (0.5kg/TP)	Treated (1.0 kg/TP)
Brightness (% ISO)	96.8	97.4 (+0.6)	97.9 (+1.1)
CIE whiteness	139.5	141.1 (+1.6)	142.7 (+3.2)
L*	97.81	97.86	97.90
a*	2.86	2.87	2.87
b*	-10.28	-10.61	-10.96
Fluorescence	23.9	24.5	24.7
Opacity (% ISO)	80.1	80.8	81.0
Scattering Coeff. (m^2/kg)	37.0	37.3	38.0

**Optical properties determined using Spectraflash 300 UV (Datacolor, USA)*

a dose level of 0.50, 1.0 and 1.5 kg/TP. Increase in brightness and whiteness was almost same at 50^{0}C as well as ambient temperature. Increase in brightness was 1.1, 1.3 and 1.4 ISO points at ambient temperature and 1.2, 1.3, 1.4 ISO points at 50^{0}C; CIE whiteness increased by 2.0, 2.5 and 3.1 points at ambient temperature and 2.2, 2.7, 3.2 points at 50^{0}C at a dose level of 0.5, 1.0 and 1.5 kg PAA/TP respectively (Table 3.0). The viscosity of PAA treated pulps was comparable with the reference pulp.

Mill C pulp (35% bamboo + 65% hardwood)

Bleached pulp was given PAA treatment similar to Mill B pulp. Increase in brightness and whiteness was almost same at 50^{0}C as well as ambient temperature. The increase in brightness was 1.2, 1.5 and 1.6 ISO points at ambient temperature and 1.2, 1.4 and 1.5 ISO points at 50^{0}C; increase in CIE whiteness was 3.0, 3.1 and 3.5 points at ambient temperature and 3.2, 3.3 and 3.6 points at 50^{0}C at a dose level of 0.5, 1.0 and 1.5 kg PAA/TP respectively (Table 4). The viscosity of PAA treated pulps was comparable with the reference pulp.

Mill D pulp (dissolving grade)

Bleached dissolving grade pulp was treated with PAA at ambient temperature for 3 h at a dose level of 0.5 and 1.0 kg/TP. The brightness increased by 0.7 and 1.0 ISO points and CIE whiteness increased by 1.6 and 2.2 points when the pulp was treated with PAA at a dose level of 0.5 and 1.0 kg/TP respectively (Table 5).

Improvement in optical properties of high brightness paper by pretreatment of pulp with PAA

Bleached pulp from Mill A was treated with PAA at ambient temperature for 3 h at a dose level of 0.5 and 1.0 kg/TP. The Increase in brightness was 0.7 and 1.1 ISO points and CIE whiteness was 2.3 and 2.8 points when PAA treatment was done at a dose level of 0.5 and 1.0 kg/TP respectively (Table 6.1). Almost similar increase in brightness and whiteness was observed when PAA treated pulps were used for the

Table 7: Use of PAA in E_{OP} stage effect on pulp properties (Mill B)

Parameters	Control	PAA (1.0kg/TP in E_{OP})	PAA (0.5kg/TP in E_{OP})
PAA dose (kg/TP in E_{OP})	--	1.0	0.5
Brightness (% ISO)	90.5	90.9	90.8
CIE whiteness	85.3	86.0	85.9
Bleached pulp yield (%)	95.3	95.8	95.3
PC no.	0.70	0.67	0.65
Viscosity (cp)	9.0	8.6	8.7
Bleach chemical consumption (kg/TP)			
Cl_2	37.0	37.0	37.0
NaOH	30.4	30.4	30.4
Hypo	10.0	10.0	10.0
H_2O_2	8.6	8.6	8.6
PAA	--	1.0	0.5
ClO_2	10.6	10.6	10.6
$C_DE_{OP}HEpD$ sequence			

Table 8.1: Use of PAA in D stage effect on pulp properties (Mill B)

Parameter	Control	Treated (0.5kg/TP)	Treated (1.0 kg/TP)
PAA dose (kg/TP in D)	--	0.5	1.0
Brightness (% ISO)	90.5	90.8(+0.3)	91.0(+0.5)
CIE whiteness	85.3	85.9 (+0.6)	86.0 (+0.7)
Bleached pulp yield (%)	95.3	95.2	94.7
PC no.	0.70	0.68	0.70
Viscosity (cp)	9.0	8.8	8.8
Bleach chemical consumption (kg/TP)			
Cl_2	37.0	37.0	37.0
NaOH	30.4	30.4	30.4
Hypo	10.0	10.0	10.0
H_2O_2	8.6	8.6	8.6
PAA	--	0.5	1.0
ClO_2	10.6	10.6	10.6
$C_DE_{OP}HEpD$ sequence			

Table 8.2: Reduction of ClO_2 with addition of PAA in D stage (Mill B)

Parameter	Control	PAA (0.5kg/TP in D)	PAA (1.0kg/TP in D)	PAA (1.5kg/TP in D)
PAA dose (kg/TP in D)	--	0.5	1.0	1.5
Brightness (% ISO)	90.5	90.8	90.5	90.2
CIE whiteness	87.3	87.9	86.7	86.8
Bleach chemical consumption (kg/TP)				
Cl_2	37.0	37.0	37.0	37.0
NaOH	30.4	30.4	30.4	30.4
Hypo	10.0	10.0	10.0	10.0
H_2O_2	8.6	8.6	8.6	8.6
PAA	--	0.5	1.0	0.5
ClO_2	10.6	10.6	9.6	7.6
$C_DE_{OP}HE_PD$ sequence				

production of high brightness paper (Table 6.2). The brightness and whiteness of PAA treated (1.0 kg/TP) high brightness paper were 97.9% ISO (+1.1) and 142.7 points (+3.2) respectively whereas in case of control (no PAA treatment), the brightness and whiteness were 96.8% ISO and 139.5 points respectively.

Application of PAA in E_{OP} stage

Not much improvement in final brightness (+0.4%ISO) and whiteness (+0.7 points) was observed when PAA was used in the E_{OP} stage at a dose level of 0.5-1.0 kg/TP (Table 7).

Application of PAA in D stage

Not much improvement in final brightness and whiteness was observed when PAA was used at a dose level of 0.5-1.0 kg/TP in the D stage. It was found that about 1.0 and 3.0 kg of ClO_2 could be saved by addition of 1.0 and 1.5 kg PAA /TP in the D stage (Tables 8.1- 8.2). Use of per-acetic acid in D stage can partially replace ClO_2 where ClO_2 is a limitation.

CONCLUSIONS

Treatment of bleached mill pulps with per-acetic acid at a dose level of 0.5-1.0 kg/TP at ambient temperature and normal plant pulp pH increases the final pulp brightness by 1.0 to 1.5 points and whiteness by 2.0 to 3.5 points. Yellowness and PC number are also reduced and pulp viscosity is not affected. Treatment time of 2-3 hours is found to be sufficient.

Improvement in brightness and whiteness is sustained when PAA treated pulp is used for the production of high brightness paper (+96% ISO).

The brightness increases by 0.7 and 1.0 ISO points and CIE whiteness increases by 1.6 and 2.2 points when the dissolving grade pulp is treated with PAA at a dose level of 0.5 and 1.0 kg/TP respectively at ambient temperature for 3 hours.

Addition of PAA at a dose level of 0.5-1.0kg/TP in E_{OP} and dioxide stage does not result in much improvement in final brightness and whiteness. About 1.0 and 3.0 kg of ClO_2 can be replaced by

treatment with PAA at a dose level of 1.0 and 1.5 kg/TP respectively. Use of per-acetic acid in D stage can partially replace ClO_2 where ClO_2 is a limitation.

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